Chapter - III

Materials and Methods

3.1 Materials

3.1.1 Handloom fabrics

Five 100% cotton handloom fabrics suitable for shirting and equivalent dress material were collected for the study from village weavers of different districts of Odisha state (India), the photographs of which are shown in Fig. 3.1. The specialities of these fabrics are that the woven designs have been created by using warp and weft yarns coloured by “tie & dye” technique, and no post weaving finishing have been applied for further value addition as done in textile mills. The specifications of the shown fabrics are given in Table 3.1.

Fig. 3.1 Handloom fabric samples
Table 3.1 Specifications of handloom fabric

<table>
<thead>
<tr>
<th>Sample</th>
<th>Warp count Ne (tex)</th>
<th>Weft count Ne (tex)</th>
<th>Weave</th>
<th>Ends/inch</th>
<th>Picks/inch</th>
<th>GSM</th>
<th>Thickness (mm)</th>
<th>Cover factor</th>
</tr>
</thead>
<tbody>
<tr>
<td>Sample 1</td>
<td>50 (11.8)</td>
<td>49 (12.0)</td>
<td>Plain</td>
<td>61</td>
<td>57</td>
<td>95.25</td>
<td>0.35</td>
<td>14.26</td>
</tr>
<tr>
<td>Sample 2</td>
<td>48 (12.3)</td>
<td>44 (13.4)</td>
<td>Plain</td>
<td>70</td>
<td>72</td>
<td>77.18</td>
<td>0.30</td>
<td>17.04</td>
</tr>
<tr>
<td>Sample 3</td>
<td>44 (13.4)</td>
<td>40 (14.8)</td>
<td>Plain</td>
<td>58</td>
<td>60</td>
<td>81.92</td>
<td>0.38</td>
<td>15.27</td>
</tr>
<tr>
<td>Sample 4</td>
<td>51 (11.6)</td>
<td>53 (11.1)</td>
<td>Plain</td>
<td>86</td>
<td>78</td>
<td>72.25</td>
<td>0.26</td>
<td>18.15</td>
</tr>
<tr>
<td>Sample 5</td>
<td>60 (9.8)</td>
<td>60 (9.8)</td>
<td>Plain</td>
<td>66</td>
<td>62</td>
<td>94.68</td>
<td>0.38</td>
<td>14.10</td>
</tr>
</tbody>
</table>

3.1.1.1 Justification of selecting the above mentioned handloom fabrics for this study

The handloom sector of Odisha has achieved a very rich and traditional heritage in the country as well as in the globe. As per the information available with Directorate of Textiles, Govt. of Odisha, there are 43,652 handlooms prevailing in the state along with 1,92,339 weavers population. The production from this sector accounts to 8.42 million square meters of cotton handloom materials, which includes saree, dhoti, shirting, gamochha, lungi, bedsheets, furnishing items and chadder. Out of the above said productions of 8.42 million square meters, only 0.338 million square meters of hand woven fabrics (4.01%) are produced with requisite specification for use as dress materials and the sale value of that apparel based handloom fabrics, comes approximately 33.288 million rupees (INR). The above mentioned five handloom fabric samples were identified for the study on the basis of the sales turnover of handloom fabrics during the last three years in the state of Odisha. The specifications of the samples shown in Table 1 also justify their suitability for shirting and equivalent dress material in summer. So, during this research work, it was planned for value addition keeping in view the promotion of such fabrics in garment manufacturing.

3.1.2 Mill made fabrics

In order to justify the belief that mill made fabrics are smoother than handloom fabrics, five mill made fabrics were also procured having construction similar to handloom fabrics, the specifications of which are mentioned in Table 3.2.
Table 3.2 Specifications of mill made fabric

<table>
<thead>
<tr>
<th></th>
<th>Warp count Ne (tex)</th>
<th>Weft count Ne (tex)</th>
<th>Weave</th>
<th>Ends/ inch</th>
<th>Picks/ inch</th>
<th>GSM</th>
<th>Thickness (mm)</th>
<th>Cover factor</th>
</tr>
</thead>
<tbody>
<tr>
<td>Sample 1</td>
<td>41(14.4)</td>
<td>32 (18.4)</td>
<td>Plain</td>
<td>84</td>
<td>74</td>
<td>93.20</td>
<td>0.28</td>
<td>20.07</td>
</tr>
<tr>
<td>Sample 2</td>
<td>40 (14.8)</td>
<td>45 (13.1)</td>
<td>Plain</td>
<td>74</td>
<td>57</td>
<td>87.20</td>
<td>0.29</td>
<td>16.65</td>
</tr>
<tr>
<td>Sample 3</td>
<td>39 (15.1)</td>
<td>35 (16.9)</td>
<td>Plain</td>
<td>87</td>
<td>82</td>
<td>90.83</td>
<td>0.28</td>
<td>20.90</td>
</tr>
<tr>
<td>Sample 4</td>
<td>36 (16.4)</td>
<td>43 (13.7)</td>
<td>Plain</td>
<td>85</td>
<td>44</td>
<td>86.23</td>
<td>0.29</td>
<td>17.48</td>
</tr>
<tr>
<td>Sample 5</td>
<td>55 (10.7)</td>
<td>60 (9.8)</td>
<td>Plain</td>
<td>88</td>
<td>66</td>
<td>85.60</td>
<td>0.31</td>
<td>16.80</td>
</tr>
</tbody>
</table>

3.2 Softener and Crease resistant chemicals

Considering the present trend of research and industrial application in the field of garment finishing, fabric softener (silicone softener) and non- formaldehyde based crease resistance finish (resin) were selected for treatment with handloom fabrics for value addition. These chemicals were collected from M/S Clarient Chemicals Ltd, Kolkata (India). Magnesium chloride (MgCl₂) and acetic acid used as catalyst and pH regulator respectively during the finishing treatment of the fabric were collected from reputed scientific chemical suppliers.

3.2.1 Justification for choosing silicone as finish agent

With chemical softeners, textiles can achieve an agreeable soft hand and some smoothness. However, the disadvantages sometimes seen with chemical softeners include reduced crock fastness, yellowing of white goods, changes in hue of dyed goods and fabric structure slippage. Most of these types of softeners consist of molecules with both hydrophobic and hydrophilic part. Therefore, they can be classified as surfactants (surface active agents) and are to be found concentrated at the fibre surfaces. Most softeners have low water solubility. Therefore softening products are usually sold as oil in water emulsions containing 20-30% solids. The softener molecules typically contain a long alkyl group, sometimes branched, more than 16 and up to 22 carbon atoms, but most have 18 carbon atoms. Exceptions to this molecular structure are the silicone softeners. About one-third of the softeners used in the textile industry are silicone based as the softening treatments by silicone finish impart soft handle (supple, pliant, sleek and fluffy), smoothness and enhance flexibility, drape and pliability [97]. Other properties improved due to
treatment with silicone softener include the feeling of added fullness, antistatic properties and sew ability. Further, silicone softeners are also used in resin finishing of textiles to have a soft wrinkle resistant fabric. It is commonly observed that silicones are the most effective softening agents due to their inert nature, low surface tension and the necessary functional modifications that can be effected to the chain. Silicone softeners are becoming extremely important because of their good softness and greater wash permanence compared to other softeners. A large amount of silicones is used in textiles as softeners [98]. Roy Choudhury et.al have made an intensive research work on various types of silicone softeners and have focused on the effect of silicone treatment for better hand value of cotton fabrics [30]. The silicone softeners are used on woollen industries also, which considerably improves the quality of woolen textile materials by modification of fibre surface by changing the physical and chemical nature of wool fibre surface [4]. Silicone finishes are widely recognized as the best materials for increasing the softness of fabrics, enhancing their aesthetic feel and imparting an excellent hand. Silicones have been responsible for giving super softness to fabrics over the years and to produce the optimum handle for apparel fabrics with suitable comfort properties has presented an interesting challenge [29]. Considering the published literatures, research activities carried out related to silicone softeners and wide applications of finishing treatment with silicone in garment manufacturing industries, silicone softener was chosen in the study for softening of handloom fabrics. Accordingly, ‘Solusoft MW conc’ an amino modified silicone softener was collected from M/S Clarient Chemicals Ltd, Kolkata (India) for treatment with handloom fabrics for value addition.

3.2.2 Justification for choosing non-formaldehyde based resins with silicone for treatment with handloom fabrics to impart wrinkle free effect

The ‘wash and wear finish treatments’ to the fabric, specially to cotton fabrics to be used in garments refers to crease resistant or wrinkle free treatment or resin treatment. To impart crease resistant property to cotton materials, the hydrogen bond formation by the hydroxyl groups should be removed. A popular and widely used method of imparting the crease resistant finish is the one, in which the hydroxyl groups of adjacent macro molecules are reacted with bifunctional chemicals forming a crosslink with elimination of water or methanol molecules [31]. Various methods and chemicals are in use in order to make cotton textiles to achieve crease
resistant property. In literatures and industrial practices ‘di-methyl di-hydroxyl ethylene urea (DMDHEU)’ is most commonly used as cross linking agent.

Vaddi and Balakrishnaiah [39] have treated khadi and handloom fabrics with ‘DMDHEU’ finish and noticed wrinkle-free characteristics. Huang [41] had used a combination of ethylene urea & para-formaldehyde with different mol ratios as source material to synthesize a DMEU/MMEU resin pre-polymer in various mixing ratios in order to impart crease resistance property. Mortazavi & Boukany [40] had worked with a mixture of DMDHEU, PVAc & some other resins for developing anti crease nature of cotton fabrics. Later on, use of DMDHEU as crease resistant finish was stopped, because formaldehyde based finishing treatments causes high strength loss of treated cotton and release of excessive fumes in the atmosphere even though DMDHEU treatment is having low chemical cost and high finish durability [43]. It was found that formaldehyde and hydrochloric acid in the presence of water can form bichloromethyl ether (BCME), which is a human carcinogen, irritant and causes allergy to human beings. So Arkofix NZF, a modified dihydroxyethylene urea (DHEU) supplied by M/S. Clarient Chemicals Ltd, Kolkata (India), which is a formaldehyde-free crosslinking agent, comprising only the dihydroxyethylene groups and no methylol groups have been used during this study.

Hence, ‘silicone’ as a softening finish and silicone with non- formaldehyde based resins as crease resistant finish agents were chosen in this experimental study for enhancing value added property of cotton hand woven fabrics.
3.3 Bio based fabric softener and antimicrobial agents
Leaves of mangrove plants were collected from mangrove forests of coastal district of Odisha state, India in order to develop bio based fabric softener and antimicrobial agents for treatment with cotton handloom fabrics for value addition. The specimens were identified by the scientists of the Department of Natural Products, Institute of Mineral and Material Technology, Bhubaneswar, Odisha, India.

3.3.1 Justification of choosing leaves of Mangrove plant for imparting softening and antimicrobial property
In the present global scenario, there is a great demand and trend for development of bio active agents for giving different treatments with textile materials for imparting better functional values. Various types of plant and animal based products are also commercially available for this purpose. Treatments with enzymes are mostly used in textile finishing processes in order to improve softness, smoothness and fashionable appearance. Dhurai et al. [62] have observed that treatments with enzyme have resulted with reduction in hairiness, kinetic friction and high improvement in softness characteristics (compression characteristics) of ring-spun carded and rotor spun yarn. Khalil et al [63] have highlighted the effect of enzyme and the enzyme- silicone wash on different physical properties e.g. fabric weight, tearing strength, hairiness and colour fastness property of denim garments. Menzes [77], Gettings and Triplett [78] have reported the use of natural products such as chitosan and natural dyes, herbal products such as Aloe Vera, Tea tree oil, Eucalyptus oil and Tulsi leaf extracts and many other medicinal plants as antimicrobial finishing of textile materials. Joshi [76 & 81] had worked on natural agents, such as neem extracts, natural dyes, chitosan and other herbal products (tulsi, aloe vera, tea tree oil etc.) on textile substrates. Gupta & Laha [82] have studied the antimicrobial activity of cotton fabric treated with Quercus infectoria extract. Thilagavathi and Kannaian [83] have applied extract of prickly chaff (Achyranthes aspera Linn) leaves as herbal antimicrobial finish on cotton fabrics. Jothi et al [84] have found antimicrobial efficacy of aloe gel extract from aloe vera plant on cotton fabrics. Tilagavathi [86] has observed that tulsi leaves are suitable for textile application as antimicrobial agents. Reports are also available about many other important natural products, such as karanga oil, cashew shell oil, henna or mehndi, which are explored on textile substrates for antimicrobial property which will have tremendous application in apparels and medical
textile. Chen & Chang [87] have studied the effect of treatment of onion extracted with ethanol on cotton fabric.

Mangrove plants have wide scale uses as medicines in this country. The study by Bamroongrugs [88] reveals that their root, bark, leaves and fruits have certain medical effects. They are well known to produce natural metabolites with diverse biological activities, such as antibacterial [89], antiviral activity [90], anti-diarrhoeal activity [91], anti-mitotic, insecticidal activity [92], cytotoxic activity [93], anti-plasmodia and many other diseases. Bandaranayke [94] has analyzed that according to the chemical structure of mangrove plants, most of the isolated compounds belong to steroids, triterpenes, saponins, flavonoids, alkaloids, tannic and phenolic which having a wide range of therapeutic possibilities. Bandaranayke [95] has observed that the mangrove plant posses strong antibacterial properties against a broad range of microorganisms. However, no literatures are available on treatment of extracts of mangrove plants on textiles for imparting softness and antimicrobial property. Hence, it was planned to make experimental study with leaf extract of mangrove plants for treatment with cotton handloom fabrics as bio-softener and antimicrobial finishing agent for value addition.

3.4 Analysis of fabric specifications

3.4.1 Determination of ends and picks per inch

Ends and picks per inch in the fabrics were determined using ‘Traverse Thread Counter’ (ASIAN Make).

![Fig. 3.2 Traverse Thread Counter](image-url)
3.4.2 Determination of yarn count

Determination of count of yarns used in the manufacture of the above mentioned fabrics was carried on Beasley Balance (ASIAN Make). The following test procedure was adopted in order to find out the count of the yarn used in the fabrics.

Fabrics were cut both warp way and weft way according to the size of the template provided with the machine. Yarns were removed from the fabrics one by one and placed on the hook for coincidence of the pointer with the V-indicator. The number of yarns was then counted. The number of yarns is the ‘count’ of the given fabric.

3.4.3 Determination of Thickness of the fabric

Thickness of fabrics samples were measured by using ASIAN Make ‘Bench Thickness Gauge’.

![Fig. 3.3 Thickness Gauge](image)

**Technical specifications of the Machine**

- **Range of measurement**: 0-10mm
- **Least count of dial gauge**: 0.01
- **Diameter of anvil**: 50 mm approx.
- **Diameter of indenter**: 9.5 mm
- **Pressure on indenter**: 10 kN/m² and 24 kN/m²
- **Throat depth**: 50 mm

The indenter was lifted with the help of the lifting lever fixed at the back of the gauge. The fabric sample was placed on the anvil. The indenter was gently lowered on the sample. The reading of the dial gauge was recorded. The process was repeated from random locations on the sample fabric.
3.4.4 Determination of weight of fabric (grams per square meter)
Swiss weighing balance was used for the purpose. The samples for weighing were randomly cut equal to the size of the template (10cm x 10cm) from the fabric. The samples were weighed individually in the weighing balance. The readings were converted to the weight in grams of the fabric per square meter.

3.5 Methods for different finishing treatments

3.5.1 Softening treatment
The handloom fabrics are not traditionally subjected to post weaving finishing treatment before reaching the market. As a result, fabrics procured from the handloom weavers contain size materials applied on the coloured warp yarns, which are not removed after weaving. So before applying the finishing treatments, all the fabric samples (untreated samples) were desized in boiling water for 45 minutes, by adding 0.5% of non-ionic detergent and material to liquor ratio was maintained @ 1:20. The samples were then dried at room temperature.

Softening treatment was done by pad dry cure method. Softener (silicone) solution of different concentrations i.e. 10 g/l (grams/litre), 20 g/l, 30 g/l, 40 g/l and 50 g/l were prepared as per the following procedure.

Water calculated on material (weight of fabric): water @ 1:10 was taken in a container and heated up to a temperature of 40°C. Silicone softener of 10 grams/litre (concentration) was added slowly to the solution and stirred gentle. The pH of the solution was maintained at 5.5 by adding drop by drop of acetic acid. The desized cotton handloom fabric samples were ironed properly to remove all creases and immersed in to the solution and stirred for 15 minutes. Then the fabric was padded at 1kg/cm² pressure in an automatic padding mangle machine to achieve at least 80% wet pick-up in order to get add-on of 0.8% on the weight of material. The said procedure of treatment was repeated separately by taking 20 g/l, 30 g/l, 40 g/l and 50 g/l of silicone respectively so as to achieve treatment at different concentrations for resulting 1.6, 2.4, 3.2 and 4% of add-on of silicone on fabrics.

The padding mangle was switched on with pressure at 2 bars. The treated fabric was inserted between the two roller pads and locked. The machine was set up at a suitable speed. Then, the machine was started by turning the start knob in clockwise direction which makes the fabric
move in forward direction. The fabric was passed through the roller pads and extra liquor was squeezed. After the entire fabric was passed once between the pads, the start knob was turned in the anticlockwise direction which led the fabric to move in the reverse direction and made to pass through the roller pad second time. The padded fabric was then weighed. The pickup percentage calculated basing on weight of dry fabric and wet fabric. The same procedure was repeated for all samples. Then the fabrics were dried at 100°C for 5 minutes and cured at 150°C for 3 minutes in drying and curing chamber. The fabrics were then taken out from the curing chamber, cooled at room temperature and ironed.

### 3.5.2 Crease resistant finish treatment

The handloom fabrics are not traditionally subjected to post weaving finishing treatment before reaching the market. As a result, fabrics procured from the handloom weavers contain size materials applied on the coloured warp yarns, which are not removed after weaving. So the fabric samples were desized first in boiling water for 45 minutes maintaining material (weight of fabric): liquor (water + 0.5% non-anionic detergent) = 1:20, and dried in room temperature.

Crease resistant treatment was done by pad dry cure method. Water calculated on material (weight of fabric): water @ 1: 20 was taken in a container and heated up to a temperature of 40°C. Non formaldehyde resin finish (liquid) @ 10 g/l was poured in to the container. Silicone softener @ 30% by weight of resin liquor and MgCl₂ (as catalyst) @ 10% by weight of resin liquor were added to the said container & mixed well by stirring. The pH of the solution was maintained at 5.5 by adding drop by drop of acetic acid. Then, the desized handloom cotton fabric sample was dipped into the said container of finish solution and stirred for around 05 minutes with the help of a glass rod. Then, the treated fabric was padded at 1kg/cm² pressure in
an automatic padding mangle machine to achieve 80% - 90% expression in order to get optimum
pick up of 0.8- 0.9 on the weight of material. Then the fabric was dried at 100\(^\circ\)C for 3 minutes
and cured at 150\(^\circ\)C for 4 minutes in drying and curing chamber. The fabric was then taken out
from the curing chamber, cooled at room temperature and ironed. Same procedure was repeated
separately by taking 20 g/l, 30 g/l, 40 g/l,50 g/l of the non-formaldehyde resin component and
silicone softener @ 30% by weight of resin liquor and MgCl\(_2\) (as catalyst) @ 10% by weight of
resin liquor, so as to make anti crease finish treatment with fabric sample at different
concentrations. All the sample fabrics were treated in the similar way in the above manner.

3.6 Different test methods

3.6.1 Scanning Electron Microscope Testing

Fig. 3.5 Front view of the Scanning Electron microscope

Specification of scanning electron microscope (SEM):

Model : EVO 18
Make : Carl Zeiss Microscopy Ltd, U.K.
Resolution : 4 nm, in low vacuum mode at 30kV and 3 nm in high vacuum mode at
30kV
Magnification : 5x to 3,00,000 x continuously variable
Filament : Tungsten (W) filament
Electron Source/ Gun : Tungsten (W) hair pin filament
Vacuum System : Fully automated Rotary pump with Turbo molecular pump
Both untreated and treated fabrics were subjected to Scanning Electron Microscope (SEM) test available in the Institute laboratory for getting the highly magnified internal structure. The binding of the finish materials into the internal structure of cotton fabrics, treated at different concentrations of silicones, were verified by Scanning Electron Microscope (SEM) test. Fabric samples were mounted on sample mount made of aluminium. The samples were then placed in the plasma chamber and gold palladium coating was given. After successful coating was done, the samples were placed in the chamber of the SEM and chamber door was closed. Proper vacuum was maintained inside the chamber and gun with the help of vacuum pump. When the preset vacuum was reached, the gun EHT (High Voltage) started emitting electron beams. The magnification and resolution was decided as per the best viewing conditions on the screen of the monitor. The fabrics were magnified to the extent of 500 to 600 and yarns, in the range of 10,000 to 11,000.

3.6.2 Stiffness measurement

Bending length of fabric samples were measured using Asian make ‘Stiffness Tester’ following IS 6490:1971(BIS 2000) method.

![Fig. 3.6 Stiffness Tester](image)

Fabric samples of 25 x 200 mm along warp way and weft way direction were cut and were conditioned in the hot air oven at 95°C for 30 minutes. Each sample was placed on the bending
length measuring equipment and a measuring scale was placed on it. It was ensured that the zero mark on the ruler should coincide with the inner edge of the sample. The ruler gradually slides along the sample so that the inner end of the sample bent at the edge of the inclination. The reflection of the bent part of the sample was observed in the mirror attached to the inner side of the equipment. The moment the reflection of the bent portion of the sample coincided with the inclined line marked on the mirror i.e. 41.5°, further sliding of the ruler was stopped and the value of the bending length was recorded from the ruler.

### 3.6.3 Softness measurement

Objective evaluation of softness of fabric samples in terms of pulling force in grams were measured using Fabric Feel Tester (Fig. 3.7.A), designed by IIT, Delhi and manufactured by Texlab Industry, Ahmedabad (India).
The basic principle of objective measurement of fabric feel characteristics through this machine is based on computerized nozzle extraction method that measures the extraction force while extracting a fabric sample through a conical nozzle (52 mm height, 12 mm top diameter and 60 mm bottom diameter). The nozzle is made of steel and chrome plated inside to minimize the frictional force between the fabric and the metal while it is being extracted. The nozzle is slit through the centre and made into two equal halves as shown in Fig. 3.7.B, which are mounted on the base plate with the help of a metal piece. The fabric samples were made free from wrinkles and crease by ironing and cut into circular shape with diameter of 240 mm and attached to sample holder in the instrument. Then the samples were drawn through the conical shaped nozzle (Fig. 3.7.C). Two load cells are connected to the back of these halves such that they form a closed nozzle loop when joined. As the clamp moves upward, it extracts the fabric specimens through the nozzle.

The force required for extracting the fabric specimens through the nozzle changes as increasing portion of the fabric was introduced in the nozzle. The fabric specimen gets folded, sheared, rubbed, compressed and bent (multiple directions) during extraction.

A typical force displacement graph is generated which is displayed in the monitor of the Fabric feel Tester (Fig. 3.8) and the values are recorded.

![Graph generated by Fabric Feel Tester during testing of a fabric](image-url)
3.6.4 Objective measurement of surface roughness by using Digital Image Processing (DIP)

The degrees of roughness of untreated fabrics as well as the improvement in the feel of the fabrics due to silicone treatment were evaluated objectively by ‘Digital Image Processing (DIP)’ method using Matlab software. This method is based on the principle of surface height variation. The surface roughness measurement of woven cotton handloom fabric by using Digital Image Processing (DIP) process is a simple and efficient process as it is free from mechanical errors (non contact method). The process of measurement of surface roughness starts with scanning of the samples. Untreated and treated handloom fabric samples were scanned using a 600 dpi scanner. After scanning, the images obtained were cropped at a fixed level of pixels (100 x 100) so as to have a uniform and regular figure.

The surface of fabric is supposed to be of minimum roughness. So, in order to compare the roughness of different fabrics, a reference is needed. Human finger (thumb) was taken as reference during this study since it presents the most pleasant feel for human tactile sensation because the frictional coefficient of textiles is generally related to their touch by human (handle properties) which is termed as an ideal surface. So, human thumb was scanned, cropped at a fixed level of pixels and used as reference during comparison of roughness of fabric samples.

A suitable programme (Annexure – I) was made as mentioned below taking into consideration of the parameters like;

a) Number of peaks, where a peak is defined as a point at which height is higher than those of its neighbourhood points and it has equal height to some of its neighbours.

b) Distance of peak to origin and its variance.

c) Volume of the simulated profile from the image.

d) Ratio of variance to the mean of the gray scale levels of the images

e) Variance of grey scale levels of peaks in the profile

f) Three dimensional surface plot

Simulation was done by using Matlab software. Gaussian filters were applied on the image to blur it and remove noise. The main justification for using Gaussian filters as a smoothing filter is to remove high spatial frequency components from the image. All the scanned images were loaded in Matlab and by using the programme (as per Annexure 1), a surface plot and a set of data compared to the ideal surface were generated for each image for different parameters as shown in Fig. 3.9 and Table 3.3.
Fig. 3.9 Scanned image of handloom fabric sample and surface plot

Table 3.3 Parameters generated from scanned images by processing in Matlab

<table>
<thead>
<tr>
<th>Sl. No</th>
<th>Parameters</th>
<th>Formula</th>
<th>Description</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>Number of peaks (n)</td>
<td>n - ni / n</td>
<td>Number of peak points in an ideal and real profile</td>
</tr>
<tr>
<td>2</td>
<td>Distance of peak to origin and its variance (d)</td>
<td>d - di / d</td>
<td>Variance of the distance vector in an ideal and real profile</td>
</tr>
<tr>
<td>3.</td>
<td>Volume of the simulated profile from the image (v)</td>
<td>v - vi / v</td>
<td>Integral of the surface under peaks in an ideal and real profile</td>
</tr>
<tr>
<td>4.</td>
<td>Ratio of variance to the mean of the gray scale levels of the images (x)</td>
<td>x - xi / x</td>
<td>Variance of in an ideal and real surface</td>
</tr>
<tr>
<td>5.</td>
<td>Variance of grey scale levels of peaks in the profile (g)</td>
<td>g - gi / g</td>
<td>CV% of in an ideal and real profile</td>
</tr>
<tr>
<td>6</td>
<td>Roughness index achieved = (n + d + v + x + g) / 1000</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>
The surface roughness index values have been shown and discussed in the result section. Co-efficient of correlation was calculated between the roughness indexes as determined by Digital Image Processing (DIP) method, softness values as calculated in terms of pulling force in grams, measured by Fabric feel tester and stiffness of fabrics in terms of bending length in cm, as measured by Stiffness tester which are also shown in result section.

3.6.5 Measurement of draping property

Drape coefficient value of fabric samples were measured by using MAG Make Drape testing machine which follows the testing standard of IS 8357:1977 (BIS, 2000).

![Fig. 3.10 Drape Testing Machine](image_url)

Fabric samples of 254 mm in diameter were cut and mounted on the circular stand of the disc in the upper chamber of the machine. A piece of ammonia paper was placed under the glass on which the circular stand was mounted. A bowl filled with ammonia solution was placed in the lower chamber of the machine. A Light source of 100 watt was allowed to pass through the ammonia paper for 15 minutes. For the next 15 minutes, the paper was turned upside down and placed above the bowl in the lower chamber. This resulted in developing the image of the drape of the fabric, produced on the paper. The shaded part on the paper was cut and weighed. The drape coefficient value was derived by the following formula:

\[
F (\text{drape coefficient}) = \frac{(As - Ad)}{(AD - Ad)}, \quad \text{where,}
\]

‘AD’ is the area of the specimen,

‘Ad’ is the area of the supporting disk, and

‘As’ is the actual projected area of the specimen
3.6.6 Tearing strength measurement

![Elmendorf Tear Tester](image1)

The tearing strength of fabric samples were determined by measurement of work done in tearing through a fixed length of the test specimen using Asian Make Elmendorf tear tester which follows testing standard of ASTM D1424 (2009) method.

3.6.7 Testing for rubbing fastness property

UNILAB make ‘Digital Crock meter’ was used to determine colour fastness of the fabrics by rubbing under both dry and wet conditions.

![Digital Crock Meter](image2)

In this test a moving brass finger of specified shape and size rubs against the test specimens and the amount of colour transferred to a piece of abrading fabric fixed over the base of the moving finger, are graded with the help of standard gray scales to evaluate the colour fastness against rubbing.

Technical specification of the machine:

Diameter of the finger : 16 mm±1
Load on the finger : 900 gram
Length of travel of the finger : 100 +/- 3 mm
Size of the test specimen : 200 mm × 50 mm
Testing standard : AATCC 8

The staining of the rubbed cotton cloths were assessed with the grey scale as per Grey scale rating (Table 3.4).

<table>
<thead>
<tr>
<th>Grey Scale Rating</th>
<th>Interpretation For Staining</th>
</tr>
</thead>
<tbody>
<tr>
<td>5</td>
<td>No Staining</td>
</tr>
<tr>
<td>4</td>
<td>Slight Staining</td>
</tr>
<tr>
<td>3</td>
<td>Moderate Staining</td>
</tr>
<tr>
<td>2</td>
<td>Severe Staining</td>
</tr>
<tr>
<td>1</td>
<td>Very Severe Staining</td>
</tr>
</tbody>
</table>

3.6.8 Test for assessment of pilling property

Pilling property of fabrics under study was measured using ‘ASIAN’ make Pilling Tester (Fig. 3.13), which follows ASTM D4970 standard of testing of textiles.

It consists of two cubical boxes (225mm x 225mm x 225mm), inner walls of boxes are lined by 3mm thick cork sheet. The boxes are rotated (60 rpm) by means of motor about the horizontal axis. A pre-set digital counter is provided for stopping the tester after pre-determined number of revolutions. A template, metal cylinder, comprising jig and 8-rubber tuber are provided with the instruments.
The test specimens were cut each 125 mm x 125 mm dimensions. The back side of the fabric was marked in length direction. 12 mm from edges of the test specimens (fabric) on the backside were sewed to form a tube. Each specimen was turned inside out and 6mm of each end of the fabric tube was cut to remove any sewing distortion. The prepared specimens were mounted on the specimen tube. PVC tape was used around each of the cut ends of each specimen, so as to fix the spacemen on to the tube and 6mm of tube was left exposed.

The inside of the pill testing box was cleaned thoroughly so as to make it free from lint. The mounted specimens were placed inside the pill testing box and the lid was closed. Then the machine was started for 7,000 revolutions. After completion of 7000 revolutions, the machine was stopped automatically. The specimens from the box were removed and their stitches were opened. The specimens were assessed subjectively by three experts with reference to original specimen. The assessment was done according to ASTM pill grade photographic views in a standard viewing cabinet, keeping the specimens at an angle 15° to the plane of the cabinet. Photographic standard and the standard grey scale rating are shown in Fig. 3.14 and Table 3.5.

![Fig.3.14 ASTM Photographic standard for pilling resistance](image-url)
Table 3.5 Grey scale rating

<table>
<thead>
<tr>
<th>Grey Scale Rating</th>
<th>Interpretation of Pilling Resistance</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>Very Severe Pilling</td>
</tr>
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<td>2</td>
<td>Severe Pilling</td>
</tr>
<tr>
<td>3</td>
<td>Moderate Pilling</td>
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<td>4</td>
<td>Slight Pilling</td>
</tr>
<tr>
<td>5</td>
<td>No Pilling</td>
</tr>
</tbody>
</table>

3.6.9 Measurement of tensile strength

Tensile strength and breaking elongation of fabric samples were determined by using Universal Tensile Tester (Surya Systems), working on the principles of constant rate of elongation.

Fig. 3.15 Universal Tensile Tester

Technical specification of Universal Tensile Tester

- Principle : Constant rate of extension
- Pre load : 0.5Kg
- Test speed : 200 mm/min
- Load high limit : 180 kg
- Extension high limit : 200%
- Break load : 100 kg
- Grip length : 200 mm
- Testing standard : ASTM D5035
The fabric samples were also tested at low extension level i.e. 10% and 20% of the breaking extension. For this purpose, moveable jaw was set in the tensile testing machine in such a way that after 10% of breaking extension, jaw would stop automatically and come back to the original position. The load at the instant of 10% and 20% extension was noted as well as the recovery from the extension was calculated for one cycle, 100 cycles and 200 cycles.

3.6.10 Test for Thermal Conductivity

Thermal insulation value of the fabric samples were tested on Asian make ‘Thermal Conductivity Apparatus’ which follows ASTM D 1518 standard method.

![Thermal Conductivity Apparatus](Fig. 3.16)

**Fig. 3.16 Thermal Conductivity Apparatus**

**Procedure of testing**

The guard plate was switched on after setting its heater at 50°C and waited till the temperature of guard plate reached 50°C (variation +/ - 2°C). Then the hot plate was switched on and waited till the temperature reaches 51°C. Gradually the temperature dropped to 45°C. Fabric sample to be tested was cut earlier as per template provided with the machine. The sample was placed on the heater plate so as to cover the hot plate and then the template was also placed on the sample and allowed the temperature to rise to 51°C (+/- 2°C) again. The temperature started falling gradually once it reaches 51°C. At this juncture, the time taken in seconds by hot plate to decrease the temperature from 50°C to 49°C was measured by using a standard stop watch. The same procedure was repeated for other samples.
The thermal insulation value was measured in ‘Clo’ values (1 Clo = 0.155 cubic meter-K / watt). The time taken in second by hotplate to decrease the temperature from 50°C to 49°C as measured above was divided by 240 to find the Clo value. For more convenient and as a common practice in textiles, the ‘Clo’ value was converted to ‘Tog’ value by the following formula;

‘Tog’ value of textile = 0.645 × Clo value

3.6.11 Air permeability measurement

Air permeability value of the fabrics samples were measured using Asian make Air permeability tester.

![Fig. 3.17 Air Permeability Tester](image)

Technical specification of the machine
Test area : 4cm², 10cm²
Rota meter : 6-60 LPH at NTP, 0-900 LPH at NTP, 600-6000 LPH at NTP
              (Accuracy 3% of FSR),
Vacuum gauge : 0-24mm x 0.1mm of water column
Standard : ASTM-D-737

Fabric samples were cut from random locations using the GSM cutter and held tightly between the clamps of the machine. The adjusting knobs of the three rota meters were closed initially. The vacuum system was then started and the knob of the first rota meter was opened till the liquid in the inclined tube (manometer) reached the reading of 5. Thereafter, the knob of the second rota meter was opened till the liquid in the manometer reached up to 9. Again, the knob of the third rota meter was opened till the liquid level in the manometer reached up to 10. Without switching off the vacuum system, the readings in the three Rota meters were recorded individually and air permeability value was calculated from the following formula,

Air Permeability R = \( r \times 1000 / (60 \times 60A) = 1000 \times r / 3600A \) cc/sec/cm²
Where \( r \) = Rotameter reading in litre per hour and \( A \) = Area of cross section of ring in cm\(^2\), which is 4 in the present study.

### 3.6.12 Determination of crease recovery angle

Crease recovery property of fabric is measured in terms of crease recovery angle. The crease recovery angle of untreated and treated fabric samples were measured in Asian Make Crease Recovery tester following IS 4681:1981 standard of testing of textiles.

![Fig. 3.18 Crease recovery tester](image)

The specimen of size 15mm x 40mm were folded in half and placed under load of 1 kg and kept for 5 minutes. After removal of load, the crease recovery angle was measured.

### 3.6.13 Test for water absorbency

Water absorbency property of the fabric samples was determined with the help of Asian make Spray Tester following IS: 390 method of testing.

![Fig. 3.19 Spray tester and AATCC standard spray test rating chart](image)
The Spray tester consists of a stand for holding the spray device. The button portion has a sample holder at 45 degree angle, whose centre lies at the centre line of the spray device. Spray device is made up of plastic laboratory funnel which is connected to the spray nozzle by rubber tube. Fabric sample of 230 mm diameter were taken and conditioned in normal room temperature for period of 24 hours. The specimen was mounted in the specimen holder with face upward. The warp threads direction of the fabrics was maintained toward the clamping side of the ring. The sample holder was put on the stand properly. A coloured liquid mixed with 250 ml of distilled water was poured from the sides quickly into the funnel and allowed it to spray on the specimen. The specimen holder was removed from the stand when the spraying has ceased. The lowest point of the holder was tapped three times in succession against a horizontal surface. Immediately after the tapping, comparison was made under reflected light of the wetting of the face of the specimen with the photographic standard of spray test ratings and rated to which its area of wetting best approximates.

3.6.14 Test for perspiration fastness

Perspiration fastness property of the fabric samples was determined with the help of Asian make Perspirometer following standard IS 971-1983.

![Perspirometer and Hot air oven](image.png)

Fig. 3.20 Perspirometer and Hot air oven

Specimens of the textile in contact with adjacent fabrics are treated in two different solutions of acidic and alkaline and placed between two plates under a specified pressure in testing device. These specimens and the adjacent fabrics are dried separately. The changes in colour of each specimen are accessed with the help of gray scale. Alkaline solutions were prepared by taking 200 ml of distilled water, 0.1gram of L-histidine monohydrochloride monohydrate, 1 gram of sodium chloride, one gram of disodium hydrogen
ortho phosphate dodecahydrate. This solution was maintained at a pH of 8 with 0.1 N sodium hydroxide solution. Similarly Acidic solution was prepared by taking 200 ml of distilled water, 0.1 gram of 1- histidine monohydrochloride monohydrate, 1 gram of sodium chloride, 0.44 gram of sodium dihydrogen orthophosphate dehydrates. This solution was maintained at a pH of 5.5 with 0.1 N acetic acid solutions.

White cotton fabric was taken to measure the colour staining due to perspiration on it. The white cotton fabrics were sewed on the samples. Then the composite specimens (white fabric along with the untreated and treated fabric specimen) were wet thoroughly in alkaline solution in liquor ratio of 50:1 and allowed to remain in the solution at room temperature for 15 to 30 minutes, pressed and moved from time to time to ensure good and uniform penetration of the liquor. Then, the solution was poured and wiped the excess of liquor from the specimen. The said composite specimens were placed between the two acrylic plates and all the plates (21 plates) were placed into the machine units regardless of number of specimens. Then the upper steel plate was placed over it and dead wet of 5 kg were kept on the top of the plates and the pressure plate was locked in position by turning the thumbscrews. The unit was left for 24 hours in the testing room at normal atmosphere. Next day, the weight was removed from the unit and the total unit as such was placed in oven for 4 hours at 42°C +/- 1°C. Then, the composite specimens were opened out by breaking the stitching on all sides except one of the stitched side and dried by hanging it in air at temperature not exceeding 60°C, with 3 parts in contact only at the remaining line of stitching. Assessment of fading of colour on white fabric was done by using gray scale.

![Grey scale and evaluation chart](image)

Fig. 3.21 Grey scale and evaluation chart
3.6.14.1 Study on effect of human perspiration on treated samples
Effect of human perspiration on treated samples was also studied by sewing the treated samples on garments. White cotton fabrics were sewed on treated specimen samples in order to measure the colour staining due to perspiration on it. Then the composites i.e. (stitched white fabric along with the treated fabric specimen) were attached to the garment in such a way that the specimen were in contact to the human body part where maximum sweating occurs.

Fig. 3.22 Perspiration sample attached to garment
The garments were then put on by subject for a period of 18 hours during hot day time without washing. After that the specimen and the white cotton fabric was removed from the garment and evaluation was done in the same procedure as detailed above.

3.7 Development of bio-softener and antimicrobial agents from leaf extracts of

*Excoecaria agallocha*, a mangrove plant for treatment of cotton handloom fabrics

3.7.1 Collection of plant materials and preparation of extracts
Systematic position of the mangrove plants

- Biological name : *Exocoecria agallocha L.*
- Division : *Spermatophyta*
- Subdivision : *Angiospermae*
- Class : *Dicotyledonae*
- Subclass : *Gamopetalae*
- Series : *Curvembyae*
- Order : *Euphorbiales*
- Family : *Uphorbiaceae*
- Genus : *Excoecaria*
- Species : *agallocha*
Leaves from mangrove plants, *Exocoecaria agallocha* L. were collected from coastal district of Odisha state (India). The leaves of the plants were dried for 15 days and then made into fine leaf powder using a grinder.

![Mangrove plants in the coastal area of Odisha State, India](image)

**3.7.2 Preparation of leaf extracts**

Two types of leaf extracts viz. methanol extract and aqueous extract were prepared for the experimental purpose. Two conical flasks of 250 ml capacity were taken. Ten grams each of dried leaf powder was added in to both the flasks. 100 ml of methanol was added to one of the flask and the other with 100 ml of only water. The mouth of both of the conical flasks were covered with aluminum foil and kept on orbital shaker for 48 h running at 100 rpm speed (Fig. 3.24A).

After 48 h, both the flasks were removed from the orbital shaker. The solutions were then filtered using gauge cloth, kept in two different beakers of 250 ml capacity and both were dried inside the oven (Fig. 3.24.B) at 30-50°C in order to obtain the methanol and aqueous extracts of mangrove leaves. The dried powdered extracts were scrapped (Fig. 3.24C) and stored for further use (Fig. 3.24D). The yield percentage in both the cases was also calculated by considering the actual weight of extract obtained from the weight of leaf powder taken during extraction.
3.7.3 Preparation of bio-softener (mangrove leaves extract) and procedure of treatment of cotton handloom fabrics

Bio-softener of different concentrations (10 g/l, 20 g/l, 30 g/l, 40 g/l and 50 g/l) were prepared by taking leaf extracts. The extracts of above concentrations were dissolved in water taken in a beaker, stirred for 10 min at a temperature of 60° C and allowed to settle. Then 0.6 ml each of lemon grass oil (concentration 5 ml/l) and vinegar (concentration 5 ml/l) were added to the beaker and mixed properly by vigorous stirring for 5 min at the same temperature. The bio-softener was then filtered and stored for further treatment with fabrics. Handloom fabric samples were dipped into the above solution and allowed to soak for 45 minutes at room temperature. Then the fabrics were padded at 1kg/cm² pressure in an automatic padding mangle machine to achieve at least 80% expression in order to get optimum pick up of 0.8 on the weight of material. Then the fabrics were dried at 100° C for 5 minutes and cured at 150° C for 3 minutes in drying and curing chamber. The fabrics were then taken out from the curing chamber, dried at room temperature and ironed.
3.8 Testing methods for evaluation of stiffness, softness/feel and surface roughness of handloom fabric samples treated with bio-softener

The handloom fabrics treated with bio-softener were subjected to the following tests in order to compare the improvement in mechanical and comfort properties with respect to silicone treated fabrics.

A. Stiffness in terms of bending length in cm using stiffness tester as per the procedure mentioned in 3.6.2.

B. Softness or feel in terms of pulling force in gms using Fabric Feel tester as per the procedure detailed in 3.6.3.

C. Surface roughness in terms of roughness index by Digital Image Processing method as described in 3.6.4.

3.9 Treatment procedure of mangrove leaves extract as antimicrobial agent with handloom fabrics and its evaluation methods.

3.9.1 Procedure of treatment of methanol and aqueous mangrove leaf extract on handloom fabrics for developing antimicrobial property

From each type of handloom fabric samples, thirty two small fabric pieces of size 10 mm diameter were cut, out of which eight pieces were treated with silicone finish of 40 g/l concentration, as per the procedure of treatment mentioned in 3.5.1. For convenience, these samples were coded as follows,

F = Control fabric/untreated (8 Nos.)
S = Fabric samples treated with silicone softener (8 Nos.)
F + E = Control fabric samples to be treated with mangrove leave extract (8 Nos.)
S + E = Silicone treated fabric samples to be treated with mangrove leave extract (8 Nos.)

Solutions were prepared in two separate test tubes by mixing 50 mg of methanol extract with 1 ml of water in one test tube and 50 mg of aqueous extract with 1 ml of water in another. The solution was left for half an hour. In a similar way solutions were prepared in four sets of test tubes. Then the solutions were poured in to separate beakers.

The above four types of fabric samples were dipped in to the above prepared methanol extract solution as well as aqueous extract solution separately in the beakers and kept for 5 min so as to
absorb the solution. The samples were then taken out from the beaker and dried in hot air oven at 60°C for 5 minutes. Then they were taken out from the hot air oven and kept ready for treatment with pathogenic stains.

The preparation of pathogenic strains for treatment with the prepared fabric samples involves the following steps:

- Preparation of flasks

100 ml of water was taken in conical flask. 3.1 gms of nutrient agar was added to the flask (Fig. 3.25A). It was slightly heated for complete dissolution of nutrient agar in the water.

- Sterilization

Flask containing culture medium along with Petri plates, pipettes, beakers, forceps, cotton swabs and test-tubes were wrapped with paper tied with thread and sterilized in autoclave at 15 lb. pressure and 121°C for 15 minutes. Eight Petri plates were then kept in oven for drying. Then all the above mentioned equipments were kept in laminar air flow (Fig. 3.25.B).

![Fig. 3.25 Preparation of nutrient agar flasks and sterilization](image)

**Inoculation**

Each Petri dish was filled up to half with nutrient agar. The agar medium was then allowed to solidify. Inoculation was done in totally sterile conditions in laminar flow. The following pathogenic bacterial strains were used during this study.

1. *Staphylococcus aureus* (MTCC 1144, Gram +ve)
2. *Shigella flexneri* (Lab isolated, Gram -ve)
3. *Bacillus licheniformis* (MTCC 7425, Gram +ve)
4. *Escherchia coli* (MTCC 1089, Gram -ve)
MTCC strains were obtained from the Microbial Type Culture Collection and Gene Bank (MTCC), Institute of Microbial Technology (IMTECH), Chandigarh, Punjab state (India) and Lab isolates were isolated from laboratory of College of Engineering and Technology, Bhubaneswar, Odisha state (India).

100 micro litres of each type of the above pathogenic strains contained in test tubes were added to each of the eight Petri dishes (four Petri dishes for methanol extract treated fabrics and another four Petri dishes for aqueous extract treated fabrics) and spread all over by means of cotton swabs. Then the above prepared fabric samples were placed in each petri plate and labelled by means of sterilized forceps.

**Incubation**

The incubated Petri dishes were then kept in an incubator at 37°C for 24 h. After incubation for 24 h, the anti-microbial activity was evaluated by measuring their developed ‘zone of inhibition’.

Similar process was repeated for assessing antimicrobial efficiency due to treatment with mangrove leaf extract (methanol and aqueous) for all the five types of handloom fabrics.

**3.10 Evaluation for bacterial growth of antimicrobial finish treated fabrics towards repeated washing**

The garments are usually subjected to repeated washes. If the bioactive antimicrobial finishes in fabrics do not last for a good number of washes, then this type of finishes are not of much use for garments. Hence, washing fastness property of the fabrics treated with the antimicrobial finishes was also studied.

The samples (cotton handloom fabrics) were treated with solution of methanol leaf extract as well as with aqueous leaf extract in the same way as mentioned above and were dried in hot air oven. Treated samples were washed in a laundry-o-meter for 5 min, 10 min, 15 min and 20 min cycle respectively. Samples were collected after wash, dried and then placed on the agar plates containing the above four types of pathogenic bacterial strains as prepared in the same manner as described above. The anti-microbial activity of these fabrics was evaluated by the ‘Agar diffusion test method’ (measuring the developed zone of inhibition).