Chapter 3

Experimental
3.1 Materials

3.1.1 Fabrics
Conventional H\textsubscript{2}O\textsubscript{2} bleached plain weave jute fabrics having 63 ends/dm, 59 picks/dm, 220 g/m\textsuperscript{2} (area density), 195 tex warp, 214 tex weft, and 0.80 mm thickness were used for the present study.

3.1.2 Chemicals

**Fire Retardant Chemicals**
Laboratory reagent grade Diammonium phosphate [(NH\textsubscript{4})\textsubscript{2}HPO\textsubscript{4}], Borax(Na\textsubscript{2}B\textsubscript{4}O\textsubscript{7}), Boric acid(H\textsubscript{3}BO\textsubscript{3}), Sodium perborate(NaBO\textsubscript{2},4H\textsubscript{2}O), Tetra-sodium-pyro-phosphate(Na\textsubscript{4}P\textsubscript{2}O\textsubscript{7}), Magnesium chloride(\text{MgCl}_2,6H\textsubscript{2}O), Urea(CH\textsubscript{4}N\textsubscript{2}O), Ortho-Phosphoric acid,H\textsubscript{3}PO\textsubscript{4} (85%) , Thio-urea(CH\textsubscript{4}N\textsubscript{2}S ) ,Dicyandiamide(C\textsubscript{2}H\textsubscript{4}N\textsubscript{4}), Ammonium Sulphate [(NH\textsubscript{4})\textsubscript{2}SO\textsubscript{4}] obtained from E Merck ,India, were used. Laboratory reagent grade Sodium Stannate(Na\textsubscript{2}SnO\textsubscript{3},3H\textsubscript{2}O) of Loba Chemie Pvt.Ltd.,Mumbai was used.

Commercially available ready flame retardant formulations like Pekoflam DPN((Organo-Phosphorous Compound) from Clariant, India, Pyrovatex CP[ Huntsman, Mumbai,(old Ciba)], were also used. Commercially available Finish-KVS (DMDHEU i. e. dimethylol-di-hydroxy ethylene urea) resin from Clariant ,India were also used.

**Rot Resistant Chemicals**
Laboratory reagent grade Citric acid(C\textsubscript{6}H\textsubscript{8}O\textsubscript{7},H\textsubscript{2}O) as crosslinking agent, Sodium hypophosphite monohydrate (NaH\textsubscript{2}PO\textsubscript{2},H\textsubscript{2}O) as catalyst and Poly ethylene glycol 400 obtained from E Merck ,India, were used. Chitosan powder from shrimp shells having 86% degree of deacetylation obtained from Sigma Aldrich Chemicals Co.USA ( Product Number C3646) was also used.

3.2 Methods

3.2.1. Fire-retardancy:

(a) **Fire retardant finishing treatment by single FR compound (Boron/Sulphur/Nitrogen/Phosphorous): Pad-Dry-Cure.**

Bleached jute fabric was padded(100% expression with 2 dip 2nip system) in a solution containing fire retardant chemicals dissolved in water. The padded fabric was then dried at
100°C for 10 min followed by curing at 150°C for 5 minutes. Finally, the fabric was washed and
dried to obtain final phosphorous or nitrogen based or other FR chemical treated/finished jute
fabric.

(b) Fire retardant treatment by different formulations having two or more FR compound in presence of crosslinking agent

Bleached jute fabrics were padded (100% weight pick-up by 2 dip 2 nip process) with the
following flame retardant formulations (Formulation 1 to Formulation 6) as given below
followed by drying at 100°C for 10 min and curing at 150°C for 5 minutes and then finally
washed and dried.

<table>
<thead>
<tr>
<th>Formulation-1</th>
<th>Formulation-2</th>
<th>Formulation-3</th>
</tr>
</thead>
<tbody>
<tr>
<td>DAP- 4%, 8%, 12%, 16%</td>
<td>TSPP-4%, 8%, 12%, 16%</td>
<td>Borax + Boric Acid (7:3) 4%, 8%, 12%, 16%</td>
</tr>
<tr>
<td>(Diammonium phosphate)</td>
<td>(Tetra-sodium pyrophosphate)</td>
<td></td>
</tr>
<tr>
<td>Borax + Boric acid (7:3)-12%</td>
<td>DAP- 4%</td>
<td>TSPP-12%</td>
</tr>
<tr>
<td>DMDHEU-4%(active)</td>
<td>DMDHEU-4%(active)</td>
<td>DMDHEU-4%(active)</td>
</tr>
<tr>
<td>MgCl₂ catalyst-1%</td>
<td>MgCl₂ catalyst-1%</td>
<td>MgCl₂ catalyst-1%</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>Formulation-4</th>
<th>Formulation-5</th>
<th>Formulation-6</th>
</tr>
</thead>
<tbody>
<tr>
<td>Sodium Perborate- 4%, 8%, 12%, 16%</td>
<td>Borax + Boric acid (7:3)- 4%, 8%, 12%, 16%</td>
<td>Pekoflam DPN-4%, 8%, 12%, 16%</td>
</tr>
<tr>
<td>DAP-4%</td>
<td>Sodium Perborate-12%</td>
<td>DAP-4%</td>
</tr>
<tr>
<td>DMDHEU-4%(active)</td>
<td>DAP-4%</td>
<td>DMDHEU-4%(active)</td>
</tr>
<tr>
<td>MgCl₂ catalyst-1%</td>
<td>DMDHEU-4%(active)</td>
<td>MgCl₂ catalyst-1%</td>
</tr>
<tr>
<td></td>
<td>MgCl₂ catalyst-1%</td>
<td></td>
</tr>
</tbody>
</table>
(c) Flame Retardant Treatments with Nitrogen and Phosphorous based Formulations

Bleached jute fabrics were padded (100% weight pick-up by 2 dip 2 nip process) with following flame retardant formulations as given below followed by drying at 100 °C for 10 min and curing at 150°C for 5 minutes and then finally washed and dried.

<table>
<thead>
<tr>
<th>Formulation-7</th>
<th>Formulation-8</th>
<th>Formulation-9</th>
</tr>
</thead>
<tbody>
<tr>
<td>Urea-10%, 20%</td>
<td>Urea-20%</td>
<td>Thiourea-8%, 10%</td>
</tr>
<tr>
<td>OrthoPhosphoricAcid-4%, 6%, 8%, 10%</td>
<td>Di ammonium Phosphate-5%, 10%, 15%, 20%</td>
<td>Di ammonium Phosphate-5%, 10%, 15%</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>Formulation-10</th>
<th>Formulation-11</th>
</tr>
</thead>
<tbody>
<tr>
<td>Dicyandiamide-8%, 10%</td>
<td>PyrovatexCP-10%, 15%, 20%, 25%, 30%</td>
</tr>
<tr>
<td>Di ammonium Phosphate-5%, 10%, 15%</td>
<td>DMDHEU-8% (solid add-on)</td>
</tr>
<tr>
<td>Ortho-PhosphoricAcid-2%</td>
<td></td>
</tr>
</tbody>
</table>

(d) Fire-retardant treatment using Stannic compound:

Sodium Stannate Treatment: - Pad-Dry-Pad-Dry

Bleached jute fabric was padded (100% weight pick-up by 2 dip 2 nip process) with aqueous solution of Sodium Stannate and dried at 100 °C for 10 min. The dried fabric was further padded (100% weight pick-up by 2 dip 2 nip process) by ammonium sulphate solution and finally dried at 100 °C for 10 min.
Stannate -Phosphate Process: An additional treatment with Di ammonium Phosphate and urea was done by pad, dry, cure method on stannate treated fabric (stannate followed by ammonium sulfate treatment).

3.2.2 Rot resistant treatments

**Treatment 1-Citric Acid treatment**

Bleached jute fabrics were padded (100% wet pick-up by 2 dip 2 nip process) in a solution containing Citric Acid(6% to 12%), Sodium hypophosphite monohydrate (6%) and wetting agent (0.1%) followed by drying at 100 °C for 10 min and curing at 150°C for 5 min. The cured fabric was finally washed and dried.

**Treatment 2-Citric Acid and Poly ethylene Glycol treatment**

In another experiment, bleached jute fabric was also padded (100% wet pick-up) with 10% Citric acid, Poly ethylene Glycol of molecular weight 400 (2% to 12%), Sodium hypophosphite monohydrate (6%) and wetting agent (0.1%) followed by drying at 100 °C for 10 min and curing at 150°C for 5 min. The cured fabric was finally washed and dried.

**Treatment 3-Chitosan treatment**

The chitosan solution was prepared by stirring a dispersion of chitosan (1.0g) in 2.0% (v/v) aqueous acetic acid solution (100 ml). Bleached Jute fabric was padded(100% wet pick-up) with the chitosan solution and dried at 100 °C for 10 min. followed by curing at 150°C for 5 min. The chitosan treated fabric was finally washed and dried.

**Treatment 4-Citric Acid and Chitosan treatment**

In another experiment, bleached jute fabric was also padded (100% wet pick-up) with 10% Citric acid, Chitosan (0.25% to 1.0%), Sodium hypophosphite monohydrate (6%) and wetting agent (0.1%) followed by drying at 100 °C for 10 min and curing at 150°C for 5 min. The cured fabric was finally washed and dried.

3.2.3 Testing Methods

**(a) Flammability measurement:** The flammability of the fabric was assessed by 45° inclined Flammability test for a specified ignition time (for cotton 1 sec, for jute 10 sec.)as per ASTM-D-1230-94 and Limiting Oxygen Index(LOI)-test as per ASTM-D-2863-77.
**45° inclined Flammability Test**: Selected untreated and treated jute fabric samples of specified length (15cm length and 5cm wide) were mounted in the specific sample holder and placed in 450 angle specimen holder and then was exposed to a standard flame for a specified ignition time (for cotton it is 1sec, for jute it is 10 sec, standardized in this laboratory) and was allowed to burn in a position of the inclined plane (45° inclined) in Standard flammability tester (Make: Paramount, India,) following ASTM-D-1230-94 standard method\(^1\). The fabric samples was mounted at 45° angle in a specimen holder and was then exposed to a standard flame of specified height exposed at 90° with the specimen for 10 second and is left for burning, to note flame spread time, afterglow time, and char length (in cm) after the burning. The time taken (\(t\) in seconds) for the flame to travel 12.5cm of the fabric sample mounted in the frame at 45° angle for the fabric of specified length and width to burn was recorded on digital timer provided in the instrument. After glow time was measured with a stop watch and char length was physically measured by a scale.

**Determination of Limiting Oxygen Index**

Limiting Oxygen Index (LOI) is the critical oxygen index value indicating the relative measure of flammability of any materials or textiles. If LOI values are above certain critical limit (say 27\(^1\)), there is hardly any chance of fire propagation. LOI testing instrument thus provides a precise method for determining the critical oxygen index of the sample by measuring the minimum volume concentration of oxygen gas in a flowing stream of mixture of oxygen and nitrogen gases (mixed in different volume ratio) required to maintain candle like burning of a sample for a specified time.

LOI values of selected untreated and treated jute fabric samples were determined by a standard LOI tester (Make: S C Dey & Co., Kolkata) as per ASTM-D-2863-77 method\(^2\) by using the following formula.

\[
\text{Limiting Oxygen Index (n) = } \frac{100 \times \text{Volume concentration of } O_2}{\text{Volume concentration of } N_2 + \text{Volume concentration of } O_2}
\]
(b) Rot resistance

Rot resistance i.e. resistance to microbial attack of the fabric samples was assessed by determining the % retention of tensile strength after subjecting the fabric to a standard soil burial test for 21-days as per IS:1623:19603,4.

For the soil burial test, a composted soil consisting of a thorough mixture of fertile garden soil, cow dung and sand in 2:1:1 weight proportions was used maintaining a moisture content of the composted soil at around 25-27%. The soil burial test for the yarn samples was conducted for 21 days using the composted soil ambient temperature (30 ± 2°C) in the pot. At the end of the specified periods, the fabric specimens were taken out, washed gently in plain water, kept covered in a beaker containing ethyl alcohol for two hours and finally dried in air. The tensile strength of jute fabric samples was evaluated before and after the soil burial process following a standard method of tensile strength testing.

(c) Measurement of Bending Length

The bending length of the selected fabric samples was measured as per IS-6490-1971 method5 using Cantilever type Sasmira fabric stiffness tester with a specimen size of 200 mm × 25 mm.

In this test, the rectangular strip is extended beyond the edge of the platform of the stiffness tester and the free edge of the fabric is allowed to bend under its own weight until the free edge makes an angle of 41.5⁰ (already marked in the instrument) with the horizontal platform. The bending length was assessed from the observed length of the fabric required to bend to a particular angle. Higher the bending length, stiffer is the fabric. The test results reported are an average of 5 tests in each case.

(d) Measurement of Crease recovery Performances

Dry Crease recovery angle (warp +weft) of selected fabric samples were measured by the Sasmira crease recovery tester in accordance with ASTM-D-1295-67.

The total crease recovery angle (CRA) of treated and untreated fabric samples (warp-way and weft-way) was assessed as per ASTM-D-1295-67 (1972) method 6 with 5 min loading and 5 min recovery time using a Sasmira Crease Recovery Tester. Fabric samples having a specimen size of 4.0 x 1.5 cm were cut and each cut fabric piece was folded midway along the length and
pressed with 500 g load (pressure of 167 g/cm²) between two plastic plates for 5 min. the pressed specimen was then transferred to the crease recovery tester and the angle recovered by the folded specimen after 5 min. of unloading was measured. The total crease recovery was then calculated by adding both warp-way and weft-way crease recovery angle for each sample. The test results reported are an average of 5 tests for each sample.

(e) Measurement of Tensile Properties

Tensile strength of selected fabric samples were measured by the raveled strip method as per IS-1969-1968 method using an Instron (Model-1445) CRT-Universal tensile tester with a traverse speed of 100 mm/min and a pretension of 0.5 N. The final gauge length(sample size) of the fabric sample was 50 mm. x 20 mm. under the jaws.

Warp-way breaking tenacity (cN/tex) of selected fabric samples were measured by the ravelled strip method as per IS-1969-1968 method¹⁷ using an Instron (Model-1445) CRT-Universal tensile tester with a traverse speed of 100 mm/min and a pretension of 0.5 N. The final gauge length(sample size) of the fabric sample was 50 mm.x 20 mm. after raveling.

(f) Measurement of Whiteness

Whiteness index as per Hunter Lab-Scale formula,⁷ of the selected jute fabric samples were directly evaluated using a computer aided Macbeth 2020 plus reflectance spectrophotometer (with D<sub>65</sub> standard illuminant and 10° standard observer setting) and associated colour measurement software,using following relationship.

\[
\text{Whiteness Index (Hunter Lab-Scale)} = \frac{L - 3b}{\sqrt{Y}} = \frac{10V_Y - \left[21(Y-Z \%)\right]}{\sqrt{Y}}
\]

where, X, Y and Z are the tristimulus values of the sample, L is the lightness/darkness indicator in CIE Lab-Scale [L* or simply \(L = 16 \left(\frac{Y}{Y_0}\right)^{1/3} - 16\) as per CIE Lab-1976 formula ⁸], b is the blueness/yellowness indicator in the CIE Lab-Scale[^8] [b* or simply \(b = 200 \left(\frac{Y}{Y_0}\right)^{1/3} - \left(\frac{Z}{Z_0}\right)^{1/3}\)] , B = Z/1.181 = 0.847 Z, G = Y = L<sup>2</sup>/100 and \(X_0\), \(Y_0\) and \(Z_0\) are the CIE-tristimulus values[^16] for D65 standard illuminant and 10° standard observer.

(g) Study of Thermal Behaviour by TGA and DSC Thermograms

Differential Scanning Calorimetric(DSC) thermogram indicate different thermal transitions and thermal decomposition temperatures of the constituting materials of the polymer fibre sample in certain temperature range . In Thermo-Gravimetric Analyser (TGA), with constant heating from ambient to any limiting temperature range, the change in sample weight due to thermal
degradation, evaporation, dehydration is measured. This technique is effective for quantitative analysis of thermal degradation at any or at use temperature, rate and type reactions that are accompanied by mass change due to thermal decomposition, evaporation, gas adsorption and dehydration etc. The TGA and DSC thermograms of jute not only give an indication of its thermal behaviour, but also give a clear reflection regarding the degree of chemical changes/interaction of the major constituents of jute after chemical treatments and modifications, altering the rate of mass loss on heating (in TGA) and also showing relative thermal changes in degradation temperatures (in DSC) for major jute constituents.

TGA and DSC thermograms of untreated and treated jute fibre samples (finely crushed) after being taken out from the corresponding untreated and treated jute fabrics were obtained from a Shimadzu Thermo-Gravimetric Analyser (Model - TGA-50) and Shimadzu Differential Scanning Calorimeter (Model DSC-50) under atmospheric air (for TGA) and under flowing nitrogen (for DSC at nitrogen flow rate of 50 cm³/min) respectively at a heating rate of 10°C/min, using a pre-fixed sample weight of exactly 2 mg over a temperature range from 30°C (ambient) to 500°C, following usual procedure 9,10.

(h) Scanning Electron Microscopy (SEM) Surface morphology of untreated and treated jute fibre samples taken out from the corresponding raw and treated fabrics were examined according to the prescribed procedures using a scanning electron microscope (Model: Jeol - JSM-5200 Scanning Electron Microscope). Jute fabric samples or jute fibre samples taken out from the relevant fabric sample were mounted on a specimen stub with double sided adhesive tape and then subjected to coating with gold-palladium alloy using a sputter coater to avoid charging of the specimen 11,12. The observations were made at an operating voltage of 20 KV using usual magnification of 1000, or using a higher or lower magnification wherever specifically required.

(i) Fourier Transform Infrared Spectroscopy (FTIR)

Selected jute fibre (finely crushed) samples (3mg) taken out from untreated and treated fabrics were examined in a double beam FTIR spectrophotometer (BOMEM, MB 104) using KBr disc technique 12,20,21,22.
References