CHAPTER-3

MATERIALS AND METHODS OF TREATMENT

3.1. Overview of experiment

In order to achieve the objectives described in chapter one, investigations were carried out in Wood Preservation Discipline, Forest Products Division, Forest Research Institute, Dehradun. An overview of the experiments carried out is given below and schematic representation of the same is given in fig.3.1.

Treatments carried out for present study on radiata pine wood (Table 3.1) are:

1) **Unified thermal and neem seed oil treatment** (UTNSO): Where, thermal treatment to the specimens at different temperatures and durations was given followed by dipping treatment with neem seed oil.

2) **Thermal treatments** (HT): Where, specimens were heated at different temperatures and durations.

3) **Neem seed oil treatment** (DNSO): It was given by dipping/soaking method to specimens for 24 h.

4) **Linseed oil treatment** (DLSO): It was given by dipping method for 24 h to separate set to determine its contribution towards the modification.

5) **Untreated control** (C): It was maintained to compare the results.

After completion of above mentioned treatments, treated and untreated samples were subjected to different tests for evaluation of changes in physical, chemical and biological properties of wood. Weight gain in wood samples treated with UTNSO (Section 3.3.3.4), DNSO (Section 3.3.3.5) and DLSO (Section 3.3.3.6) was determined. Simultaneously, weight loss (Section 3.3.3.3.) and density loss (Section 4.2.3.2) due to
the HT treatment was also studied. Change in colour of wood (Section 4.2.3.1.) due to various treatments were visually observed. Durability assessment after treatment against fungi (Section 5.2.3.1) was tested by soil block bioassay method, while termite mound test was performed for termite resistance (Section 5.2.3.2). Hygroscopicity of wood was determined by studying changes in the EMC at different relative humidities (Section 4.2.3.3.1). Study of dimensional stability was conducted by swelling coefficient at different relative humidities and on water submersion (Section 4.2.3.4). Difference in water absorption behaviour was also studied (Section 4.2.3.3.2). Chemical analysis (Section 6.2.3.) of wood was used to find out changes in chemical composition during thermal modification of wood. Changes in pH due to thermal treatment were also studied (Section 6.2.4.). The data was analysed using SPSS (version 16) software package.

Table 3.1 Details of treatments performed on Pinus radiata wood samples

<table>
<thead>
<tr>
<th>S. no</th>
<th>Treatment</th>
<th>Temperature of heat treatment(°C)</th>
<th>Time of heat treatment (min)</th>
<th>Time of dipping (h)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>Unified thermal and neem seed oil (UTNSO)</td>
<td>180</td>
<td>30</td>
<td>24</td>
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<tr>
<td></td>
<td></td>
<td>180</td>
<td>60</td>
<td>24</td>
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<tr>
<td></td>
<td></td>
<td>180</td>
<td>90</td>
<td>24</td>
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<td>24</td>
</tr>
<tr>
<td></td>
<td></td>
<td>220</td>
<td>90</td>
<td>24</td>
</tr>
<tr>
<td>2</td>
<td>Thermal/Heat (HT)</td>
<td>180</td>
<td>30</td>
<td>-</td>
</tr>
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<td></td>
<td></td>
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<td>60</td>
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<tr>
<td></td>
<td></td>
<td>180</td>
<td>90</td>
<td>-</td>
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<td>200</td>
<td>30</td>
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<td></td>
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<td></td>
<td></td>
<td>220</td>
<td>90</td>
<td>-</td>
</tr>
<tr>
<td>3</td>
<td>Dipping in neem seed oil (DNSO)</td>
<td>-</td>
<td>-</td>
<td>24</td>
</tr>
<tr>
<td>4</td>
<td>Dipping in linseed oil (DLSO)</td>
<td>-</td>
<td>-</td>
<td>24</td>
</tr>
<tr>
<td>5</td>
<td>Untreated control (C)</td>
<td>-</td>
<td>-</td>
<td>-</td>
</tr>
</tbody>
</table>
Materials and Methods of treatment

**Procurement of Pinus radiata log sawing into planks (3.2.1.)**

**Seasoning and conversion (3.2.1)**

**Sanding, cleaning and selection of defect free samples (3.3.1.)**

**Conditioning and determination of moisture content (3.3.1.)**

**Fig.3.1 Schematic structure of course of the experiment methods applied.**

**Procurement of Pinus radiata log**

**Sawing into planks**

**Seasoning and conversion**

**Sanding, cleaning and selection of defect free samples**

**Conditioning and determination of moisture content**

**Treatments (3.3.3)**

- **UTNSO (3.3.4)**
- **DNSO (3.3.5)**
- **DLSO (3.3.6)**
- **HT (3.3.3)**
- **Control**

**Evaluation of modified and unmodified specimens various properties (chapter 4, 5 and 6)**

**Methodologies adopted**

- **Soil block bioassay (O. placentus & T. versicolor)**
- **Termite mound test**
- **Conditioning at 35, 65 & 98 % Relative humidity**
- **Water immersion test**
- **OD weight/ OD vol.**
- **Water submersion 24h**

**Chemical changes (chapter 6)**

- **pH (6.2.4)**
- **Chemical Analysis (6.2.3)**

**Chemical Analysis**

- **Cellulose**
- **Hemicellulose**
- **Lignin**
- **Extractives**

**FTG**

**FTG (4.2.3.2)**

**Chemical Analysis**

**Chapter 6**

**Water submersion**

24h
3.2 Materials

3.2.1 Procurement of materials and conversion

Logs of *Pinus radiata* D. Don of New Zealand origin were procured from Green Gold Timbers Pvt. Ltd. Dehradun. Logs were sawn into planks (Plate-3.1-3.2) at saw mill of Forest Research Institute, Dehradun, Uttarakhand (India). Seasoning of planks was carried out in seasoning kiln in electrically heated kiln of Wood Seasoning Discipline, Forest Research Institute, Dehradun. Seasoned planks were converted into desired dimensions as per the requirement of different tests (Section 3.3.1.). Neem (*Azadirachta indica* A. Juss.) seed and linseed (*Linum usitatissimum* L.) oils were procured form local market of Dehradun. The mechanically extracted oil was taken in view of industrial applications.

3.3 Methods

3.3.1 Sample preparation

The test blocks of different sizes were prepared from the sapwood of radiata pine to study different parameters (Plate 3.3-3.7). Blocks were made from seasoned planks, free from knots, mould, stain and any other defects. A total of 1790 test samples were selected to study different parameters. The detail of sample size and number is given in Table 3.2. Sanding of selected samples was done firstly with 80 grit size and than by 120 grit size sand papers. After sanding all samples were conditioned at 65% RH and 25ºC temperature in desiccators over saturated salt solution of sodium nitrite (NaNO₂) (Plate-3.8), except samples meant for determination of weight loss on oven dry basis which were kept in desiccators only after determination of oven dry weight ($W_1$) with the help of an electronic weighing balance (Make: Citizen) after heating in an hot air electric oven (Make: Shamboo scientific glass work) at 103±2ºC till constant weight. Moisture content (MC) was determined by using following equation.

$$MC = \frac{W_2 - W_1}{W_1} \times 100$$
Where  
$W_1$ is initial oven dry weight  
$W_2$ is constant weight after conditioning.

Table 3.2 Size and number of samples for different parameters of study

<table>
<thead>
<tr>
<th>S. no</th>
<th>Parameter under study</th>
<th>Sample size (mm) used</th>
<th>Number of samples (replicates × no. of treatments)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>Weight loss on oven dry basis</td>
<td>20×20×20(LTR)</td>
<td>90(10×9)</td>
</tr>
<tr>
<td>2</td>
<td>Weight gain</td>
<td>60×20×20(LTR)</td>
<td>110(10×11)</td>
</tr>
<tr>
<td>3</td>
<td>Equilibrium moisture content (EMC)</td>
<td>60×20×20(LTR)</td>
<td>126(6×21)</td>
</tr>
<tr>
<td>4</td>
<td>Volumetric swelling over various EMCs</td>
<td>60×20×20(LTR)</td>
<td>126(6×21)</td>
</tr>
<tr>
<td>5</td>
<td>Radial swelling over various EMCs</td>
<td>50×20×20(RLT)</td>
<td>126(6×21)</td>
</tr>
<tr>
<td>6</td>
<td>Tangential swelling over various EMCs</td>
<td>50×20×20(TLR)</td>
<td>126(6×21)</td>
</tr>
<tr>
<td>7</td>
<td>Volumetric swelling in water submersion test</td>
<td>60×20×20(LTR)</td>
<td>126(6×21)</td>
</tr>
<tr>
<td>8</td>
<td>Radial swelling in water submersion test</td>
<td>50×20×20(RLT)</td>
<td>126(6×21)</td>
</tr>
<tr>
<td>9</td>
<td>Tangential swelling in water submersion test</td>
<td>50×20×20(TLT)</td>
<td>126(6×21)</td>
</tr>
<tr>
<td>10</td>
<td>Liquid water absorption</td>
<td>60×20×20(LTR)</td>
<td>126(6×21)</td>
</tr>
<tr>
<td>11</td>
<td>Density loss</td>
<td>60×20×20(LTR)</td>
<td>60(6×10)</td>
</tr>
<tr>
<td>12</td>
<td>Termite resistance</td>
<td>100×25×6(LTR)</td>
<td>210(10×21)</td>
</tr>
<tr>
<td>13</td>
<td>Resistance against brown rot fungi</td>
<td>19×19×19(LTR)</td>
<td>126(6×21)</td>
</tr>
<tr>
<td>14</td>
<td>Resistance against white rot fungi</td>
<td>19×19×19(LTR)</td>
<td>126(6×21)</td>
</tr>
<tr>
<td>15</td>
<td>Chemical analysis</td>
<td>60×20×20(LTR)</td>
<td>30(3×10)</td>
</tr>
<tr>
<td>16</td>
<td>pH measurement</td>
<td>60×20×20(LTR)</td>
<td>30(3×10)</td>
</tr>
<tr>
<td>17</td>
<td>Standardisation of dipping time</td>
<td>60×20×20(LTR)</td>
<td>10</td>
</tr>
</tbody>
</table>

Where: L=longitudinal; radial; T=tangential directions of samples.

Before the treatment, samples meant for studying each parameter as given in Table 3.2 were randomly divided into six groups. Group one and two were further divided into nine subgroups each. Different codes were assigned and marked on samples by permanent marker depending upon the treatments.

**Group -1:** Unified thermal and neem seed oil treatment (UTNSO)

Total of 9 sub-groups were maintained to perform the study. Samples of sub-group 1, 2 and 3 received temperature of 180°C, sub-group 4, 5 and 6 received 200°C and sub-group 7, 8 and 9 received 220°C for 30, 60 and 90 min followed by dipping in neem seed oil.
Materials and Methods of treatment

Study on unified thermal and neem seed oil treatment of Pinus radiata for durability and dimensional stability improvement (PhD Thesis Y. U. Bhoru)

Seed oil for 24 h. Total of 720 samples were subjected to treatment; each sub-group contained 80 numbers of samples for treatment.

**Group-2**: Thermal treatment under nitrogenous atmosphere (HT)

Nine sub-groups containing 92 samples each were subjected to treatment at different temperatures i.e. 180°C, 200°C and 220°C for 30, 60 and 90 min. Total 828 samples received this treatment.

**Group-3**: Neem seed oil treatment at 25% was given for 24 h (DNSO). Total 80 samples received the treatment.

**Group-4**: Linseed oil treatment through dipping for 24 h (DLSO) was received by 80 numbers of samples.

**Group-5**: Total 82 samples without any treatment served as control

**Group-6**: Total 10 samples were used for standardisation of dipping time

### 3.3.2. Standardisation of dipping time

Ten conditioned wood samples of *P. radiata*, having dimension 60×20×20 mm (LTR), were selected for the study. After recording the initial weight (*W*1), samples were placed in laboratory tray and covered with wire mesh frame with weight block to avoid floating. The tray was filled with 25% of neem seed oil diluted with linseed oil. The concentration of neem seed oil was selected on the basis of the study of Dhyani (2008) where efficacy of Neem seed oil is reported at 25%. Samples were removed at regular intervals and weighted (*W*2) for NSO retention with time. Retention of NSO was calculated using following equation (Kumar and Dev, 1993).

\[
Retention \ (R) = \frac{GC}{V} \times 10 \ \text{kg/m}^3
\]

Where,  
\( G \) = Mass of the treating solution absorbed by block (*W*1 – *W*2), in g;  
\( C \) = Mass of the preservative present in 100g of the treating solution, in g;  
\( V \) = Volume of the test block, in cm³.
3.3.3. Treatments

3.3.3.1 Heat treatment plant

Thermal treatments were carried out in fabricated thermal treatment plant (Plate-3.9-3.13) layout of which is given in Fig. 3.2. It consist of treatment vessel (1) made of mild steel (16 gage) fitted with four 1KW heating rods (2), placed at equal intervals under the insulation jacket (3), connected to temperature controller unit (11). Temperature recording sensor (4) was placed inside the treatment vessel. Vacuum gauge (G) was also fitted to treatment vessel to note the vacuum, generated by vacuum pump. Vacuum pump (5), oil storage tank (6) and Nitrogen cylinder (7) were connected to treatment vessel with the help of different valves (V₁, V₃ and V₂ respectively). Sample container made of stainless steel (20 gage) fitted with wooden tray was made to place samples at the centre of treatment vessel.

![Diagram of heat treatment plant](image)


**Fig. 3.2 Layout of heat treatment plant**
3.3.3.2 Treatment plant working

Thermal treatment plant (Plate- 3.9) was used for heat treatment of wood samples. Samples were placed on the wooden frame (9) of stainless steel vessel (8) (Plate- 3.10) and covered with wire mesh frame (Plate-3.11). Weight blocks were placed on top of wire mesh frame to avoid floating of samples during treatment. Stainless steel vessel holding samples was then charged inside treatment plant (Plate-3.12) and door was closed (Plate-3.13). Initial vacuum of 400 Hg in treatment plant was created and maintained for 15 min with the help of vacuum pump through valve V₁. After creating the vacuum valve V₁ was closed and Nitrogen was flushed in the vessel through valve V₂ connected to the nitrogen cylinder. The valve V₂ was closed subsequently to maintain nitrogenous atmosphere in the cylinder. For the thermal treatment, temperature of heat treatment plant was increased from ambient to desired operating temperature at the rate of 3°C/min. After completion of treatment schedules, temperature of treatment plant was set at 25°C (room temperature). When temperature of plant reached 25°C samples were removed from plant.

For unified thermal and neem seed oil treatment the procedure described above was opted. The thermally treated samples were left in the vessel at 25°C. It was followed by the introduction of neem seed oil through the valve V₃ which was connected with oil storage tank. The samples were left in oil for 24 h dipping.

3.3.3.3 Thermal treatment (HT)

Thermal treatment was performed by retification method (Dirol and Guyonnet, 1993 and Vernois, 2000). Samples of group-2 of section 3.3.1 of different sizes were subjected to different levels of thermal modification in thermal modification plant as described in section 3.3.3.2. Initial vacuum of 400 mm Hg was created in the thermal treatment vessel with the help of vacuum pump through valve V₁ connected to the treatment vessel. After creating the vacuum the valve V₁ was closed for maintaining the vacuum in treatment vessel. The nitrogen gas was supplied from the nitrogen cylinder through valve V₂ after flushing the nitrogen the valve V₂ was closed for providing the
nitrogen atmosphere to the samples. The vessel temperature was increased from ambient to desired operating temperature. The heat treatment was carried out at 180, 200 and 220°C for 30, 60 and 90 min.

The test samples were removed from the treatment vessel. Out of 828 samples 738 were placed in desiccators to study different parameters and rest of 90 samples of dimension 20×20×20 mm were oven dried to constant weight to study weight loss due to treatment. Weight loss was calculated on oven dry basis by using following equation:

\[
Weight \ loss \ (%) = \frac{W_1 - W_2}{W_1} \times 100
\]

Where, \( W_1 \) = oven dry weight before thermal treatment  
\( W_2 \) = oven dry weight after thermal treatment

3.3.3.4 Unified thermal and neem seed oil treatment (UTNSO)

Samples of group-1 were subjected to unified thermal and neem seed oil treatment, which is a two step process. It involves step one for thermal treatment followed by dipping in oil. In present study step one was thermal treatment under nitrogenous atmosphere at different temperatures (180, 200 and 220°C) for different time durations (30, 60 and 90 min.) and step two was dipping in neem seed oil for 24 h. For step one, thermal treatment was conducted by the method described in section 3.3.3.3. After completion of thermal treatment the heating unit was switched off and valve \( V_3 \) connecting treatment plant with oil storage tank was opened to inject neem seed oil (25% diluted with linseed oil) into steel tray holding samples. When the container holding samples inside the treatment plant was filled with oil, valve \( V_3 \) was closed and samples were allowed to remain dipped into oil for 24 h. Samples were then removed from treatment plant, wiped off with cloth to remove excess oil.

Out of 720 samples subjected to above treatment, 90 samples of dimension 60×20×20 mm (LTR) were taken to study the weight gain due to oil uptake and retention of NSO. Weight gain (%) was calculated using following equation.
Materials and Methods of treatment

\[ \text{Weight gain (\%)} = \frac{W_2 - W_1}{W_1} \times 100 \]

Where, \( W_1 \) = weight before treatment
\( W_2 \) = weight after treatment

The retention of NSO was also calculated using equation given in section 3.3.2

3.3.3.5 Dipping in neem seed oil (DNSO)

Total 80 defect free wood specimens of group-3 as given in section 3.3.1 of different dimension as given in Table 3.2 were prepared from seasoned wood of \( P. \ radiata \) as described in the same section. For volumetric swelling, EMC, weight gain, water absorption total 34 samples of dimension 60×20×20 mm, for radial and tangential swelling total 24 specimen of size 50×20×20 mm, for termite resistance test 10 samples of size 100×25×6 mm and for fungal resistance 12 sample of size 20×20×20 mm were taken. All the test samples were placed in a tray covered with wire mesh frame with weight to avoid floating. Neem seed oil 25% (diluted with linseed oil) was poured into the tray till the level of oil was high enough to allow 24 h complete soaking. The tray was kept for 24 h and then samples were removed and wiped off with cloth to remove excess oil. Weight gain due to oil absorption was recorded in ten samples of dimension 60×20×20 mm. Weight gain and retention was calculated by using equations given in section 3.3.3.4.

3.3.3.6 Dipping in linseed oil (DLSO)

Defect free wood specimens of group-4 received this treatment. Samples (Total 80) of different dimension, as per the requirement for studying different parameters given in table3.2 were prepared from seasoned wood of \( P. \ radiata \) by method given in section 3.3.1. For volumetric swelling, EMC, weight gain, water absorption total of 34 specimen of dimension 60×20×20 mm, radial swelling and tangential swelling total 24 specimen of size 50×20×20 mm, for termite resistance test 10 samples of size 100×25×6 mm and for fungal resistance 12 sample of size 20×20×20 mm were taken. All the test samples were placed in a tray. After wire mesh frame and weight block were kept on wood samples,
tray was filled with pure linseed oil. Samples were submerged for 24 h in linseed oil and then removed, wiped off with cloth to remove excess oil. Weight gain due to oil absorption was recorded in ten samples of dimension 60×20×20 mm. Weight gain and retention was calculated by using equations given in section 3.3.3.4.