Chapter II

Materials and Methods

The studies on the environmental impact of using chromated copper arsenate (CCA) treated wood preservative for aquatic applications were carried out mainly under two heads. Leaching studies and the corrosion studies. The leaching studies were further categorized into the effect of retention, the influence of leaching media and the control of leaching. The importance of wood sample size in leaching is also experimented. These experiments were conducted both in the laboratory and field conditions. In the laboratory, preservative treated wood panels were kept in water under accelerated condition (using magnetic stirrers) and in stagnant condition by immersing in aquaria. In the field this experiment was conducted by exposing the treated wood panels in the estuary. For this, wood was treated with CCA into different retentions and the effect of retention in leaching of CCA was experimented. The influence of leaching media in controlling the rate of leaching was experimented by exposing the treated wood panels in water of varying salinity and pH. The role of exposed surface area in leaching of preservative components was also experimented in both these methods by using two different sizes of wood panels. The experiment to identify methods to control the leaching of CCA from preservative treated panels was conducted by using multiple treatments like dual preservative treatment, by using physical barriers
like FRP sheathing and by painting after CCA treatment. The accumulation pattern of CCA components in the sediment near the exposure site was also experimented in the laboratory by exposing preservative treated wood of different retentions in the vicinity of sediment collected from the field. The speciation of CCA in the water where treated wood was exposed was also studied by analyzing the species of preservative components leached into the water in the laboratory experiment.

The corrosion studies were categorized into two. The effect of CCA retention on corrosion of nails and screws used for boat building and the nail and screw holding capacity of wood panels treated to different CCA retentions. The first experiment was conducted by exposing preservative treated wood nailed with different types of nails and screws in the salt spray chamber in the laboratory and by immersing in the estuary for field experiment. The latter experiment was done by conducting the nail and screw holding capacity experiment of wood (IS 1708-1969) after nailing the preservative treated wood with nail and screw.

2.1. Materials

2.1.1. Rubber wood

Rubber wood is a light hard wood of density 450-626 kg m\(^3\) having characteristic strength properties comparable to that of any commercially accepted durable wood species. Rubber wood does not offer much resistance to
the penetration of wood preservatives and because of the above-mentioned qualities rubber wood is selected for the study.

The freshly felled plantation-grown rubber wood tree samples were collected from local suppliers at Cochin area. The tree was of 30 years of age. The portions of the tree free from knots, without visible evidence of infection from mould, stains or decay fungi, were used for the preparation of panels. The rubber wood panels of size 150 x 100 x 25 mm were cut and the edges of the panels were smoothened using a planer. Immediately after collecting the panels, they were immersed in 2% CCA solution to prevent the fungal attack.

Air seasoning of the panels was carried out for a period of 4 weeks promptly after immersion in 2% CCA. After the seasoning period, moisture content of the wooden panels were determined by oven dry method. In this method, representative samples of size 25 x 50 x 50 mm were weighed and dried at 102 ± 1 °C and allowed to attain a constant dry weight. The moisture content of the samples were calculated using the formula

\[
\text{Moisture content} = \frac{\text{Wet weight} - \text{Oven-dry weight}}{\text{Oven-dry weight}} \times 100
\]

The panels below 25% moisture content and devoid of any cracks were selected for the study.

2.1.2. Marine plywood

Marine plywood has been extensively used for marine construction especially for boat building due to high economical viability and relatively low
damage in aquatic conditions. Marine grade plywood comprises as much as 80% of the material of any plywood vessel. To improve the quality of the plywood CCA treatment is usually employed because of greater penetration and fixation of CCA into the veneers. Marine plywood sample from Greenply manufacturers was purchased from the local market in 203.2 x 101.6 x 19 mm size. Panels of size 150 x 100 x 19 mm were cut and used for the experiment. Analysis of representative plywood samples in Inductively Coupled Plasma Atomic Emission Spectrophotometer (ICP-AES) showed retention of 4.05 kg m\(^{-3}\) of CCA.

2.1.3. Preservative solutions

CCA: The commercially available CCA manufactured by ASCU was taken for the study. CCA is prepared according to Indian Standard 10013-1981. The formulation is given in the Table 2.1. The 7.5 % (w/v) of CCA solution was prepared by dissolving CCA in water by gradual increase in temperature up to 45\(^\circ\)C. The precipitate was removed and the supernatant solution was cooled and used for preservative treatment.

Creosote: Commercially available light creosote oil with a specific gravity 1.03 was purchased and used for the experiment.

2.1.4. Paint

Coal tar epoxy finish paint (Asian paints) was purchased commercially and used for the experiments. A total of twenty-five panels were coated of coal
tar epoxy paint. The base and hardener was mixed together in a ratio 4:1 as specified by the manufacturer. Two coats of the paint were given with an intermittent drying period.

2.1.5. Fibreglass Reinforced Plastic (FRP)

The FRP sheathing was done using Chopped Strand Mat (CSM) of weight 450 g m\(^{-2}\) used for boat building purposes. The resin used for reinforcement was general-purpose polyester resin. Twenty-five numbers of panels were given two layers of resin coating. The panels after proper curing were used for the experiment.

2.2. Methods

2.2.1. Preservative Impregnation Procedure

The selected panels were treated with 7.5\% (w/v) CCA solution to get retentions of 16 kg m\(^{-3}\), 29 kg m\(^{-3}\) and 42 kg m\(^{-3}\). The above-mentioned retentions were selected in such a way that they cover the minimum (16 kg m\(^{-3}\)) and maximum (32 kg m\(^{-3}\)) retentions (Findlay, 1985) recommended by AWPA for aquatic purposes. The wet weight retention of the preservative in the panel was calculated as per ASTM D2481-81. After air seasoning for a period of two weeks, 25 panels were selected and pressure treated with creosote (dual treatment).

Preservative treatment was done by Full Cell or Bethell process according to IS-401:1960. The process called pressure impregnation was
carried out in vacuum pressure impregnation chamber of 400 l capacity which is fixed vertically. The impregnation chamber was connected to a supplementary tank for storing the preservative. The panels were loaded in the treatment chamber and screwed airtight. A vacuum of 56 cm of Hg was applied for 30 minutes with a vacuum pump in order to remove the air and moisture present in the wood cells. The preservative solution from the supplementary tank was passed into the treatment chamber under vacuum. When the chamber was filled with the preservative solution the vacuum was released. The valves were closed and pressure was applied, so that preservative solution gets imbibed into the wood cells. The conditions provided in the preservative chamber to get retentions of 16 kg m$^{-3}$, 29 kg m$^{-3}$ and 42 kg m$^{-3}$ of CCA and retention of 150 kg m$^{-3}$ for dual preservative are given in the Table 2.2. The time and amount of pressure applied varies according to the required net wet weight retention. A final vacuum of 38 cm of Hg for 15 min was applied to drain the excess of preservative from the panels and to facilitate drying.

The retention of the preservative in the panels on wet weight basis was calculated as per ASTM D2481-81.

$$\text{Retention, kg m}^{-3} = \frac{1000 GC}{V}$$

where, $G = T_2 - T_1$, weight in grams of the treating solution absorbed by the wood.

$C =$ Grams of preservative in 100 grams of treating solution.

$V =$ Volume of the block in cm$^3$. 

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The qualitative estimation of preservative penetration in wooden panels was conducted to confirm the extent of penetration of CCA into the panels. In this method, a piece of preservative treated wood sample is sprayed with a solution freshly prepared by dissolving 0.5 g of diphenyl carbazide in 50 ml isopropyl alcohol and made up to 100 ml (IS: 2753, 1991). The reagent treated surface was examined after 15 minutes. The purple coloration indicated the area where the preservative solution has penetrated. The panels selected for the studies are given in plate 2.2.

2.3 Experimental Procedures

The factors influencing the leaching of the preservative into the aquatic environment were first studied in the laboratory condition. The important factors like retention of the preservative, the influence of leaching media and the methods to control leaching were done under this category. Detailed procedure is described in the corresponding chapters.
Table 2.1: Formulation of CCA according to Indian Standard

<table>
<thead>
<tr>
<th>Element</th>
<th>Compound</th>
<th>Proportion (%)</th>
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<tbody>
<tr>
<td>Copper</td>
<td>CuSO$_4$.5H$_2$O</td>
<td>37.5</td>
</tr>
<tr>
<td>Chromium</td>
<td>K$_2$Cr$_2$O$_7$</td>
<td>50</td>
</tr>
<tr>
<td>Arsenic</td>
<td>As$_2$O$_3$.2H$_2$O</td>
<td>12.5</td>
</tr>
</tbody>
</table>

Table 2.2: Conditions of Vacuum-Pressure Impregnation

<table>
<thead>
<tr>
<th>Retention (kg m$^{-3}$)</th>
<th>Initial vacuum (cm of Hg)</th>
<th>Time (min)</th>
<th>Pressure (kPa)</th>
<th>Time (min)</th>
<th>Final vacuum (cm of Hg)</th>
<th>Time (min)</th>
</tr>
</thead>
<tbody>
<tr>
<td>CCA</td>
<td>16</td>
<td>56</td>
<td>30</td>
<td>172.37</td>
<td>38</td>
<td>15</td>
</tr>
<tr>
<td></td>
<td>29</td>
<td>56</td>
<td>30</td>
<td>448.16</td>
<td>38</td>
<td>15</td>
</tr>
<tr>
<td></td>
<td>42</td>
<td>56</td>
<td>30</td>
<td>517.11</td>
<td>38</td>
<td>15</td>
</tr>
<tr>
<td>Creosote</td>
<td>150</td>
<td>56</td>
<td>30</td>
<td>344.74</td>
<td>38</td>
<td>15</td>
</tr>
</tbody>
</table>
Fig. 2.1: Rubber wood panels selected for the study

* Clock wise from top: Untreated, treated with CCA to 16 kg m$^{-3}$, treated to 29 kg m$^{-3}$, treated to 42 kg m$^{-3}$, marine plywood, painted after treatment with CCA to 16 kg m$^{-3}$, FRP sheathed after CCA treatment to 16 kg m$^{-3}$ and dual treated with creosote after CCA treatment to 16 kg m$^{-3}$