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Influence of Heat Treatment on Chemical and Mechanical Properties of *Toona ciliata* M. Roem. Wood

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Abstract

Thermal modification or heat treatment was performed on the samples of *Toona ciliata* at various temperatures (80°C, 120°C, 160°C, 200°C) and durations (2h, 4h, and 6h) which revealed that chemical and mechanical properties of the wood improved at particular temperature with certain time duration. These results suggest that thermal treatment considerably improves wood properties like dimensional stability and durability. Vacuum oven was used to assess the process of thermal modification and different procedures were used to study the chemical and mechanical properties of Toona wood. In chemical properties the maximum value of holo cellulose content (71.73%) and lignin content (27.52%), were observed at 200°C. While, the minimum value of holo cellulose content (61.29%) and lignin content (24.42%), were found to be at 200°C and at control, respectively. In mechanical properties the maximum value of tensile strength (0.072), bending strength 0.022), compression parallel to the grain (0.052) and compression perpendicular (0.039) to the grain was observed at 120°C. Whereas, the minimum tensile strength (0.039), bending strength (0.008), compression parallel (0,041) the grain and perpendicular (0.027) to the grain was found at 200°C. All the chemical and mechanical properties of Toona wood improved after heat treatment at particular temperature and duration.

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Wood is heterogeneous substance composed of cellulose, hemicelluloses and lignin[1] and their composition varies among different morphological areas.[2] It is a sustainable resource which has been used in both indoor and outdoor applications since ancient times. *Toona ciliata* M. Roem, is also known as the Red Cedar, Tuni and Indian Mahogany, of family Meliaceae, grows in a subtropical climate

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in moist localities such as ravines, banks and even in swamps. In India, this tree is found in Sub - Himalayan tracts of Assam and throughout hilly regions of Central and Southern India. The species also found in the plains of Madhya Pradesh, Tamil Nadu, Karnataka, Eastern and Western Ghats extend up to 1200 masl (rarely 1300 masl).[3] In Himachal Pradesh, T. *ciliatais* mainly found in Mandi, Bilaspur, Hamirpur, Kangra, Solan, Una and Sirmour districts. As a biological material, dimensional instability under changing moisture conditions and biodegradability are the main disadvantages of wood.[4]

Toon woodis a most suitable material for construction since ancient times because of its ease of working and other excellent properties like; high strength, heat insulation, and ease of handling and processing, however there are several factors which affect the properties of wood like; moisture, fire, defects *etc*. For outdoor construction purpose, moisture is the main problem, which degrades the quality of wood and results in several defects that make it susceptible to insect and fungal attack. Modification of wood properties by using different preservation techniques *i.e*. use of chemical treatment affects the environment, humans, and animals and also involves higher cost. Therefore, thermal modification technique can improve the quality and value of wood for different construction purposes.

Thermal modification is one of the most environmentally friendly and appropriate ways to change the properties of wood. This technology treats wood at high temperatures for increased durability and dimensional stability. Under the action of heat, the decomposition of amorphous cellulose increases crystalline cellulose. As a result, the accessibility of hydroxyl groups in water molecules is reduced and the water content of heat treated wood is reduced.[5] A proper treatment increases hydrophobic properties, reduces the mass andmodify other physical properties of wood, especially colour, specific gravity, density, swelling, shrinkage and weathering resistance etc.[6-7] These changes are observed in wood elasticity, and dimensional stability that increase the effectiveness of wood for construction.[8-9] The first study on heat treatment of wood had been conducted in 1920 on high temperature wood drying process and results of this work showed that thermal modification decreases the equilibrium moisture quality and consequently the wood swelling.[10] Heat treated wood has a number of advantages that appear after its modification. Such a wood is lighter and stronger than the untreated timber and also exhibits higher lignin content as well as a lower acid number than untreated woods resulting in the degradation of some hemicelluloses and extractives compounds.[9] This technique is one of the effective method for improving the solidness, durability and dimensional stability of wood without using chemicals.

Material and Methods

The wood of the *Toonaciliata* was converted into logs and then into required dimensions as per the test specifications. After thermal treatment the samples were converted into saw dust and divided into three (replication) for chemical analysis. The remaining log was converted to form the following samples for testing of mechanical properties. The dimensions of the samples prepared for conducting various tests are as below:

1. 300 mm x 10 mm x 10 mm (for tensile strength)
2. 300 mm x 20 mm x 20mm (for static bending)
3. 50 mm x 20 mm x 20 mm (for compression parallel and perpendicular to the grain)

The toonwood sample was modified in the following way. Step 1: Ovendry the sample at room temperature to evaporate the water from the sample. Step 2: Heat the sample in a vacuum oven at predetermined temperatures (80±2°C, 120±2°C, 160±2°C, and 200±2°C) for the expected time (2, 4, 6 h). Step 3: Place the sample in a desiccators to cool the sample to room temperature and constant weight without moisture. Rapid chemical analysis was performed by standard method TAPPI (Pulp and Paper Industry Technology Association). The shade dried wood samples were chopped using a wood chippercum grinder and then further dried in an oven at a temperature of 50 ± 1°C for 24 h. The experiment was based on the Completely Randomized Design factorial. A total of 3 replicates were performed for this species and 3 samples were used for each replicate. The water-soluble extract was determined by employing the following methods.

Cold Water Solubility

2 gram of oven dried coarsely ground wood was weighed and transferred to a conical flask containing 300 ml of distilled water The mixture was brought to a boil at room temperature with frequent stirring for 48 hours. The material was then filtered through an IG-1 crucible, washed thoroughly with distilled water, and dried to constant weight in an oven at 105 ± 2 °C. Solubility in cold water was determined by calculating the weight loss of the sample taken and expressed as a percentage based on the dry weight of the wood in the oven.[11]

Hot Water

Place two grams of oven dried raw wood in a flask containing 100 ml of distilled water. It is digested in a bath of boiling water for 3 hours. The contents were then filtered through the IG-1 crucible and the residue was dried in an oven at 105 ± 2°C to constant mass. Solubility is determined by calculating the mass loss of the sample taken and expressed as a percentage.[11]

Alcohol-Benzene Extractives

10 gram of Oven dried; coarsely ground wood was placed in a porous thimble (oven drying and weighing). Thimble was placed in a soxhlet apparatus and extracted with alcohol-benzene (ratio 1:2 by volume) for 6 hours. Then, the porous thimble was taken out, dried outdoors, and finally dried to a constant weight in an oven at 105 ± 2 °C. The solubility of alcohol-benzene was determined by calculating the mass loss of the sample taken and expressed as a percentage.[12]

Klason-Lignin Content

2 grams of pre-extracted dried sample with alcohol-benzene (1:2 by volume) was treated with 15 ml of 72 % sulphuric acid for 2 h at 18-20 °C and stirred continuously. The material was reduced to 3% by adding 545 ml of distilled water. The solution was refluxed for 4 h and then allowed to settle. The contents were filtered and washed with hot distilled water. The material was then dried in an oven at 105 ± 2°C to constant mass and determined by calculation on the basis of the oven dry weight.[13]

Holocellulose

Fivegrams of the dried sample pre-extracted with alcohol-benzene (1:2 by volume) were placed in a conical flask and 160 ml of distilled water were added to it. The contents were treated with 1.5 g of sodium chlorite and 10 drops of acetic acid (25 ml pipette) at 70-80 °C on a water bath for one hour. This process is repeated four times until the meal turns white. The contents were then filtered through an IG-2 crucible, washed with distilled water and finally with acetone. The sample was dried in an oven at 105 ± 2 °C to constant mass. The extracted *holocellulose* content was calculated on the basis of oven dry weight.[14]

The mechanical properties of wood were determined as per the procedure followed for testing in Universal Testing Machine (Model:UTN-10). The mechanical test of toon wood samples was determined by the procedure prescribed by Indian Standard IS: 1708.[15]

Tensile Strength (kN/mm^2)

The standard size of the sample taken for test was 300×10×10mm. The computer generated data and graph were generated as per the software, so as the values of maximum load, maximum displacement and fracture pattern of thermally modified wood. Adequate care was taken to ensure that each sample was exposed to the same type of test measurement/ conditions(all the three tests were performed on the same machine/ UTM).

Bending Strength (kN/mm^2)

The standard size of the sample for this test was 300×20×20mm. Proper care was taken so that each specimen faced similar type of test measures/ conditions. The generated data were used for further analysis and comparison.

Compression Strength Parallel To The Grain (Kn/Mm^2)

This test was done in the direction along the grain and the data were generated in the Universal Testing Machine (Model: UTN-10). The standard size of specimens for this compression test was 50×20×20mm.

Compression Strength Perpendicular To The Grain (Kn/Mm^2

The specimen size of 50×20×20mm was taken across the direction of grain for carrying out this test. The data recorded were used for further analysis and interpretation of results.

The experiments were carried out in the Completely Randomized Block Design (CRD)[16-17]

Results And Discussion

Chemical Properties of Wood

Cold Water Soluble Extractives (%)

Among temperatures, the minimum cold water soluble extractives (4.69%) was recorded in 200°C which was at par with control (4.93%) and maximum (7.20%) was found at 120°C. In duration, highest cold water soluble extractives were recorded at 4h (6.02%) which was at par with 2h (6.01%) and lowest at 6h (5.64%). The interaction between temperature and duration was significant where maximum (7.30%) value was observed at 120°C for 4h which was at par with 120°C (7.26%) at 6h; 120°C (7.03%) at 2h; 160°C (6.82%) at 2h and minimum (3.75%) at 200°C for 6h (Table 1).

Species with large quantities of extractives are more durable, more stable and have better plasticity (It is a universal truth that the wood durability directly proportional to the extractives). The cold water extractives present in the wood are mainly tannins, gums, sugars and salts etc. According to Esteves,[18] in the thermally treated wood of *Eucalyptus globulus*, there is increase in the release of extractives form wood in beginning then reduction has been observed. This might be because of evaporation of the different extractives during heat treatment.[19] So excessive heating causes more loss in the extractive content in wood. Similarly, finding illustrates thatin cold water extractives of *Fraxinus angstifolia*, there is decrease in cold water extractives content with rise in temperature and time durations.[20] Some researcher has shown that, at 160°C and and 200°C maximum reduction in extractives content has been observed in *Corymbia citriodora wood*.[21] The present study on the cold water soluble extractives of thermally modified *Toonaciliata* wood also get support from above findings carried out by different researchers.

Hot water Soluble Extractives (%)

The data was observed to be statistically different at 5 per cent level of significance. For temperatures, the maximum (12.09%) hot water soluble extractive was recorded at 120°C and minimum (6.90%) was recorded in 200°C. Among durations the highest value (9.69%) was recorded at 2h and lowest (9.47%) at 6h, which was at par with 4h (9.48%). The interaction between temperature and durations was notable. however, maximum (12.55%) was recorded at 120°C for 6h and minimum (6.15%) at 200°C for 6h (Table 1). The hot water soluble content present in wood generally consist of tannins, gums, sugars, fats, waxes, alkaloids salts and phenols. In the present findings, firstly there was increase and then decrease in hot water soluble extractives with increase in temperature. This may be because, hot water increases the solubility of the extractives more than cold water and also resin acids decompose above 200°C which results in decrease in the extractive content. According to Yildiz, at 180°C to 200°C, relative content of hot water soluble extractives decrease with increase in temperature.[22] Similarly, in pine wood, at temperatures 100 to 160°C, waxes and fats move to the wood surface resulting in increased extractive content and at 180°C decomposition starts, which resulted the decrease in the extractive content. 23 The present study on the hot water soluble extractives of thermally modified *Toonaciliata* M. Roem. wood are in line with above finding carried out by different researchers in which firstly there was increase and then decrease was found with rise in the temperature.

Alcohol Benzene Extractives (%)

Alcohol benzene extractives (%) and showed that there is a significant difference between the values of alcohol benzene extractives obtained from thermally modified wood. For different temperature maximum alcohol benzene extractives (13.13%) were found at 200°C and minimum (8.03%) recorded at control. Among durations maximum value was recorded at 6h (11.29%), which was at par with 4h (11.06%) and minimum (10.76%) was recorded in 2h. In the interaction between temperature and duration the maximum (13.61%) value was at 200°C for 6h and minimum in control (8.03%) (Table 1).

The group of extractives (waxes and oils), which are responsible for various functions such as energy reserves, tree metabolism and defence mechanism against microbial attack as well as influence pulping quality of wood are alcohol-benzene soluble extractives. Alcohol benzene extractives increase with increase in temperature and duration in *Fraxinus angstifolia.*[20] Similar results have been reported by researcher, where it has been found that minimum content

of alcohol benzene is recorded in control and maximum at 220°C for 6h in *Bombaxceiba* wood. Sikora *et al.* (2018) has revealed that at temperatures 100 to 160°C in pine wood, waxes and fats move to the wood surface resulting in increased extractive content and at 180°C decomposition starts, which resulted the decrease in the extractive content.

Table 1: Chemical properties of heat treated wood

Temperature (°C) And Time (h)		Cold water extractives (%)	Hot water extractives (%)	Alcohal benzene extractive (%)	Holocellulose	Lignin
Control	4.93	9.25	8.03	68.27	24.55	
80°C	2h	5.81	9.29	9.38	68.95	21.78
	4h	6.48	9.57	10.22	69.42	21.40
	6h	6.59	10.33	10.30	70.70	22.14
120°C	2h	7.03	11.35	11.53	72.12	24.71
	4h	7.3	12.36	11.65	71.15	25.93
	6h	7.26	12.55	11.84	71.92	25.62
160°C	2h	6.82	10.65	12.03	70.35	26.05
	4h	6.53	9.55	12.46	69.58	26.33
	6h	5.68	9.09	12.68	68.54	26.96
200°C	2h	5.47	7.91	12.84	64.62	27.08
	4h	4.84	6.65	12.94	60.82	27.52
	6h	3.75	6.15	13.61	58.43	27.95
Mean	80°C	6.29	9.73	9.97	69.69	21.78
	120°C	7.2	12.09	11.67	71.73	25.42
	160°C	6.34	9.76	12.39	69.49	26.45
	200°C	4.69	6.9	13.13	61.29	27.52
	Control	4.93	9.25	8.03	68.27	24.42
Mean	2h	6.01	9.69	10.76	68.86	24.76
	4h	6.02	9.48	11.06	67.85	25.15
	6h	5.64	9.47	11.29	67.57	25.44
$CD_{0.05}$	T	0.299	0.039	0.035	0.505	0.684
	D	0.231	0.031	0.027	0.391	0.53
	T×D	517	0.068	0.061	0.875	NS

In the present investigations similar results have been observed in the thermal modification for Toon wood.

Holocellulose Content

The maximum *holocellulose* content (71.73%) was recorded at 120°C and minimum (61.29%) was recorded at 200°C. For durations the highest value (68.86%) was recorded at 2h and lowest value (67.57%) at 6h which was at par with 4h (67.85%). The interaction between temperature and duration revealed statistically significant results. The maximum value (72.12%) was recorded at 120°C for 6h which was at par with 120°C (71.92%) for 6h and minimum value (58.43%) at 200°C for 6h was found (Table 1). According to Rowell (2013),[25] *holocellulose* contributes to the major portion of the wood (Cellulose + hemicelluloses) up to 65-70 per cent of the dry weight. According to the studies by Sikora, hemicelluloses are the least stable component of wood during thermal treatment as their amount decreases in thermally modified wood[23,26] The study carried out by Gupta[19] has reported that with increase in temperature there is a decrease in the *holocellulose* content. Similarly, Pingale[24] found

decrease in the content of hemicelluloses with rise in temperature and stated that this was because of depolymerisation of hemicelluloses and degradation of the cellulose content in heat treatment. The present findings of thermally modified Toon wood are in accordance with the above researchers.

Lignin Content

The maximum Klason-Lignin content (27.52%) of heat treated wood was recorded at 200°C and minimum (21.78%) was recorded in 80°C. For durations, the highest value (25.44%) was recorded at 6h which was at par with 4h (25.15%) and lowest value (24.76%) recorded at 2h. However, a non significant difference was found between interaction of temperature and durations and ranged between 21.40 to 27.95 per cent (Table 1).

Lignin is a class of complex organic polymer present in secondary cell wall and provides support to the tissue. Among all components it is the most stable and resistant component in thermal degradation (Fengel and Wegener, 1989).[27] According to various researchers[21] with increase in temperature there is increase in lignin content in *Corymbiacitrodora* and maximum lignin content has been recorded at 200°C. In present study, the maximum amount of lignin has been observed at 200°C and with increase in temperature and duration the lignin content has also increased. This might be due to the resistance of lignin and decline of polysaccharide (hemicelluloses and cellulose) in thermal modification.[28] Similarly, Sikoraand his co- workers has shown that the amount of lignin in modified wood increases slightly with increases in temperature.[23]

Mechanical Properties

Tensile strength of Wood (kN/mm^2)

For different temperatures, maximum tensile strength (0.072kN/mm^2), of wood was observed at 120°C which was statistically at par with control (0.071 kN/mm^2) the minimum (0.039 kN/mm^2) was at 200°C. Among durations, the highest (0.066 kN/mm^2) and lowest (0.055 kN/mm^2) was recorded at 2h and 6h, respectively. Whereas, in the interactions between temperatures and durations the maximum value (0.074 kN/mm^2) was noticed for 120°C for 4h which was at par with 120°C (0.073 kN/mm2) for 2h; 120°C (0.068 kN/mm^2) for 6h; 160°C (0.070 kN/mm2) for 2h; 120°C (0.066 kN/mm^2) for 4h; 80°C (0.071 kN/mm^2) for 2h and minimum value (0.032 kN/mm^2) was recorded at 200°C for 6h (Table 2).

Tensile strength is the maximum load that a material can bear without fracture when applied in tension. In the present investigation the tensile strength has shown first increasing trend and then decreasing after heat treatment. The decrease in tensile strength is due to decrease in the amount of extractives, degradation of amorphous region of cellulose and hemicelluloses. According to the Boonstra and Tjeerdsma,[29] thermal modification results in degradation of cellulose component in the wood and degradation of cellulose component leads to decrease in tensile strength of the wood.[30] The present study is in obedience with the earlier study that has been performed by Pingale[24] where there is first increasing and then decreasing trend in thermal modification of *Bombaxceiba* wood.

Bending Strength of Wood (kN/mm^2)

At different temperatures, the maximum bending strength (Plate 7) of thermally modified wood of *Toonaciliata* was recorded as 0.022 kN/mm^2 at 120°C and minimum 0.008 kN/mm^2 at 200°C. For durations, the data was observed to be statistically non significant. However, the interaction between temperature and durations was found to be significant, whereas highest value was noticed at 160°C (0.024 kN/mm^2) for 2h, which was at par with 160°C (0.020 kN/mm^2) for 4h; 120°C (0.020 kN/mm^2) for 2h; 120°C (0.023 kN/mm^2) for 4h; 120°C (0.022 kN/mm^2) for 6h; 80°C (0.022 kN/mm^2) for 6h and lowest value at 200°C (0.007 kN/mm^2) which was at par with 200°C (0.008 kN/mm^2) for 4h; 200°C (0.010 kN/mm^2) for 2h; control (0.011 kN/mm^2), respectively (Table 2).

Bending strength is the load, that wood can withstand perpendicular to the grain. According to Boonstraand his coworkers and Gupta after heat treatment there is a decrease in bending strength in Radiate Pine and *Pinusroxburghii*, respectively.[8,19] Although, hemicelluloses and cellulose are mainly responsible for decrease in bending strength of wood.[31] In the present study after heat treatment decrease in bending strength of the Toona has been recorded and the results are in accordance with earlier study performed by some scientist where reduction was found in the

bending strength of Douglus fir and bamboo wood, respectively after heat treatment.[32-33]

Compression Strength (kN/mm^2) Parallel to Grain
Table 2 depicted that, for temperatures, the highest value recorded in control and 120°C (0.052 kN/mm^2), which was found to be statistically at par with 160°C (0.048 kN/mm^2) and lowest value was recorded at 200°C (0.041 kN/mm^2). While, at durations and interactions data was found to be non significant and ranged from 0.048 to 0.049 kN/mm^2 and 0.038 to 0.057 kN/mm^2, respectively.

Table 2: Mechanical properties of heat treated wood

Temperature (°C) and Time (h)		Tensile strength	Bending strength	Compression strength	
				Parallel to grain	Perpendicular to the grain
Control	0.071	0.011	0.052	0.052	0.052
80°C	2h	0.071	0.012	0.051	0.051
	4h	0.052	0.018	0.052	0.052
	6h	0.054	0.022	0.053	0.053
120°C	2h	0.073	0.02	0.048	0.048
	4h	0.074	0.023	0.052	0.052
	6h	0.068	0.022	0.057	0.057
160°C	2h	0.07	0.024	0.047	0.047
	4h	0.066	0.02	0.049	0.049
	6h	0.049	0.014	0.049	0.049
200°C	2h	0.044	0.01	0.043	0.043
	4h	0.042	0.008	0.041	0.041
	6h	0.032	0.007	0.038	0.038
Mean	80°C	0.059	0.017	0.051	0.051
	120°C	0.072	0.022	0.052	0.052
	160°C	0.062	0.019	0.048	0.048
	200°C	0.039	0.008	0.041	0.041
	Control	0.071	0.011	0.052	0.052
Mean	2h	0.066	0.015	0.048	0.048
	4h	0.061	0.016	0.049	0.049
	6h	0.055	0.015	0.049	0.049
$CD_{0.05}$	T	0.004	0.002	0.006	0.006
	D	0.003	NS	NS	NS
	T×D	0.008	0.004	NS	NS

Compression strength is an ability of wood to resist force from opposite ends which tends to come closer to each other. The cellular composition and porosity is mainly responsible for compression strength of the wood. According to Boonstra and his co-workers found that compressive strength parallel to grain increases after heat treatment[8] present studies same has been observed at 120°C. The increase in compressive strength in the longitudinal direction might be due to the presence of lesser amount of bonded water in heat treated wood which is one of the factors responsible for strength of the wood. In current study there is first increase and then decrease in compression strength of the wood andsimilar trends of results has been observed in the earlier studies by Gupta[19] and Pingale.[24]

Compression Strength (Kn/Mm^2) Perpendicular to Grain

The perusal of the data on temperature revealed that the minimum (0.027 kN/mm^2) value was recorded at 200°C and maximum (0.039 kN/mm^2) at 120°C. For durations, the highest value (0.035 kN/mm^2) was recorded at the lowest (0.031 kN/mm^2) value recorded at 4h and 6h. However, the interaction between temperature and duration was observed to be non-significant and ranged from 0.025 to 0.041 kN/mm^2 (Table 2).

Determining the compression strength is an important parameter for better utilization of the wood for different construction purposes. It mainly depends on the chemical bond, extractive and porosity of the wood. Compression perpendicular to the grain has less value than compression parallel to grain.[8]

This difference is due to presence of several types of bonds present in the transverse direction and orientation of the polymer molecules and fibers in wood.[34] The similar results have been reported in *Pinusroxburghii* and *Bombax ceiba*.[19,24] The results of present study are in obedience with above statement.

Concluding Remarks

- From above investigations, we can conclude that heat treatment at a particular temperature and duration is suitable and can be utilized as a potentialgreen alternative for different hardwoods.
- In thermal modification, we use heat to increase or decrease the amount of extractives present in wood.
- There was also an increase in the content of lignin after thermal modification which leads more stability and strength of the wood.
- In this investigation we got maximum positive results at temperature 120°C (6h) for Toona wood. At this temperature all the parameters showed significant results and also found maximum amount of extractives.
- Hence for thermal modification of Toona wood the temperature of 120°C for 6h is recommended for more stability and durability.

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Conflict of Interest

The authors declares that there is no conflict of interest.

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