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Effect of microwave pre-treatment on preservative retention and treatability of *Melia composita wood*

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Abstract: The species *Melia composita* has come to prominence only during the last decade and has been found suitable for several applications and end uses. However, being non-durable and difficult to treat some pre-treatment is required to improve preservative uptake. Hence the effect of microwave (MW) pre-treatment was studied on the samples of *Melia composita* wood in order to ameliorate its retention, depth of penetration and treatability class to ensure its sustainable use for a longer duration. The samples were exposed to MW radiation of different intensities and treated with 6% solutions of CCA, ZiBOC, CCB and Borax boric acid (BBA) preservatives by a dip diffusion method for 24 hours. Results showed a significant improvement in preservative uptake after dipping for 24 hours, in comparison with the controls. The highest retention was found in the samples treated with ZiBOC preservative after their MW exposure to 840 $MJ \cdot m^{-3}$. Wood anatomical analysis after MW modification was also performed in order to assess the changes taking place within the wood microstructure. The cross-sectional analyses of the controls and the treated specimens were briefed accordingly. Results of the anatomical study exhibited a significant improvement in the effective vessel diameters of the microwave irradiated specimens due to the clearance of the occlusions blocking the vessels with respect to the control samples, which aids in increasing wood permeability and results in higher retention and penetration.

Keywords: microwave modification; permeability; *Melia composita*

Depleting supplies of durable wood in the present day has resulted in the use of many non-durable timber species. Such a use, however, becomes uneconomical if the wood is used in its natural form as products manufactured from such species deteriorate rapidly, particularly in tropical climatic conditions and necessitate early and frequent replacements. However, such situations can be easily averted by adopting effective preservation and modification techniques and ensuring a trouble-free long service life. The role of wood preservation hence lies in eliminating the replacement of wood components or at least in shortening the replacement cycle. This can result in the sustainable use of plantation species like Eucalyptus, Poplar, Rubber Wood and *Melia composita* which are mostly non-durable in nature and are degraded very soon.

The wood of *M. composita* can be used for a varied lot of end uses starting from packing cases, cigar boxes, ceiling planks, building and construction materials, agricultural implements, pencils, matchboxes, splints to even plyboards (Sharma et al. 2012). The species is newly known to researchers and stakeholders and is now catching up among the plywood industrialists and farmers leading to the establishment of plantations. Presently, the plywood and panel industry is facing an acute shortage of raw materials and a number of alternative fast-growing tree species have been explored for the production of wood-based panel products. Parthiban et al. (2009) integrated *Melia composita* in agro-forestry farms as an alternative pulpwood species. However, the species is reported treatability class "C" (Tripathi 2012) (equivalent to class

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2 as per EN 350: 2016) and hence for its sustained use some pre-treatments prior to preservation can be opted for in order to enhance its permeability, retention and penetration percentages. Microwave wood (MW) modification is an innovative method to increase the wood permeability which helps in improving the preservative penetration in the wood of various species (Torgovnikov, Vinden 2009; Dashti et al. 2012). Torgovnikov and Vinden (2009) reported that green wood readily absorbs MW energy because of its high moisture content (MC). When microwave energy is applied to wood, the steam pressure generated within the wood cell ruptures in the pit membranes and ray cells, allowing an easier flow within the substrate. A several thousand-fold increase in wood permeability in radial and longitudinal directions could thus be achieved (Vinden et al. 2011). Xu et al. (2015) reported microwave pre-treatment of poplar wood to result in improved transverse permeability, which had a positive influence on the amount of impregnation. Thus, MW modification of wood can increase the wood permeability, rate of drying in hardwoods, improve drying quality and open new opportunities for increasing timber durability by impregnation with preservatives. MW energy applied for wood modification needs to be in the range of 250 to 1,200 MJ·m^{-3} depending on the purpose (Torgovnikov, Vinden 2010). MW processing to increase the uptake of water- based preservatives for some softwood species (Sitka Spruce, Radiata Pine) needs energy in the range of 250 to 400 MJ·m$^{-3;}$ this energy needs to be higher for highly refractory hardwood species (Torgovnikov, Vinden 2010). It is thus important to optimise MW energy so that it does not excessively damage the wood structure or significantly affect the mechanical properties, still improving the permeability. The energy required depends on the size of the wood and initial moisture content.

Liu et al. (2005) reported that the permeability of larch wood can be improved without noticeable reduction in the strength and stiffness if the conditions of microwave pre-treatment were optimized. Treu and Gjolsjo (2008) also reported that the microwave processing of Norway spruce (*Picea abies* L.) caused a significant increase in the uptake of a 2% copper-based preservative after MW treatment with more than 180 MJ·m^{-3} at a frequency of 2.45 GHz. The capacity of microwave energy to rapidly heat wood through its thickness was successfully attempted for a fast fixation of CCA preservative (Smith et al. 1996). Torgovnikov and Vinden (2010) reported that the technology of MW wood modification is based on the supply of high-intensity MW power, up to 486000 MJ·m^{-3} at frequencies of 0.922 and 2.45 GHz. Such power induces significant changes in the microstructure of wood and a dramatic increase in wood permeability. In light of the above literature *Melia composita* was exposed to MW radiation at different intensities and was given preservative treatment using the normal dipping process with copper chrome arsenate (CCA), copper zinc borate (ZiBOC), copper chrome boron (CCB) and Borax boric acid preservatives. Further the samples were analysed anatomically in order to study the changes taking place within the wood microstructure.

MATERIAL AND METHODS

Procurement of *Melia composita* logs was done from Forest Research Institute, Dehradun (30°19'N, 78°04'E) and they were subjected to conversion and seasoning. The specimens used for this purpose were of dimensions 3.8 cm (width) × 3.8 cm (thickness) × 10 cm (length). Relatively straight-grained and defect-free specimens free from insects, borers, termite attack or any other notable damaging factor were chosen so as to obtain an optimum result. A total of 144 samples were prepared. The samples were divided into six groups (5 groups with MW treated samples and one group consisting of the control samples as shown in Table 1) each

Table 1. Sample distribution

MW Energy (MJ·m^{-3})	CCA	ZiBOC	CCB	BBA	Total
–	6	6	6	6	24
360	6	6	6	6	24
480	6	6	6	6	24
600	6	6	6	6	24
720	6	6	6	6	24
840	6	6	6	6	24
Total	36	36	36	36	144

MW – microwave, CCA – copper chrome arsenate, ZiBOC – copper zinc borate, CCB – copper chrome boron, BBA – Borax Boric Acid

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having 24 specimens and one group corresponding to each MW treatment. Initial MC of the samples was determined on oven dry (OD) basis as per BIS (IS 11215:1991) prior to the experiment and was recorded as 30.9 (± 1.03) %.

MW treatment was carried out in a microwave heating device (Model 30SC3, IFB Industries, India) at a frequency of 2.45 GHz and maximum output power of 900 W. Treatments were defined based on power and the volume of wood specimens in order to obtain the desired energies as given in Table 1. After MW treatment the samples were conditioned to achieve MC of 12% and treated with CCA, CCB, Borax boric acid and ZiBOC (Tripathi 2013) preservative at 6% concentration by dipping for 24 hours. Six samples exposed to each MW energy level were treated with each preservative. After preservative treatment, the samples were taken out and the excess preservative was blotted out with filter paper and specimens were weighed immediately to determine the preservative uptake and retention (IS 401:2001). The amount of preservative solution absorbed by specimens (retention value R in $kg \cdot m^{-3}$) was calculated as follows equation 1:

$$R = (GC/V \times 10)\ (kg \cdot m^{-3}) \qquad (1)$$

where:

G – mass of the treating solution absorbed by block (g);

C – mass of the preservative present in 100 g of the treating solution (g);

V – volume of the test block (cm^3).

Penetration of the preservative was determined by cutting the specimens into equal halves for a spot test. The exposed surfaces were then sprayed with turmeric solution (for Borax: boric acid) to indicate the presence of boron whereas Chrome Azurol S solution was used to indicate the presence of copper in the other treatments (IS 2753:1991). The wood turned reddish brown for BBA and blue where CCB, CCA and ZiBOC (copper) had penetrated.

The anatomical analysis of MW exposed samples was carried out primarily to assess the changes taking place within the wood microstructure after MW radiation.

The samples were first checked for the orientation of rays and accordingly a cross-sectional cutting pattern for the preparation of blocks was decided so that the intersection of growth rings remained as close as possible to 90°. When analysing cross-sections, the wood samples were cut perpendicularly to the axially oriented xylem cells to avoid over- and underestimation of the measured anatomical features. In the present study, only the cross-sectional analysis was performed to assess the impact of MW modification.

The samples were sliced to smaller dimensions and were soaked for 24 hours at least. Boiling or just soaking the samples in water, embedding in paraffin, or using a corn starch solution helps to avoid damage to cell structures while cutting (Schneider, Gärtner 2013; Yeung et al. 2015). The samples were further boiled for 2 days in cycles of 30 mins in order to render them fit for the microtome. Sections of roughly 12–20 μm thickness were made using a microtome 358926 (Reichert, Austria). The sections were stained in Heidenhain's haematoxylin and safranin and mounted following the standard laboratory schedule, i.e. passing through grades of alcohol (10–100%), and finally in xylene and clove oil (50:50) for making permanent slides. Finally, the sections were mounted in Canada balsam. Vessel diameters were measured after microscopic analysis. The statistical analysis was carried out using SPSS (Version 25).

RESULTS AND DISCUSSIONS

MW treated samples were compared with untreated samples for the occurrence of drying defects. It was observed that no checks and cracks occurred at a lower intensity of MW exposure but the MW intensity of 840 $MJ \cdot m^{-3}$ resulted in the occurrence of minor checks or cracks in *M. composita* wood samples. The reasons for such defects may be the higher intensities of MW treatment.

The mean retention obtained in *Melia composita* specimens and in the control samples after treatment with CCA, ZiBOC, CCB and BBA for different exposure levels is given in Table 2. The obtained data set shows that the increase in retention is proportional to the increasing exposure of MW radiation irrespective of the preservatives used. Here, maximum retention was obtained for ZiBOC exposed to 840 $MJ \cdot m^{-3}$ intensity. A comparative analysis between the preservatives reveals that for all the given treatments the performance of ZiBOC was found to be considerably better than the rest in terms of retention as the preservative retained was higher in all the cases. Fig. 1 shows

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Table 2. Duncan analysis for retention after treatment with CCA, ZiBOC, CCB and BBA

Intensity ($MJ·m^{-3}$)	*n*	Subset ($kg·m^{-3}$)			
		CCA	ZiBOC	CCB	BBA
Control	6	2.7[a]	3.77[a]	3.34[a]	3.10[a]
360	6	3.7[a]	4.78[a]	3.57[a]	6.12[b]
480	6	4.77[b]	6.77[b]	5.65[b]	6.29[b]
600	6	6.64[c]	8.42[c]	6.93[bc]	6.95[bc]
720	6	7.55[cd]	8.68[c]	7.51[c]	7.55[bc]
840	6	8.50[cd]	8.75[c]	7.65[c]	7.97[c]

values with the same letter belong to the same subgroup, *n* – number of replicates, CCA – copper chrome arsenic, ZiBOC – copper zinc borate, CCB – copper chrome boron, BBA – borax boric acid

the effect of MW treatment on the permeability of *M. composita* wood samples treated with CCA through a colour test. It can be inferred that with an increase in MW intensity, the depth of penetration increased proportionally. With an increasing MW exposure, impregnation also increased substantially as can be seen in Fig. 1. Mottle impregnation was found in samples exposed to 360, 480 and 600 $MJ·m^{-3}$ whereas through and through impregnation was obtained in case of 720 $MJ·m^{-3}$ and 840 $MJ·m^{-3}$ intensities.

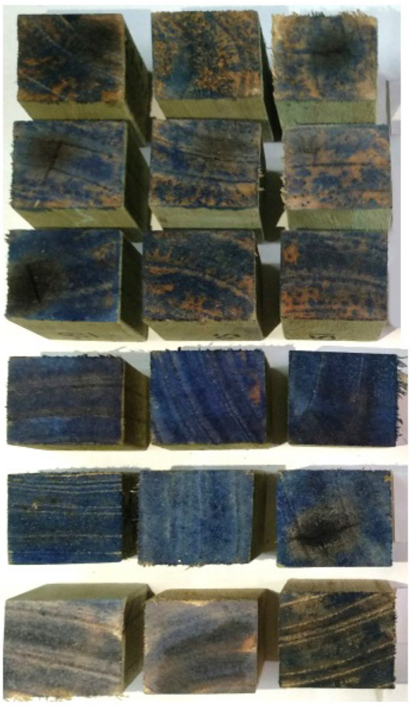

Fig. 1. MW irradiated samples of *M. composita* after treatment with CCA

The analysis of variance also indicated that the treatments (control and treated specimens) were different ($P \leq 0.05$). This documents that the control samples, which were subjected to preservative treatment without receiving the MW exposure, showed significantly lower retention than the treated ones, which substantiates the success of the process overall. The mean retention ($P \leq 0.05$) by the specimens were recorded and statistically analysed at a 5% significance level. It was inferred from Duncan's subsets (Table 2) that there were statistically significant differences between the MW treatments as is evident from the retentions of CCA, ZiBOC, CCB and BBA preservatives.

The Duncan analysis further establishes that there is a significant improvement in the retention of the treated specimens compared to the control samples. Four Duncan subsets were formed in CCA treated samples whereas three subsets were formed in samples treated with CCB, ZiBOC and BBA. However, the retention in control samples and samples exposed to 360 $MJ·m^{-3}$ MW intensity is non-significantly different except for BBA.

In case of CCA treated samples, there were significant ($P \leq 0.05$) differences between the remaining treatments while samples treated with 720 $MJ·m^{-3}$ and 840 $MJ·m^{-3}$ MW intensities exhibited statistically similar results as depicted in Table 2.

M. composita samples exposed to 600, 720, 840 $MJ·m^{-3}$ MW and treated with ZiBOC and CCB fell in the same subset. Whereas in case of BBA, samples exposed to 360, 480, 600,720 $MJ·m^{-3}$ fell in the same subset and samples exposed to 840 $MJ·m^{-3}$ were statistically different from the other treatments. However, it is very pertinent to mention that for ZiBOC the maximum retention of 8.75 $kg·m^{-3}$ was achieved followed by CCA, BBA and CCB in samples exposed to 840 $MJ·m^{-3}$.

To further substantiate the observations, a cross-sectional anatomical study was performed in order to assess the changes taking place within the wood microstructure. For the anatomical analysis, effective vessel diameters were considered and they were measured before and after treatment. Significant ($P \leq 0.05$) differences in the diameters before and after treatment were exhibited, which is in conformity with the work done by previous researchers (Torgovnikov, Vinden 2009; He et al. 2014). The cross-sectional anatomical study was performed which exhibited a notewor-

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Table 3. Duncan analysis for vessel diameter

Treatment ($MJ \cdot m^{-3}$)	*n*	Vessel Diameter (μm)		
		1	2	3
Control	20	170.5	–	–
600	20	–	225.0	–
360	20	–	247.0	–
480	20	–	253.5	–
720	20	–	255.5	–
840	20	–	–	299.0
Significance		1.0	0.09	1.0

n – number of replicates, significance at P = 0.05, vessel diameter 1, 2, 3 – three different subsets formed by Duncan's analysis for different values of the vessel diameter

thy increase in vessel diameters compared to the control samples after treatment. The ANOVA table for vessel diameter showed a significant improvement over the control samples which aided in creating pathways within the wood microstructure and improved retention and permeability.

To further investigate the data, Duncan's subsets were formed (Table 3). From the subsets it can be inferred that the control and treated specimens were statistically different from each other and with increasing MW exposure the diameter increased proportionally. Maximum diameter of 299 μm was obtained in samples exposed to MW, which also enhanced the retention as the flow through these vessels increased significantly. From Fig. 2 it can be observed that the degree of occlusion literally diminished and the vessels became clearer, which helped in increasing the permeability and resulted in significantly higher retention.

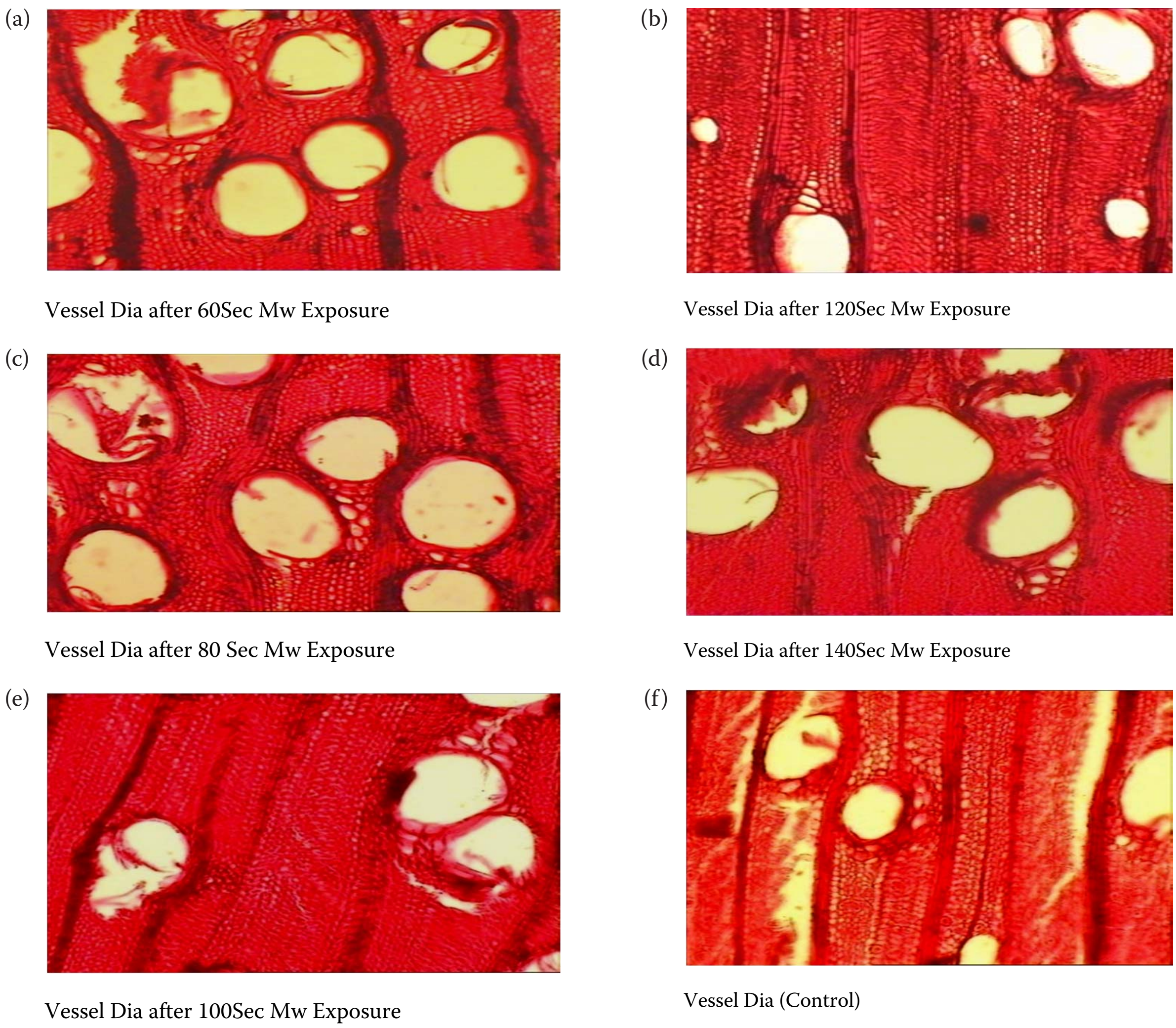

Fig. 2. Vessel diameter of MW exposed (a–e) and control specimens (e), 10× optical microscope magnification factor

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CONCLUSION

MW pre-treatment is useful to ameliorate the performance of refractory wood species and difficult to treat wood species and can be optimized and used at the industry level for pilot trials. The results showed a significant ($P \leq 0.05$) improvement in retention and penetration of preservatives with increasing treatment time as compared to the controls. MW treatment with 840 $MJ \cdot m^{-3}$ exhibited an up to threefold increase in the retention of preservatives in treated samples as compared to the controls. This improvement in retention can be attributed to the improvement in effective vessel diameter due to a reduction in the degree of occlusion and improved permeability of the particular species. The improvement in retention and penetration by this method will automatically improve the treatability class of the timber, rendering it fit for several specific end uses. The results indicate that MW irradiation of refractory species may be tested and applied after optimization of the parameters on a pilot scale for application in wood preservation industries for improvement of wood permeability and treatability to impart substantial service life.

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