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QUANTITATIVE EVALUATION OF MICROWAVE IRRADIATION ON SHORT-ROTATION PLANTATION WOOD SPECIES

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ABSTRACT

The durability of imported timber is a matter of growing concern in the tropical Indian climate, with their refractory nature further adding to the woes with respect to further processing. In the present study, the effect of microwave pre-treatment, exposure time and initial wood moisture content on retention, treatability and cross-sectional anatomical properties of *Tectona grandis* and Southern yellow pine imported from Ghana and South America were evaluated. Water based preservative copper chrome borate (CCB) of 2 % concentration was used for the study. The experimental study in combination with dip-diffusion method returned with significant improvement in retention of about 5-6 folds more than the control sets in Southern yellow pine and *Tectona grandis*. Another set of Southern yellow pine and *Tectona grandis* samples were further treated using a full cell pressure method after microwave, without initial vacuum, which showed similar trends with a 3-4 folds increase in retention over controls. Both experiments returned with significant improvement in the treatability class of *Tectona grandis* and Southern yellow pine. The anatomical analysis was performed using a light microscope with 5 and 10x magnifications on treated and untreated samples of both *Tectona grandis* and Southern yellow pine. The outcome of the anatomical study exhibited improvement in vessel diameters in the treated samples of *Tectona grandis* with a reduction in the degree of occlusion by the presence of tyloses. For Southern yellow pine, checks on micro level and cracks on macro level appeared along with the ray cells and the diameter of the resin canals was substantially expanded which ascertains that microwave pre-treatment ameliorated the flow of fluids in the wood microstructure which improved permeability and resulted in better uptake and penetration.

Keywords: Impregnation, microwave, permeability, southern yellow pine, *Tectona grandis*, treatability, wood durability.

INTRODUCTION

The threat of climate change is looming over the world due to various anthropogenic activities responsible for emissions of Greenhouse gases (GHG) like carbon dioxide (CO_2). The increase of carbon in the form of CO_2 in the atmosphere for the past few decades has resulted in a steep increase in global mean temperature from 1,8 ºC to 4 °C (IPCC 2007, Singh *et al.* 2000). Sustainable forest management and efficient utilisation of the major forest product, i.e. wood, can positively influence CO_2 removals by locking the carbon stored in

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harvested wood products and reduce carbon emissions in the global carbon cycle (UNECE 2008). For woody biomass to act efficiently as a carbon sink, the amount of CO_2 sequestered in growing forests and the pool of long-lasting wood products must be acceptably larger than the amount of CO_2 released by decomposition and combustion (Flugsrud *et al.* 2001). Due to its natural origin, aesthetic appeal, excellent workability and renewability, wood has gained the attention of the stakeholders and promoted as an eco-friendly construction material in present days across the globe. Recently, wood modification is the most sought-after field in the niche sector of wood science and technology as the dwindling supply of many durable wood species worldwide has compelled industrialists and other patrons to look for alternative species of non-durable nature. Living Forests Model predicted global wood removals of 7,168 billion m^3 in 2030 and 11,356 billion m^3 in 2050 (WWF 2012) which establishes the demand for woody biomass worldwide. The demand for wood-based panel products and furniture from India is on the rise, due to optimal labour cost and less expensive production process but the gap between demand and supply of raw material is advancing meteorically (Ganguly 2018) and managed by import. Asia Pacific region had a net import of 36 million m^3 in 2016 (FAO 2017) and India's industrial demand for wood was predicted to reach 150 million m^3 by 2018 (AHEC 2016). India, being a major importer of round logs and other allied products had an estimated import worth $2 billion in the past decade (Sood 2019) with logs estimating to about 74 % of total imported forest products (Montiel 2016, AHEC 2016**)**. Hardwood species of *Tectona grandis* (TG) and meranti along with softwood species like Southern yellow poplar (SYP) are mostly imported from Malaysia, United States of America, Myanmar and New Zealand **(**Sood 2014). However, the performance of such imported wood and allied products for several ends uses calls for attention as several imported species perform rather dubiously in the tropical climate of the Indian subcontinent (Sundararaj *et al.* 2015). This may result in early and frequent replacement of the wood in use which may not be economically and ecologically feasible (Samani *et al.* 2019). Hence, research on wood modification nowadays in India primarily focuses on several imported and indigenous species of lower durability and enhancing their performance in service (Ganguly and Tripathi 2018, Ganguly *et al.* 2020, Hom *et al.* 2020a, Samani *et al.* 2020, Hom *et al.* 2020b, Saha *et al.* 2020).

Additionally, wood in its service span is often exposed to harsh climate (Cheung 2019) and thus, despite being a good building material, it has certain limitations that restrict its extensive use outdoors. Biodegradation of wood in its natural form is elementary but needs to be controlled significantly in service (Kutnik *et al.* 2014). Wood preservation is the most convenient and efficient method to impart a substantial life span to timber and timber products. However, the extent and execution of this method vary from species to species due to their refractoriness and treatability indices. Poor treatability of wood often results in moderate uptake of treating solutions but meager penetration which fails to address the purpose. To make amends with this nuisance, several wood modification techniques were developed and are being explored to facilitate uptake of treating chemicals. Present day research is primarily focusing on eco-friendly modification practices that limit or reduce excessive use of wood preservatives and frequent replacement of wood in use.

Microwave modification is an eco-friendly technique that reduces energy consumption (Sethy *et al.* 2016) and aids in several wood processing operations such as seasoning and preservative treatment by significantly improving wood's permeability and preservative uptake, resulting in its effective use for a longer period in service. Previous research on microwave (MW) showed a positive impact on retention and penetration of wood pertaining to moderate and high refractory index with different chemicals and catalysts (Samani *et al.* 2019, Ganguly and Tripathi 2018, Vinden *et al.* 2017, Terziev and Daniel 2013, Sethy *et al.* 2012, Dashti *et al.* 2012, Torgovnikov and Vinden 2009). Rapid heating of wood microstructure during MW modification results in delamination of the weak anatomical structures like ray cells and parenchyma, resulting in better connectivity of free space in the capillary system and in return an easier fluid flow (Dömény *et al.* 2014). Using optimum to severe levels of to MW intensity, improvement in permeability in radial and longitudinal direction up to several thousand times in comparison to the untreated samples can be achieved (Liu *et al.* 2005, Torgovnikov and Vinden 2010, Vinden *et al.* 2011). Torgovnikov and Vinden (2010) further highlighted the importance of initial moisture content (IMC) of wood prior MW modification. The micro cracks formed in wood during the treatment are basically due to the high-pressure gradient of the vapour, which is generated during MW heating. Green wood performs better in this case in comparison to wood having very low moisture content (MC). MW was explored by Gašparik and Gaff (2013) for wood plasticizing and they also reported the importance of higher MC for this intense process which was later endorsed by Gašparik and Barsik (2014). Further, in some preliminary laboratory trials it has been found that wood having very low MC can be heated upto 170 °C after MW modification which may result in some chemical modification, slow pyrolysis of the chemical constituents of wood and charring. The char formed by microwave heating has a specific surface area of approximately 450 m^2/g (Miura *et al.* 2004). Hence, selecting the modification parameters based on optimum MC is of high importance.

Anatomical changes after any physical or thermal wood modification is fundamental and well understood. Similarly, MW wood modification also results in a significant change in wood microstructure as reported by several researchers (Hong-Hai *et al.* 2005, Jiang *et al.* 2006, Torgovnikov and Vinden 2009, Li *et al.* 2009, He *et al.* 2014, Samani *et al.* 2019) although, in case of moderate modification intensities or species with better structural integrity it may not be evident (Vongpradubchai and Rattandecho 2009).

Based on the above cited literature and findings, the present study was designed to assess the impact of MW modification on retention, treatability and anatomical properties of imported TG and SYP. The species were considered keeping in mind their potential in the context of Indian wood industries in the future and their increasing import to India of late. One refractory hardwood species which is known to have caused hindrance in preservative treatment and one softwood species that was easy to treat were taken to assess the difference of treatment parameters.

MATERIALS AND METHODS

Sample preparation

Seasoned planks of SYP and TG imported from South America and Ghana respectively, were procured through local vendors. The study was executed at the Wood Preservation Discipline of Forest Research Institute, Dehradun, India. The planks were subsequently converted into cube samples of side length 3,5 cm (dimension 3,5 (Longitudinal) x 3,5 (Radial) x 3,5 (Tangential)) cm. Relatively straight grained samples free from any visual abnormalities were selected from the same part of the board and were considered for the study to obtain optimum results and to optimize variability in the data. Samples contained both sap and heartwood portions. The samples were conditioned at 20 °C ±2 °C and 85 % RH in a conditioning chamber for 14 days prior experiment. In total 188 samples (94 each of TG and SYP) were considered for the study (Table 1). Each set of experiments had 10 replicates whereas for anatomical analysis 2 replicates per set were considered. For the oven drying (OD) set, samples of each species were selected from the lot at random and IMC of the samples were determined on an OD basis as per (IS 11215 1991). The IMC (with standard deviation values in parenthesis) of TG was found as 42,0 (±0,8) % and that of SYP was 40,8 (±1,52) % prior commencement of the experiments.

Table 1: Sample distribution.

MW Energy (MJ/m^3)	TG	SYP	Total
1200	20	20	40
1350	20	20	40
1500	20	20	40
Controls	20	20	40
Oven Drying	10	10	20
Anatomical Analysis (Untreated/ Control)	2	2	4
Anatomical Analysis (Treated at 1500 MJ/m^3)	2	2	4
Total	**94**	**94**	**188**

Microwave pre-treatment and preservative impregnation

MW modification was carried out in a kitchen MW device (Model 30SC3, IFB Industries, India) with frequency 2,45 GHz and maximum output power of 900 W. Treatments were defined based on power and volume of samples and the energies defined in Table 1 were calculated accordingly (Samani *et al.* 2019). After MW modification, the treated samples were immediately dipped in a test vessel containing a 2 % solution of copper chrome borate (CCB) preservative. Dipping was done for 5min and the samples were then taken out of the chamber, dripping preservatives were soaked with a tissue and the mass was recorded. Another set of samples from both species were further subjected to pressure impregnation after MW at 1034212,5 N/m^2 (10,3 bar) for 2 h without initial vacuum, although, a final vacuum for 15 min was applied. Retention (kg/m^3) was checked for an absorbed quantity of the preservative on a wet weight basis as per (IS 401 2001) using the following Equation 1:

$$Retention\ (kg / m^3) = \left(\frac{GC\ x\ 10}{V} \right) \quad (1)$$

Where,

G = Weight of the treating solution absorbed by sample in kg;

C = Concentration of treating chemical (%) and V = Volume of the specimen, in m^3.

Treatability evaluation

After preservative treatment, specimens were allowed to dry in a controlled chamber with 20 °C $\pm$2 °C temperature and 50 % $\pm$ 2 % relative humidity (RH) for better fixation of the preservative chemicals. After 21 days of conditioning, both treated and untreated specimens were cut into equal halves to detect the presence of copper. The exposed surfaces were sprayed with Chrome Azurol S solution. The spot test exhibited blue color (Figure 1) on cross sections where copper had penetrated and the untreated zones turned red (IS 2753 1991). The percentage area of the treated zone was assessed visually. All four measurements (Figure 1) were averaged to obtain a single penetration value. The penetration data were analyzed as per IS 401 (2001) to determine the treatability class. Treatability class and ICCA was evaluated as per the following representation (Tripathi 2012).

Treatability Class	Treatability Class as per EN 350 (Nearest Approximation)	ICCA (Impregnated Cross Cut Area) Average of four sections	Demarcation
a	1	65-100 %	Very Permeable
b	2	47-65 %	Permeable
c	3	21-42 %	Moderately permeable
d	4	10-15 %	Very resistant
e	4	Nil	Impermeable

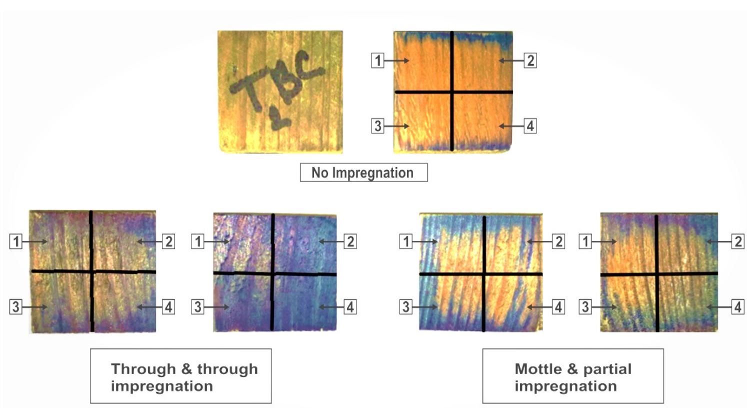

Figure 1: Representative image for treatability evaluation (1,2,3 and 4 represents four sections in each sample).

Anatomical analysis

For anatomical analysis two replicates per species from the control sets and two replicates per species from the set of 1500 MJ/m^3 were chosen. The extreme sets were selected for better understanding of the changes taking place within wood microstructure. The samples were first checked for the orientation of rays and accordingly a cross sectional cutting pattern for preparation of blocks was decided so that the intersection of growth rings remains as close to 90° as possible. Samples were cut perpendicular to the axially oriented xylem cells to avoid over- and underestimation of the measured anatomical features. In the present study, only the cross-sectional features were analysed to assess the impact of MW modification. The selected specimens were first sliced into blocks of smaller dimensions (2 x 2 x 2 cm^3) and were soaked in distilled water for 24 h at least to avoid damage to cell structures when cutting (Von Arx *et al.* 2016, Schneider and Gärtner 2013, Gärtner and Schweingruber 2013, Yeung *et al.* 2015). The samples were further boiled in cycles of 30 min to prepare them for the microtome. Sections were made of roughly 12 µm -20 µm thickness using a Reichert Microtome (Austria, 358926). Heidenhain's haematoxylin and safranin were used for staining the sections and standard laboratory schedule was followed afterwards by passing the section through grades of alcohol (10 % to 100 %), and afterwards putting it in xylene and clove oil (50:50) for making permanent slides. Finally, the sections were mounted in Canada balsam. For SYP the resin canal diameters and for TG the vessel diameters were measured. Twenty observations per slide were made and the mean values are reported.

Statistical analysis

The data were analysed using the SPSS Version 25 package from (IBM 2017) to determine the mean, standard deviation, Pearson correlation and to perform Kruskal-Wallis H test and ANOVA. Mean values were considered to examine significant differences between treatments and Duncan's modified LSD was performed afterwards to examine differences between individual means.

RESULTS AND DISCUSSIONS

Retention

Previous studies revealed that improvement of preservative retention in wood is directly proportional to the applied MW energy and results in increase in retention values (Ramezanpour *et al.* 2015, Ganguly and Tripathi 2018, Samani *et al.* 2019). MW modified wood samples were dipped in the preservative solution immediately after irradiation so as to simulate the vacuum process in the full cell method and assess its effect in preservative retention. This theory has not been previously reported and hence can be explored further in future to optimise treatment cost. It was found that the steam coming out of the sample through numerous micro cracks formed within the wood, facilitated the preservative uptake and retention. For less refractory SYP, the improvement in retention with only 5 min of dipping returned with retention values of about 7 kg/m^3 for the highest MW energy class which was approximately 9 times more than that of the control set (Figure 2).

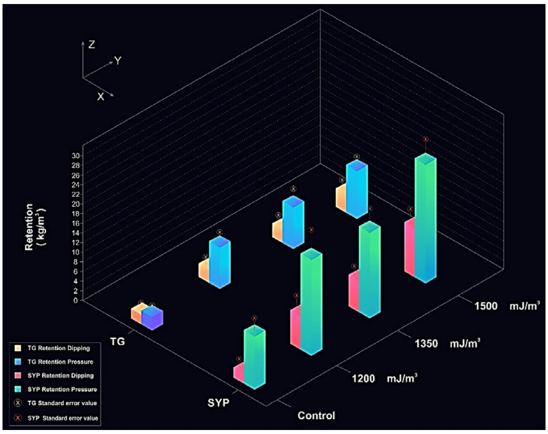

Figure 2: Retention obtained by TG and SYP after different MW treatments and the controls.

Around 6,5 kg/m^3 to 8 $Retention\ (kg/m^3) = \left(\frac{GC\ x\ 10}{V}\right)$ of absorption is recommended for Copper based preservatives for several above ground applications (IS 401 2001) and the same was achieved in the present study by dipping of MW modified wood, with minimal energy consumption and without much effort. Such retention values impart adequate protection to the timber when it is not exposed to conditions with extreme humidity or in direct contact with water. For TG, a similar trend was found although with significantly lesser retention value of 2 kg/m^3 after dipping. TG falls under class "e" as per IS 401 (2001) which explains this outcome**.** However, the retention obtained by TG after 5 min of diffusion treated with highest MW energy was 6 times more than that of the controls (Table 2).

Table 2: Retention values of TG and SYP after diffusion and pressure treatment (standard error within parentheses).

Treatment (mJ/m^3)	Dipping (kg/m^3)		Pressure (kg/m^3)	
	TG	SYP	TG	SYP
1200	1,58 (±0,16) a,b	5,61 (±0,93) c	7,45 (±0,78) n	18,71 (±3,09) o
1350	1,55 (±0,27) a,b	5,77 (±0,36) c	7,51 (±0,55) n	20,62 (±1,26) o
1500	2,18 (±0,20) b	7,17 (±0,66) d	8,67 (±0,24) n	23,91 (±2,2) p
Control	0,35 (±0,02) a	0,78 (±0,05) a	1,59 (±0,09) m	9,94 (±0,53) n

Different alphabets denote different homogeneous subgroups as per Duncan Analysis. Duncan Analyses were done separately for pressure and diffusion and the results are represented with a,b,c and d being the subsets for diffusion retention and m,n,o and p being the subsets for pressure retention.

Dipping as well as pressure treatment resulted in statistically improved retention of all the treatments when compared to the controls as revealed by Duncan Subset in both the species (Table 2) which establishes the overall efficacy of the study. Similar trends regarding uptake of water or treating solutions were observed by Treu and Gjolsjo (2008) and Hong-Hai *et al.* (2005) after MW pre-treatment. With additional pressure, the impregnation values improved further with SYP showing retention values of 19 kg/m^3 to 24 kg/m^3 which was 2-2,5 folds more than that achieved by non-MW modified controls. Both the treatments yielded statistically significant outcomes (Table 3). Wood obtained from fast grown plantation softwood like SYP usually has a high proportion of juvenile wood and less of heartwood hence low natural durability (Hill 2007) resulting in restricted uses in extremely harsh climatic conditions or outdoors which can be alleviated by this method. SYP, treated with MW and impregnated with preservative after pressure, can be explored in severe climatic conditions and may exhibit durability beyond its otherwise specified period of 60 months (IS 401, 2001) which may result in its frequent use**.** Indigenous or imported TG is considered a durable species and falls under durability Class 1 (IS 401, 2001) while plantation grown TG sapwood is moderately durable and falls under Class 3. Under extreme exposure, not much protection is needed for TG timber, however, added retention of preservative is always a plus to enhance the durability of wood in service. TG samples treated with highest MW energy exhibited 4 times more retention values in comparison to the controls. Highest MW exposure yielded retention of approximately 9 kg/ m^3 for TG which may surely be enough to improve its durability substantially beyond the stipulated time range of 120 months in natural form. Table 3 exhibits that the retention of treating chemicals after diffusion or pressure impregnation, has a strong positive correlation with MW irradiation energy. The coefficient of determination (r^2) observed was 0,516 and 0,378 for SYP while 0,491 and 0,573 for TG with regards to dipping and pressure type of preservative treatment respectively, indicating the same. Enhanced retention corresponds to enhanced durability which means less frequent replacement of harvested wood products and thus a sustainable use of the woody biomass, locking the carbon stored.

Table 3: Pearson's coefficient of determination (r^2 values) between the treatments for SYP and TG ($p \leq 0,01$).

Treatment	SYP		TG	
	Dipping	Pressure	Dipping	Pressure
MW modification at different energy level	0,516 (0,718**)	0,378 (0,615**)	0,491 (0,701**)	0,573 (0,757**)

Values in parenthesis represent Pearson's correlation (r value)
**Correlation is significant at the 0,01 level (2-tailed).

Treatability

Inspection was carried out visually after MW modification which highlighted that the control specimens of TG were highly refractory in nature and belonged to class "e" with very little or practically no impregnation. SYP controls were relatively more permeable and fell in class "d" Treatability class of both the species improved significantly after the MW pre-treatment with highly refractory TG being elevated to class "a" from class "e" and SYP to class "a" from class "d" (Figure 4). Tarmian *et al.* (2020) have mentioned that treatability and permeability are strongly correlated and the finding of this study further substantiates their claim as after MW treatment the reduction in the occlusion level in TG and checks and cracks formed in the microstructure of SYP might have positively influenced the permeability of the species **(**He *et al.* 2014, Wang *et al.* 2014) resulting in an easier flow of treating chemicals. The mean ICCA obtained for each set was statistically analyzed using the Kruskal Wallis nonparametric ranking test to assess the significance of MW modification energy. The impact of MW irradiation was statistically evident on treatability and penetration of both the species (Table 4) and the improvements in treatability obtained were in line with similar findings by Ramezanpour *et al.* 2015 and Samani *et al.* 2019 where highest treatability corresponded to the highest MW energy class (Figure 3)**.**

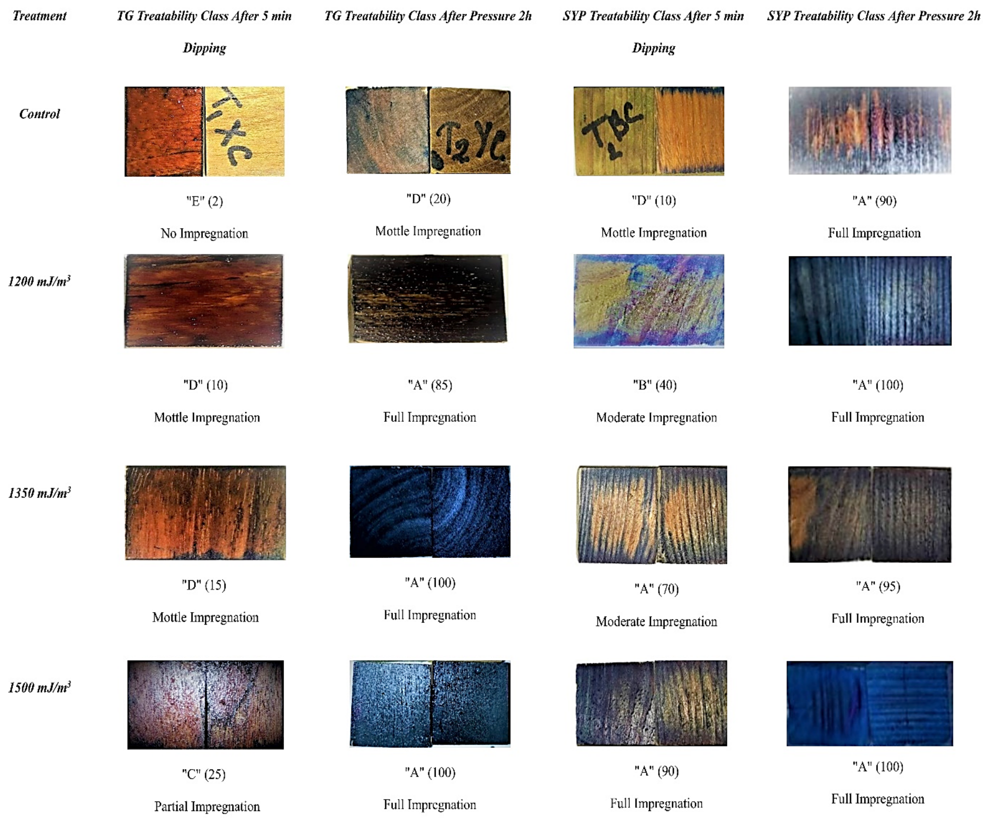

Figure 3: Treatability Evaluation of TG and SYP after Dipping and Pressure Treatment. (Values in Parentheses denote mean penetration percentage/ ICCA of each set consisting 10 replicates. Blue color indicates the presence of Copper. Corresponding photos represent each set).

Table 4 shows a significant difference of MW modification energy on treatability as p value is less than 0,05. A post hoc independent sample test (Figure 4) shows that in case of TG, dipping and pressure impregnation of preservative shows significant difference in treatability. Although, the effect of MW modification was non-significant between different MW energy levels for both dipping and pressure treatments. For SYP, the effect of different MW energy levels on treatability for dipping was significant statistically, while pressure treatment showed no statistical difference (Figure 4). It is also pertinent to mention that, Chromium based preservatives might not perform well in terms of treatability evaluation due to the high reaction rate of chromium with wood and relatively longer (24h) fixation time (Morris et al. 2002, Cooper and Morris 2007) which might have also resulted in the poor retention and treatability of the control samples after subsequent dipping of 5 min. This enhanced treatability of both hard and soft wood after MW pre-treatment can be of particular interest for several wood processing parameters such as coating, pulping, bonding of adhesives and chemical modification of timber (Tarmian *et al.* 2020) where homogeneity in treatment is a must and can be ensured.

Table 4: Kruskal- Wallis H test for treatability.

Test Statistics a,b		
	TG	SYP
Kruskal-Wallis H	73,666	71,564
df	7	7
Asymp. Sig.	0,000	0,000

a Kruskal Wallis Test; b Grouping Variable: Treatment.

Anatomical analysis

The enhanced treatability and retention were substantiated further by the finding of anatomical cross-sectional analysis of treated and untreated specimens of both the species. For TG, refractoriness can be attributed to the degree of occlusion in its vessels which is often fully or partially choked with tyloses (Figure 5A). The presence of these vessel inclusions in teak, often makes it extremely difficult to treat with preservatives prior to use. MW pre-treatment, because of its typical wood modification acumen, can prove to be an efficient solution to these hindrances. It can be hypothesized that the high steam pressure with the fast-moving steam, generated within wood during the process, may flush out all such inclusions completely or partially from the core to the periphery. These pressure gradients developed during the treatment, can improve the effective vessel diameter (Samani *et al.* 2019) and aid in fluid and vapour flow through the entire volume of the test specimen in all three major axes (Torgovnikov and Vinden 2009). It was found that the effective mean vessel diameter (EMVD) of TG samples (Figure 5J) improved significantly ($P \leq 0,05$) in comparison to the untreated samples (Figure 5I). EMVD of the treated samples was recorded (standard error values in parenthesis) as 259 ($\pm$ 5,75) µm whereas the same for the controls were 186,5 ($\pm$ 6,17) µm. Apart from this obvious change (Figure 5J), the anatomical appearance changed drastically with minor cracks (Figure 5B) and significantly clearer vessels (Figure 5F), free from vessel inclusion after MW. Anatomy of SYP also altered due to the treatment with resin canals showing a clearer appearance and visible cracks and checks along the weak ray cells which might have facilitated the fluid flow. Mean canal diameter of untreated controls were 112,5 ($\pm$ 6,17) µm which was statistically different from 266,25 ($\pm$ 5,77) µm for the modified set. The results of the anatomical study were in conformity with Weng *et al.* 2020 and He *et al.* 2014.

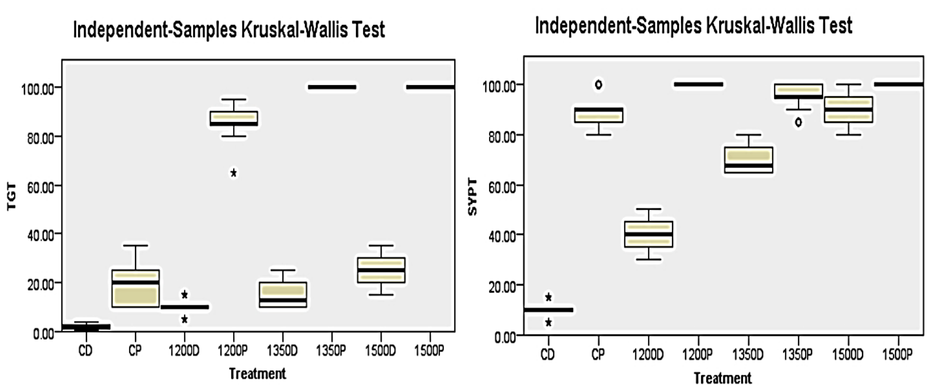

Figure 4: Kruskal-Wallis analysis for treatability of TG and SYP (TGT= TG Treatability, SYPT= SYP Treatability, D= Dipping, P= Pressure, C= Control, 1200, 1300, 1500 = MW Energy in mJ/m^3).

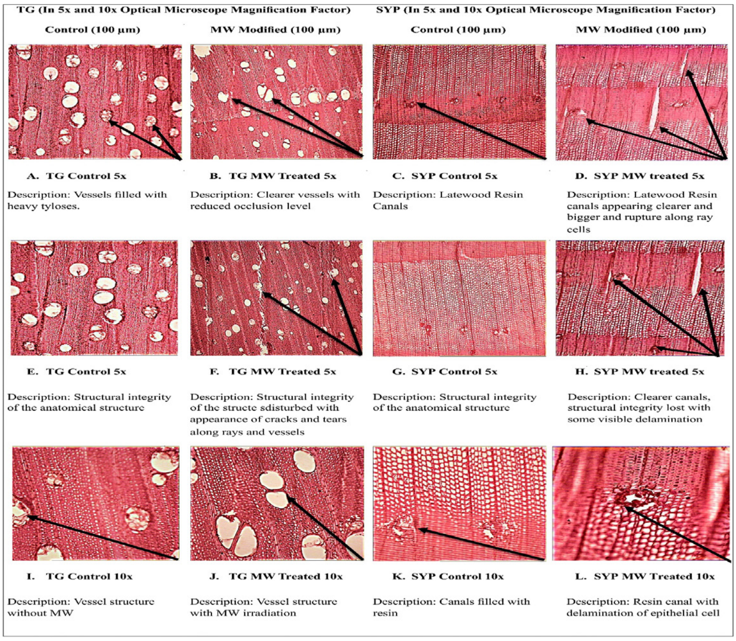

Figure 5: Anatomical Structure of MW treated and untreated samples of TG and SYP. (A-B-C-D-E-F-G-H-I-J-K-L).

CONCLUSIONS

MW wood modification is a potential tool to improve the treatability and permeability of several timber species of non-durable and refractory nature, ensuring their longevity and reducing frequent replacement in service. This maintains the carbon storage potential of harvested wood products and increases the possibilities of carbon dioxide sequestration by plantation. In addition, this method holds promise to reduce treatment time and energy consumption for the otherwise extensive wood processing techniques which can be immensely profitable and of particular interest for the small-scale industrialists. This process also ensures higher retention in relatively shorter exposure times which is established in the present study with water based preservative CCB. The anatomical structure of both soft and hardwood showed some delamination although the magnitude of the distortion varied between species mainly due to their inherent structural integrity. This phenomenon in particular must be considered prior to expose MW modified wood for extensive structural application as this may lead to some reduction in strength. However, this reduction in strength may be cosmetic if the treatment parameters are optimised.

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