

IMPROVING PRESERVATIVE RETENTION AND PENETRATION OF IMPORTED *TECTONA GRANDIS* USING MICROWAVE TREATMENT

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Abstract:

Wood samples of imported *Tectona grandis* (TG) were exposed to Microwave pre-treatment with the aim to ameliorate its retention and penetration. Throughout the experimental set up, microwave frequency was kept constant and samples were exposed for different treatment times. Samples afterwards were subjected to diffusion and then pressure impregnation with 4% solutions of Borax: Boric Acid (BBA) and ZiBOC. MW treatment aided in distribution and uptake of the treating solution as the treated samples exhibited mean retention of 8.1 Kg/M³ and 9.2 Kg/M³ respectively for BBA and ZiBOC which were 64% to 75% more than that of the control. MW treatment further reduced processing time, improved flow of treating solution within wood microstructure and subsequently upgraded the treatability of class of refractory *Tectona grandis* from class "e" (Heartwood very refractory to treatment) to "a" (Heartwood easily treatable).

Key words: microwave; impregnation; treatability; *Tectona grandis*; borax: Boric Acid (BBA); ZiBOC (Copper Zinc Borate); retention.

INTRODUCTION

Teak (*Tectona grandis* Linn. f.) is known for its aesthetically appealing nature and uniform colour (Golden yellow, brown colored heartwood), grain, strength, natural durability and good machining properties (Kaewkrom *et al.* 2005; Dungani *et al.* 2012) which make it suitable for several utilizations such as boat and shipbuilding, veneering, furniture, cooling towers and other decorative uses (Dungani *et al.* 2012). Indigenous teak falls in class I as per IS 401 (2001) and exhibits excellent durability in Indian climatic conditions. It is well documented that not much treatment is required prior putting indigenous teak to any specific end use, therefore, even today when there is a scarcity of timber in India, stakeholders are inclined towards imported timber of high commercial importance like teak. India is currently facing an acute shortage of timber with a stark contrast between demand and supply (Ganguly 2018). Majority of the industrial need in India is met by plantation species and there is a substantial gap between consumption and supply which forced the manufacturers to look for some alternatives. This situation has prompted frequent use of imported species of lower durability (Ganguly *et al.* 2020) which is evident from the study of Keerley (2019) who had estimated the value of wood and wood products imported in India as about 388.5 billion Indian Rupees (INR) in 2018 with the major exporters being China, USA, UAE, Ghana and New Zealand. Although, huge quantity of timber is being imported, even then the need is not fulfilled as not enough data are available on its durability resulting in frequent replacement of the timber in use. Hence, present day research on wood modification in India is primarily aimed at imported and indigenous species with lower durability (Hom *et al.* 2020a, 2020b, Samani *et al.* 2020).

The performance of any imported timber in different climatic conditions varies with the climate of that particular region. Hence, it becomes necessary to test the timber according to various standard specifications to find the most durable form (Sundararaj *et al.* 2015). The service life of any treated timber depends on the type of preservative, its distribution, penetration, retention and fixations. Previous studies on imported Meranti of genus *Shorea* of Malaysian origin, exhibited that the wood is non-durable and refractory in nature (Tripathi 2012). Similar studies on imported teak is not reported thus far from India. Since TG is a

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refractory timber species, to boost its impregnation and enhance its treatability with preservative solutions, some suitable pre-treatment method, other than the conventional practices, with relatively lesser processing time, needs to be explored.

Several studies on MW pre-treatment have reported in favor of improving retention and penetration of numerous timber species of moderate and high refractory index (Torgovnikov and Vinden 2009; Dashti *et al.* 2012; Ganguly and Tripathi, 2018; Samani *et al.* 2019). In MW modification, wood gets heated very rapidly and steam pressure generated within the wood microstructure, ruptures the weak ray cells and pit membranes that creates pathways for an easier flow of fluid or gas, thus increasing its permeability (Vinden *et al.* 2011; Samani *et al.* 2019). Torgovnikov and Vinden (2010) highlighted the importance of optimization of MW energy with a focus on the species and level of modification and reported the optimum level of energy to be within the range of 250-1200MJ/M³. Liu *et al.* (2005), Treu and Gjolsjo (2008), Xu *et al.* (2015) through their studies and findings have already established the efficacy of MW pre-treatment for improving fluid flow in wood and its enhanced preservative uptake. In addition to increased uptake and better penetration, Samani *et al.* (2019), reported significant changes in the wood anatomical structures of MW modified *Melia composita* wood which exhibited increased vessel diameters post MW treatment. The study is in conformity with previous findings by He *et al.* (2014), who reported generation of micro pores and increment in pore diameters along with destruction of pit membranes and damage to cell walls which might have resulted in the decrease in mechanical strength of the chosen species, although, the reduction in strength was not statistically significant which establishes the overall efficacy of the process.

In view of the above cited literatures, MW pre-treatment was studied on imported TG from Ghana with an aim to establish a two-step combination of diffusion and pressure treatment post MW modification as an alternative to conventional methods for treating refractory timbers. The study primarily focuses on the enhancement of preservative retention and penetration after the treatment and elucidating further on the impact of exposure duration on moisture loss and other process parameters involved.

MATERIALS AND METHODS

Materials

Wood Samples

Experimentation was carried out at Forest Research Institute, Dehradun, India. Planks of TG from Ghana, aged approximately 20-25 years, were converted into 100 specimens of size 3.5cm(Width)x3.5cm(Thickness)x15cm(Length) with mixed sap and heartwood. Relatively straight grained and defect free specimens free from insects, borers, termite attack or any other notable microbial contamination were selected.

Preservatives Used

1. Borax: Boric Acid (BBA) at 4% concentration. (20000 mg Borax and 20000 mg Boric Acid dissolved in 1 litre distilled water).
2. ZiBOC (Copper Zinc Borate) at 4% concentration (Tripathi 2013).

Reagents

Spot Test of Boron by Qualitative Method for samples treated with BBA

Post treatment, the specimens were dried and were cut into two equal halves for spot tests. The exposed surfaces were sprayed with turmeric solution, prepared with 2% turmeric powder in rectified spirit to indicate the presence of Boron (IS 2753:1991).

Spot Test of Copper by Qualitative Method for samples treated with ZiBOC

Similarly, for samples treated with ZiBOC, exposed surfaces were sprayed with Chrome azurol S solution, prepared by dissolving 0.5gm Chromeazurol-S and 5gm of Sodium Acetate in 80ml water and further diluting to 100ml (total quantity) to indicate the presence of Copper (IS 2753: 1991).

Methods

Initial Moisture Content (IMC)

Prior experiment, moisture Content of the sample lot was determined on Oven Dry basis (IS 11215:1991) and was found as 23.60 (± 0.8)%.

Moisture loss determination

Percent moisture loss caused by MW treatment was calculated from the weight of the blocks before and immediately after MW treatment.

$$\text{Moisture Loss (\%)} = \frac{(W1-W2)}{W1} * 100$$

where: W1 and W2 are the weights of each specimen before and after MW treatment respectively.

Microwave Treatment

MW treatment was carried out in a discontinuous laboratory microwave heating device (model: 30SC3, IFB Industries, India) at frequency 2.450GHz and maximum output power of 900W with power density (intensity) of 2300W/Cm².

Total eight MW treatments were carried out and two sets were kept as controls. Specimens received MW treatment at four time intervals: a) 1 Min b) 2 Mins c) 3 Mins and d) 4mins.

All specimens (except the controls) were immersed in the preservative solutions immediately after being exposed to MW frequencies. Weight of each sample was noted before and after MW treatment for further considerations. Samples were later exposed to full cell pressure treatment without the initial vacuum.

Treatments Performed

Treatments for BBA	Specifications	Treatments for ZiBOC	Specifications
C1	Controls	C2	Controls
T1	MW for 1 minute	T6	MW for 1 minute
T2	MW for 2 minutes	T7	MW for 2 minutes
T3	MW for 3 minutes	T8	MW for 3 minutes
T4	MW for 4 minutes	T9	MW for 4 minutes

Replicates

10 for each set of treatment (T=Treated and C=Control), total 100 samples were taken.

Impregnation with treating solution and determination of retention

The MW treated specimens and controls were pre-weighed (W1) and soaked in BBA and ZiBOC solutions for 6 hours. After each treatment, the samples were blotted with filter paper and specimens were weighed (W2) to determine preservative uptake (IS 401:2001). Controls did not receive any MW radiation. The treated samples and the controls were further subjected to pressure treatment of 100 psi for one hour in pressure treatment plant and kept immersed in the solution for 16 hours in the treatment cylinder post pressure treatment without any applied pressure. Specimens were subsequently dried for 21 days for proper distribution and fixation of preservatives and conditioning of specimens. The amount of preservative solution retained by specimens (retention value R in Kg/m³) was calculated as per the given formula.

$$\text{Retention(R)} = \frac{(GC * 10)}{V} \text{ kg/m}^3 \quad (1)$$

where:

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

C = Concentration of treating chemical (%);

V = Volume of the specimen, in cm³.

Measurement of Penetration and Treatability Evaluation

Specimens were kept in a desiccator for 21 days for subsequent mobilization and fixation of treating chemicals. After that samples were taken out, kept in room condition for 24h and, were cut into two equal halves for treatability evaluation. The exposed inner cross-sectional surfaces were sprayed with Turmeric solution to indicate the presence of Boron (C1, T1-T4) and Chrome Azurol S solution to indicate the presence of Copper (C2, T6-T9). The spot tests exhibited reddish brown and blue color (Fig. 1) on cross sections exhibiting presence of Boron and Copper respectively with the untreated zones returning yellow and red colors (IS 2753:1991). The percentage area of the treated zone was calculated visually as shown in Fig. 1. All four measurements of one cross section (Fig. 1) were averaged to obtain a single penetration value. The penetration data were analyzed as per IS 401:2001 to determine the treatability class. Evaluation was performed as mentioned by Tripathi (2012), where different treatability classes are defined as per total area covered by color development on 3.8*3.8cm² cross section as follows.

Class	Equivalent EN 350 Classification (Approx.)	ICCA (Impregnated Cross Cut Area)	Demarcation	Depth of Penetration (mm)
a	1	65-100%	Very Permeable	13-19
b	2	47-65%	Permeable	9-12
c	3	21-42%	Moderately permeable	4-8
d	4	10-15%	Very resistant	2-3
e	4	Nil	Impermeable	Nil

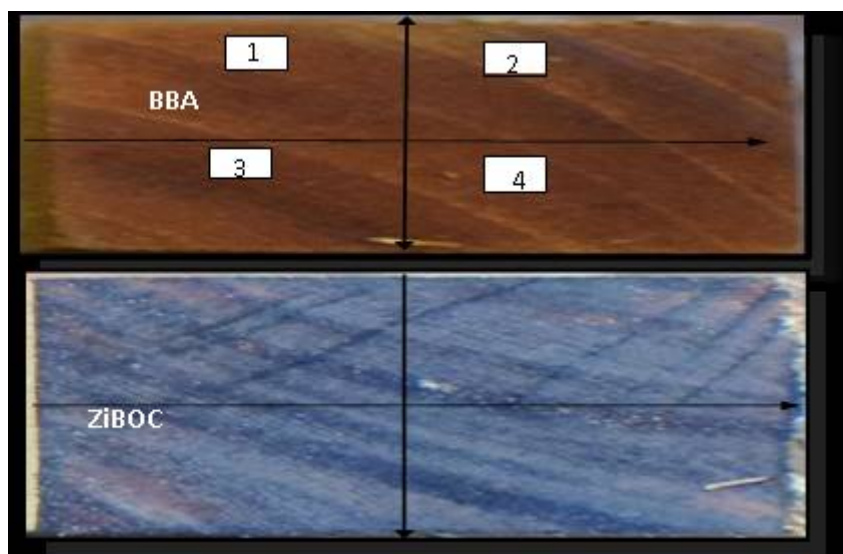


Fig. 1.

Spot Test of Samples treated with BBA and ZiBOC (Cross section of 3.5*3.5cm² divided into four equal sections denoted by 1,2,3 and 4) showing through and through penetration.

Statistical Analysis

The data recorded were subjected to statistical analysis to find out the variation between the treatments/factors and the relationship between the observed parameters. The data were analyzed using “SPSS” (Version 16) package to determine the mean, standard deviation and ANOVA. Mean values were considered to examine significant differences between treatments and the Duncan's test was performed afterwards to examine differences between individual means.

RESULTS AND DISCUSSIONS

MW exposure during the study, did not extensively change the physical appearance of the samples till 4 minutes of exposure. Some micro cracks and checks appeared in the cross section and radial surface which may have helped in improving wood permeability and aided in preservative treatment. These findings are in conformity with the studies of He *et al.* (2014) and Samani *et al.* (2019). There was no charring and no visible change in color of the wood specimens (Fig. 2). Beyond this time period, just for trial, another set of samples were exposed for 5mins to MW radiation and those samples exhibited clear signs of charring (Fig. 3). This can be attributed to the low IMC of the samples which was well below its fiber saturation point (FSP 28-32%). Several researches have previously reported highest efficiency of MW modification on freshly felled timber or when the timber was in green condition (well above FSP) and also highlighted the need to optimize MW process parameters (Torgovnikov and Vinden 2010). The findings of this study also supports the claim and from the result it is evident that process parameters need to be closely monitored and standardized in relation to the IMC of wood. Internal charring and burning of wood caused by prolonged MW exposure can result in significant damage to wood in structural applications and hence treatment time of 5mins was excluded from the study. Additionally, it is very pertinent to mention that the formation of micro checks and cracks could have negatively impacted the surface textures of the treated specimens and it can prove to be an interesting objective to study for the researchers of this field in future.

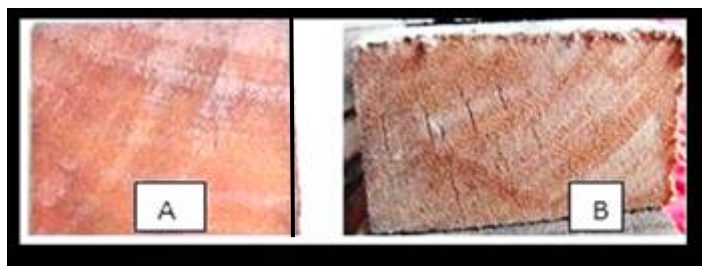


Fig. 2.

Wood Samples after MW exposure up to 4 mins (A. Overall Appearance, B. Micro cracks).



Fig. 3.

Samples after 5 mins of MW Exposure (extreme exposure resulting in internal charring and burning).

During MW treatment, timber loses moisture in the form of evaporation due to heating which is caused by the oscillation and rapid movement of free and bound water in the wood microstructure. Torgovnikov and Vinden (2010) reported the occurrence of steam explosion, caused due to the internal pressure build up which is in line with this theory. In the present study, it was observed that MC loss is directly proportional to the duration of MW exposure which has statistical significance (Table 1). Exposure to MW irradiation up to 4 minutes resulted in moisture loss up to 1% of the total mass of each sample and it is well documented that lower MC% in wood results in better uptake, distribution and fixation of the treating chemicals.

Table 1

ANOVA Table for moisture loss of TG after MW treatment

Source	Sum of Squares	df	Mean Square	F Value	Sig. (p value)
Treatments	9.17	7	1.31	20.77	0.00
Error	4.54	72	0.06		
Total	34.61	80			

It is evident from Figure 4 that all the treatments yielded satisfactory results in terms of preservative retained. ZiBOC showed slightly better performance over BBA and statistical analysis proved the significant impact of MW modification in terms of retention of both the preservatives (Table 2). The reason of a tad higher mean retention with ZiBOC could not be entirely elucidated considering the fact that both the preservatives were of similar nature (water based) and used in same concentrations. However, this phenomenon can be partially attributed to the anisotropy of wood. The retention values obtained by preservative treated samples were in line with previous studies (Ganguly and Tripathi 2018).

Table 2

ANOVA Table for mean retention

Source	Sum of Squares	df	Mean Square	F Value	Sig. (p value)
Treatments	133.677	9	14.853	32.327	.000
Error	36.756	80	.459		
Total	4579.924	90			

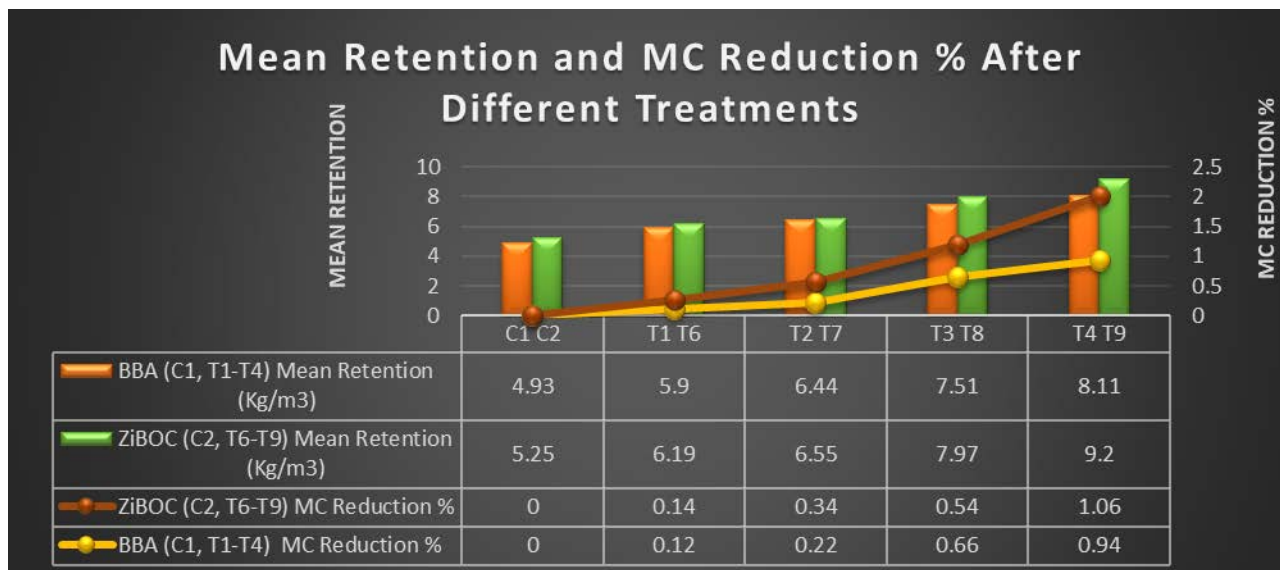


Fig. 4.
Mean Retention and MC% Reduction.

In treatments T1, T2, T3 and T4 respectively 20%, 31%, 52% and 64% increase in preservative uptake was observed (T4>T3>T2>T1) in comparison to the control C1 (Fig. 4). Whereas in treatments T6, T7, T8 and T9 similar trend for retention was found as T9>T8>T7>T6 and the increase was respectively 18%, 25%, 52% and 75% with respect to the control C2 (Fig. 4). According to IS 401:2001, wood possessing 4.93Kg/M³ and 5.25Kg/M³ retention is suitable for interior uses whereas after MW treatment of highest exposure, retention values obtained for BBA and ZiBOC treated samples were respectively 8.11Kg/M³ and 9.2Kg/M³ which ascertains its usage in exterior conditions under shade. Maximum mean retention (9.2Kg/M³) was observed for samples in T9, which had the maximum level of MW exposure (4 minutes), however the retention for treatment groups T2, T6 and T7 and groups T3, T8, and T4 returned statistically similar results. The analysis of variance also indicated that the treatments (Control and Treated Specimens) were significant (Table 3) other than T1 which was comparable statistically ($p \leq 0.05$) with C2 (Table 3) and the inference was drawn from Duncan's subsets (Table 3). The control samples, which were subjected to preservative treatment without receiving the MW exposure, showed significantly different (less) retention than the treated ones which confirms the efficacy of the process overall. The mean retention ($p < 0.05$) of the specimens were calculated and statistically analysed at 5% significance level.

Table 3

Duncan's Table of Homogeneity for Retention Values (Different alphabets denote different homogeneous subsets)

Test	Treatments	Retention
		(Kg/M ³)
Duncan (Different alphabets denote different homogeneous subsets)	C1	4.93 ^a
	C2	5.25 ^{ab}
	T1	5.90 ^{bc}
	T6	6.19 ^c
	T2	6.44 ^c
	T7	6.55 ^c
	T3	7.51 ^d
	T8	7.97 ^d
	T4	8.11 ^d
T9	9.20 ^e	

Extent of penetration was studied in the treated samples using the spot test as described in the materials and methods part. Visual inspections after spot test highlighted the fact that the control samples were highly refractory in nature and hence belonged to treatability class "e". These however were converted to easily treatable class "a" timber after being treated with MW. This confirms the fact that MW wood modification enhances its treatability and impregnability and enhances its preservative retention manifold, which is in accordance to the result found by previous researchers (Samani *et al.* 2019) however after similar

set of trails He *et al.* (2014) reported reduction in mechanical strength of wood which was not found significant. The treatability classification was based on the criteria of preservative impregnated cross-cut area of specimens. Whenever the treatment data did not suffice and comprehend with the above classification criteria, classification was made to the nearest class.



Fig. 5.
Spot tests on Samples treated with BBA and ZiBOC.

Fig. 5 illustrates that C1, T1 and C2, T6 had almost negligible impregnation (Class “e”) whereas T2 and T7 showed mottle impregnation of both the preservatives, although, it is evident that substantial retention (Kg/M^3) values of 4.96 (C1), 5.9 (T1), 6.44 (T2) and 5.25 (C2), 6.19 (T5), 6.55 (T6) respectively by BBA (C1, T1, T2) and ZiBOC (C2, T5, T6) were attained in all these treatments (Fig. 4). Here, it becomes very pertinent to mention that substantial retention achieved by the treatments mentioned above, in fact, makes these suitable for interior uses as per IS 401:2001, although, impregnation was literally nil as per visual inspection (Fig. 5). Therefore, if some carpentry work is done on these samples, which exhibit sufficient retention because of surface penetration of treating chemicals but no or very little penetration inside the core due to its otherwise refractory nature, it will result in failure of treated wood in service condition and such wood cannot be recommended for constructional and structural purposes either.

Interestingly, T3, T4 and T8, T9, which received higher exposure to MW radiation, exhibited $7\text{-}9\text{Kg/M}^3$ of retention with both the preservatives along with adequate surface penetration of the treating solution. Penetration and retention values shown by these treatments make them fit for external application and use under shade.

Significant improvement of preservative uptake and penetration of treating solution achieved in the study may be attributed to the MW radiation that resulted in de aspiration of pit membranes in treated wood by internal steam pressures, and steam explosion that resulted in formation of micro cracks and ruptured the weak ray cell and other cell wall components cosmetically. All these, in combination to each other, synergistically resulted in up gradation of the treatability class of the species concerned (Table 4).

Table 4

Treatability evaluation of TG after MW treatment

Treatments (BBA)	Treatability Class	Treatments (ZiBOC)	Treatability Class
C1	“e” (8) 4	C2	“e” (9) 4
T1	“e” (10) 4	T6	“e” (10) 4
T2	“d” (20) 4	T7	“c” (37) 3
T3	“c” (30) 3	T8	“b” (50) 2
T4	“b” (45) 2	T9	“a” (65) 1

(Values in parenthesis indicate mean penetration percentage of each set of treatment. Numerical values 1,2,3,4 corresponds to treatability class as per EN 350)

The preservative penetration through the wood specimens improved gradually due to microwave exposure and the improvement was remarkable for the samples that were MW treated for 3 and 4 mins (Table 4). The average maximum percentage of preservative penetration at T4 and T9 were up by 6-7 times compared to the control specimens, in which penetration could not be achieved. The enhanced retention and

penetration synergistically ameliorated the treatability class of TG to class “a” from class “e” after MW treatment.

CONCLUSION

Study exhibited that the MW treatment has improved treatability class of teak from impermeable to very permeable. In imported species it is often difficult to distinguish between sap and heartwood and hence in the present study samples having parts of sapwood and heartwood both were irradiated with MW which yielded satisfactory results. Prolonged MW exposure had negative effects on wood integrity as wood specimens exhibited charring which ultimately affects the strength of wood and hence should be avoided. Shorter exposures followed by dipping facilitated higher uptake of preservative and resulted in improved retention and penetration of two preservative systems into teak samples. The results illustrate the benefits of short MW treatments for improving treatability by means of a double or triple stage treatment. The study further sheds light on the fact that time taken in dipping/full cell process may be reduced after MW pre-treatment for refractory species which can reduce operational cost. The results indicate that this pre-treatment technique developed in the study may be tested and readily applied on pilot scale for application in Indian wood industries for improvement of durability of treated wood which will impart a substantial life span to the products in use.

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