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RESEARCH PAPER

Retention and leachability of organotin(IV)-treated *Hevea brasiliensis*, *Alstonia scholaris* and *Macaranga triloba* wood

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ABSTRACT

Chemical retention subsequently leaching rate of wood preservatives are most important following treatment of wood using preservatives chemical. This study investigated the chemical retention and leaching rate of newly synthesized organotin(IV)-treated three non-durable tropical wood species namely *Alstonia scholaris* (pulai), *Macaranga triloba* (mahang) and *Hevea brasiliensis* (rubberwood). The specific objectives of this study were to determine the retention and leachability of newly synthesized one monosubstituted and two disubstituted organotin(IV) compounds following treatment using full-cell treatment method. Ten 19 mm x 19 mm x 19 mm sized wood cubes of each species were treated with 1% of monophenyltin(IV) (MPT) of the monosubstituted organotin(IV), and dimethyltin(IV) (DMT) and dibutyltin(IV) (DBT) of the disubstituted organotin(IV). Chemical retentions were determined following treatment where all selected woods are gained their weight with the newly synthesized organotin(IV) complexes. The highest retention (10.59 kg m^{-3}) was found in *A. scholaris* which was treated with 1% DMT. Leaching test showed no tin was released after 270 hours where the highest (5.32 ppm) release of tin was determined in DMT-treated *A. scholaris* wood species from 2nd leachate sample.

Key words: Leachability, Organotin(IV), retention, tropical wood

Introduction

In recent years the use of organotin(IV) compounds have been increased due to its diversified activities as well as used as wood preservatives. Although safety and environmental issues limit the use of tributyltin oxide (TBTO) and tributyltin naphthenate (TBTN) wood preservatives to aboveground and industrial applications only. Interest in monosubstituted and disubstituted organotin(IV) compounds is increasing due to their interesting structural features and biocidal properties (Eaton & Hale, 1993).

Wood is one of the most attractive materials because of its complex structure and wide range of applications in the world. It is a complex cellular material of biological origin made up mainly of cellulose, hemicelluloses and lignin (Bowyer et al., 2003).These three polymeric cell wall components are the main factors influencing wood properties. The physical, mechanical and chemical properties of wood can be changed by changing these three cell wall component (Rowell, 2005). Some wood species are naturally more durable which are preferred building and construction materials due to their physical, mechanical and aesthetically pleasing performance. But most of tropical wood species are non-durable or less durable which limit their use to indoor and outdoor applications (Chao & Lee, 2003; Brelid et al., 2000). Wood especially the less durable tropical species such as *Alstonia scholaris* (pulai), *Macaranga triloba* (mahang) and *Hevea brasiliensis* (rubberwood) must be treated with preservatives in order to protect wood from fungi and insects attack. In addition, to provide a long economic service life of the wood for many end uses, preservative treatment indirectly contributes to conservation of forest resource (Eaton & Hale, 1993).

Most of the conventional preservatives causing environmental pollution and a few of them are hazardous to animals and human beings (Onuorah, 2000). The used of wood preservative chromated copper arsenate (CCA) to treat timber for outdoor applications is most widespread throughout the world. Arsenic is the

most toxic of the three components and chromium ions is known carcinogen. Environmental and health concerns with the use of CCA, including possible arsenic exposure to humans have resulted in its use being significantly restricted or limited (Pohleven et al., 2002).

Organotin(IV) compounds are chemical compounds based on tin with hydrocarbon substituent. Trialkyltin compounds like TBTO (tri-n-butyltin oxide) and TBTN (tri-n-butyltin naphthanate) are both trialkyltin compounds and are used as fungicides worldwide (Schweinfurth et al., 1991). Although they are very effective organotin to treat wood but they also not environmentally friendly. Therefore, TBTO is recommended only for aboveground use, such as mill work. It has been used as a marine antifoulant, but this use has been almost eliminated because of the environmental impact of tin on shellfish. However, organotin(IV) continues to be of interest due to their bioactivities potentials.

The consumption of wood has been rapidly increasing year by year due to population increase. In contrast, however, the production of wood has been drastically decreasing. Due to this reason there exists an imbalance between demand and supply of forest product (Tolunay et al., 2008). This has driven researchers to look for alternative low-quality resources, such as non-durable tropical wood, for value-added applications (Cai et al., 2007; Deka & Saikia, 2000). In addition, the declining supplies and rising costs of the durable woods or heavy hardwoods, has created interest in the utilization of lower grade woods such as non-durable or less durable tropical woods, whose usage can be extended by applying proper wood preservatives (Oluwafemi & Adegbenga, 2007; Kazemi, 2007; Ayer et al., 2003; Chao & Lee, 2003). One of the most effective ways is to apply suitable wood preservatives needed to improve low-quality resources in order to meet end-use requirements (Wang et al., 2007; Zhang et al., 2006). In addition, preservatives treatment can improve low-quality wood properties, sometimes making them better timbers (Schneider, 1994).

Huge non-durable tropical wood species are abundantly available in Southeast Asia (Deka et al., 2002; Yalinkilic et al., 1999). In Malaysia, especially Sarawak has the third biggest rainforest in the world. Several tropical wood species are abundantly available in Sarawak but they have not been sufficiently developed and utilized. Therefore, satisfactory use of these types of woods depends on proper treatment using proper preservatives. Besides there is now an increased awareness of the hazards associated with the production and application of wood treatment chemicals and the disposal of treated wood and unused solutions (Eaton & Hale, 1993). For this reason, it is necessary to search for new preservatives which are environmentally friendly and safe to use.

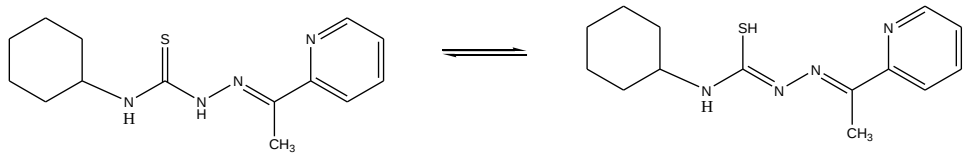
Currently there is no alternative except preservative treatment to increase the service life of many non-durable or less durable and highly susceptible woods. Therefore non-durable or less durable wood species must be treated with proper preservatives to extend their service life. The chemistry of organotin(IV) compounds continues to be of interest due to their interesting structural features and also because of their potentials as agricultural biocides, antitumor agents and other biological activities which are currently being investigated by many researchers (Singh & Kaushik, 2008; Benetollo et al., 2005). In recent years, organotin(IV) compounds have been used extensively as agrochemical fungicides, biocides and antifouling agents (Hanif et al., 2010). Therefore, the current study includes the determine retention and leachability following treatment using selected newly synthesized organotin(IV) compounds of selected non-durable tropical wood species using full-cell treatment method. Thus, the main objective of this study were to determine the retention value and leaching rate of newly synthesized one monoorganotin(IV) and two diorganotin(IV) compounds on *Alstonia scholaris* (pulai), *Macaranga triloba* (mahang) and *Hevea brasiliensis* (rubberwood).

Materials and Methods

Preparation and treatment of wood samples

In this study, three non-durable tropical wood species namely *Alstonia scholaris* (Pulai), *Macaranga triloba* (Mahang) and *Hevea brasiliensis* (Rubberwood) were chosen and collected from an old secondary local forest of Sarawak, Malaysia. The logs were quarter-sawn to 25 mm x 25 mm x 25 mm boards and kiln dried. The boards were further planed, ripped and cut into 19 mm cubes according to the AWPA standard E10-91 (1991). The cubes were then conditioned at 60°C and 70% relative humidity for four days until they reached a constant weight.

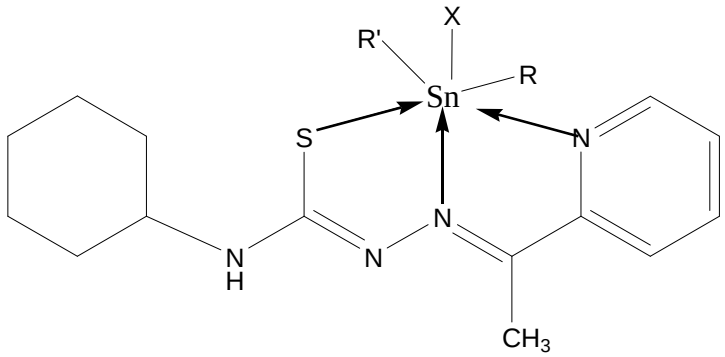
Three newly synthesized organotin(IV) complexes (Affan et al., 2011) were used as wood preservatives. In this study, 2-acetylpyridine-*N*(4)-cyclohexylthiosemicarbazone (APCT) was used as chelating ligand. Structure of 2-acetylpyridine-*N*(4)-cyclohexylthiosemicarbazone (APCT) is given in Figure 1.



Thione form Thiol form

Figure 1. Structure of 2-acetylpyridine-*N*(4)-cyclohexylthiosemicarbazone (APCT) ligand (Affan et al., 2011; Salam et al., 2013).

Structure of mono- and disubstituted organotin(IV) complexes with 2-acetylpyridine-*N*(4)-cyclohexylthiosemicarbazone (APCT) as demonstrated by Affan et al. (2011) is given in Figure 2.



R = Cl, R' = Cl, X = Ph (MPT)
R = Me, R' = Me, X = Cl (DMT)
R = Bu, R' = Bu, X = Cl (DBT)

Figure 2. Structure of mono- and disubstituted organotin(IV) complexes with 2- acetylpyridine-*N*(4) cyclohexylthiosemicarbazone (APCT) ligand (Affan et al., 2011).

One percent concentration of organotin(IV) complexes were prepared for treatment. The organotin(IV) were dissolved in solution of 20% dimethylsulphoxide (DMSO) and 80% distilled water. Treatments were carried out according to the AWPA standard E10-91 (1991). All wood samples were placed inside the beaker containing the treating solution and soaked for 2 hours and then placed inside a vacuum-pressure unit. The treatment schedule was done an initial vacuum of 100 mm Hg for 30 min followed by 100 psi of pressure for 1 hour and a final vacuum of 100mm Hg for 30 min. After treatment, the wood cubes were taken out and the excess treating solutions on the surface of the wood cubes were wiped. The weight of wood cubes after conditioned before treatment (W1) and after treatment (W2) was recorded. The volume before treatment (V1) and after treatment (V2) of the wood cubes was determined using water displacement method.

Determination of chemical retention

The uptake of organotin(IV) complexes by wood were obtained by using weight of wood after treatment (W_2) minus the weight of wood before treatment (W_1). The preservative retentions of organotin(IV) complexes in wood were calculated by the formula below shown in equation 1 according to AWPA standard E10-91 (1991).

$$R = \frac{GC}{V} \times 10 \qquad (1)$$

Where,
R= retention (kg m^{-3})
G = (W_2 - W_1) = net weight gain after the treatment (g)
C = concentration of treating solution (%)
V = Volume of wood cube (cm^3)
W_1 = Weight of cube before treatment (g)
W_2 = Weight of cube after treatment (g)

Leaching test

Evaluation of the release of tin during water leaching

Leaching tests were carried out on cubes treated with 1% MPT, DMT and DBT. Six 1%-treated wood cubes were air-dried in laboratory ambient condition to reach about 10% moisture content prior to the water leaching exposure. Evaporation tests were not carried out due to time constraint and it should be done in future studies. The six cubes were leached with 300 ml deionized water according to AWPA standard E11-97 (1997) procedure. After six hours, the leachate water was removed from each sample and replaced with 300 ml fresh deionized water. The leaching water was exchanged after 1 day, 3 days, 5 days, 7 days, 9 days, 11 days and 13 days intervals for a total of 14 days.

Determination of tin in leachates

The tin level in leachates was analyzed by Flame operation - Atomic Absorption Spectrometer (iCE 3500 Spectrometer, Flame type-nitrous oxide/acetylene). Each leachates were analyzed immediately upon collection. The Spectrometer was adjusted to atomic no. 50, primary wavelength 224.6 nm, emission wavelength 284.0 nm, flame characteristics concentration 0.5 mg/L, fuel flow rate 4.5 L/min and burner height 3 mm for detection of tin.

Data analysis

One-way analysis of variance (ANOVA) was used to determine the differences between mean values of chemical retention of different wood species using different concentrations of chemicals. Analyses were done using statistical program SPSS-18.0 for windows. One-way ANOVA was appropriate in this study because the experiment was carried out in batches. The first batch was experimentation with DPT followed by DBT, DMT, MMT and MPT. In each batch, chemical concentrations and experimental conditions were kept the same. Further analyses of mean comparisons were

done using Tukey HSD test. The factors which were analyzed are types of chemicals and wood species. The dependent variable was chemical retention.

Results

Preservative retention

The preservative retention of *Alstonia scholaris*, *Macaranga triloba* and *Hevea brasiliensis* wood using monosubstituted of organotin(IV) (namely MPT) and disubstituted of organotin(IV) (namely DMT & DBT) compound of each 1% levels of concentration was statistically analyzed and the results are shown in Table 1.

The mean preservative retentions varied significantly among three wood species (Table 1). *Alstonia scholaris* gained higher chemical retention uptake than *Macaranga triloba* and *Hevea brasiliensis* wood. *Hevea brasiliensis* showed least chemical retention uptake than *Macaranga triloba* and *Alstonia scholaris*. Differences in preservatives uptake indicated that treatability differ among wood species. The highest (10.59 kg m^{-3}) and lowest (5.65 kg m^{-3}) retention uptake was observed in *Alstonia scholaris* and *Hevea brasiliensis* wood, respectively. In this study higher retention was observed with disubstituted preservatives (DMT and DBT) in *Alstonia scholaris* and *Macaranga triloba* than the monosubstituted preservatives.

Table 1. Average chemical retention values (kg m^{-3}) in *Alstonia scholaris*, *Macaranga triloba* and *Hevea brasiliensis* heartwood following treatment with mono- and disubstituted of organotin(IV) at 1% concentration.

Treating chemicals		*H. brasiliensis*	*M. triloba*	*A. scholaris*
	N	Chemical retention values (kg m^{-3})		
Monosubstituted organotin(IV)				
MPT	10	5.76 (0.12) a	7.70 (0.11) b	7.90 (0.14) c
Monosubstituted organotin(IV)				
DMT	10	5.65 (0.14) a	9.58 (0.17) b	10.59 (0.15) c
DBT	10	6.05 (0.23) a	8.06 (0.16) b	9.49 (0.20) c

Values in parenthesis are the standard deviation.
MPT-Monophenyltin(IV) complex, DMT- Dimethyltin(IV) complex, DBT- Dibutyltin(IV) complex.
*Means followed by a different letter within a row are statistically different at $P < 0.05$ using Tukey HSD test.
n – Number of sample

Leaching rate of organotin(IV)

Leaching rates were determined to estimate the release of preservative component from 1% organotin(IV)-treated *Alstonia scholaris*, *Macaranga triloba* and *Hevea brasiliensis*. The release rates of tin at each leaching interval from wood cubes treated with 1% MPT, DMT and DBT are shown in Figures 3, 4 and 5, respectively.

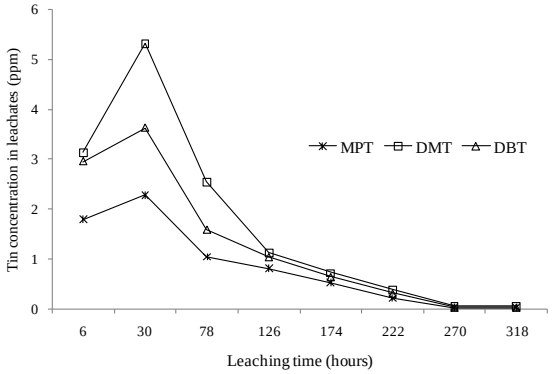


Figure 3. Tin concentration at each leaching interval from *Alstonia scholaris* wood cubes treated with organotin(IV) complexes. The retention of MPT, DMT and DBT are 7.90, 10.59 and 9.49 kg m^{-3}, respectively.

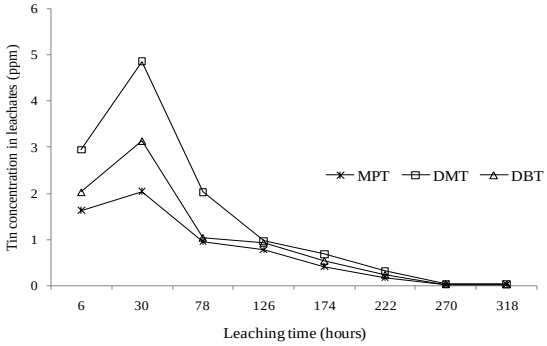


Figure 4. Tin concentration at each leaching interval from *Macaranga triloba* wood cubes treated with organotin(IV) complexes. The retention of MPT, DMT and DBT are 7.70, 9.58 and 8.06 kg m^{-3}, respectively.

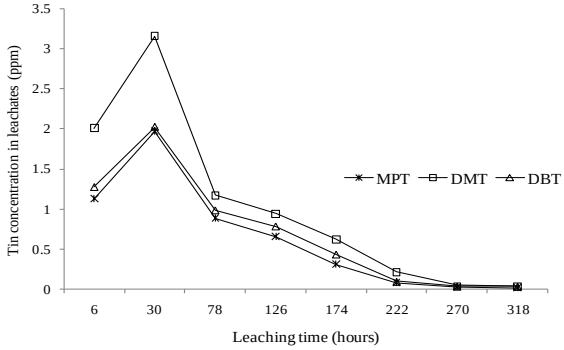


Figure 5. Tin concentration at each leaching interval from *Hevea brasiliensis* wood cubes treated with organotin(IV) complexes. The retention of MPT, DMT and DBT are 5.76, 5.65 and 6.05 kg m^{-3}, respectively.

Leaching result showed that, among all tested chemicals DMT treated wood cubes demonstrated the greatest tin release rate followed by DBT and MPT for the entire leaching cycle. Wood cubes treated with MPT showed the lowest tin release among three tested chemicals. The maximum and minimum tin was determined from 2nd and 8th (last) leachates sample among tested chemicals. Among three wood species, *Alstonia scholaris* showed the highest leaching of tin followed by *Macaranga triloba* and *Hevea brasiliensis*. The highest (5.32 ppm) and lowest (0.01 ppm) release of tin was determined from DMT treated *Alstonia scholaris* and MPT treated *Hevea brasiliensis* wood cubes, respectively.

Discussions

Penetration and retention of organotin(IV) complexes

Penetration and retention extents of treating organotin(IV) complexes in wood sample are crucial to assess the retention of wood preservatives chemical. This study explored whether three newly synthesized organotin(IV) complexes could be retained following penetrate in selected tropical wood species namely *Alstonia scholaris*, *Macaranga triloba* and *Hevea brasiliensis*.

Results presented in Table 1 suggested that organotin(IV) can successfully retained in *Alstonia scholaris*, *Macaranga triloba* and *Hevea brasiliensis*. Results showed that preservative retention for *Alstonia scholaris* was almost twice that of *Hevea brasiliensis* and retention value of wood species is in the decreasing order of *Alstonia scholaris*, *Macaranga triloba* and *Hevea brasiliensis*. This is expected because lower density wood species gain higher amounts of preservatives and vice-versa (Yap et al., 1990). *Hevea brasiliensis* is a treatable timber (Hong & Wong, 1994; Mohd. Dahlan et al., 1994; Hiziroglu, 1997; Jusoh & Kamdem, 2000). This study showed that newly synthesized organotin(IV) were penetrated to the non-durable tropical wood namely *Alstonia scholaris*, *Macaranga triloba* and *Hevea brasiliensis* where highest retention (10.59 kg m^{-3}) was recorded in *Alstonia scholaris* treated with 1% dimethyltin(IV) complex. This is comparable to the retention in *Hevea brasiliensis* treated with 2% copper chrome arsenic type C (CCA-C) after two hours of pressure by full cell method which was 13.0 kg m^{-3} (Jusoh & Kamdem, 2000).

The Malaysian Standard MS 360: 1991 did not specify the use of organotin(IV) to treat wood. However, as a point of reference the specified retention of 8 kg m^{-3} and 12 kg m^{-3} of the CCA preservative is required in the treated timber for above ground use. Retention results from this study (Table 1) indicated that disubstitute organotin(IV) treated wood showed higher retention than that of monosubstitute organotin(IV) complexes. Greaves et al. (1982) observed that treating *Pinus radiate* using 0.1% TBTO resulted in retention of 1.2 kg m^{-3}.

Leaching of organotin(IV)

Leaching is an important part of water-borne preservative performance in treated wood and environmental contamination. Organotin(IV) preservatives contain tin which is impregnated in wood and the majority of tin react with wood substrate to form water insoluble tin oxide (Yin et al., 2007; Mendes et al., 2006). Tin oxide of treated wood is most likely permanent and fix in wood cell wall. Reacted tin would stay in wood cell. However there still remains partial non-reacted free tin component which is leach out to the water. Tin from vessel which is unfix or not reacted most probably leach out to the water. This explain that organotin(IV) preservatives have relatively high tin leaching rate for the first 30 hours, and then the tin leaching rate is substantially declined till 270 hours. After that no tin was detected in the leachates. Results are consistent with the findings of Brooks (2000) and Waldron et al. (2005). They observed that the greatest preservative losses tend to occur in the period immediately after exposure of water following treatment, then decrease with time in service. Zhang and Ziobro (2009) conducted a research study to observe the release of cupric ion ($Cu2^{+}$) from treated wood with copper preservatives. They observed that wood treated with micronized copper preservatives release cupric ion at a higher level than CCA and leaching results indicated that the leaching rate is higher at first 24 hours.

Treated *Alstonia scholaris* and *Macaranga triloba* wood leached relatively higher amount of tin than *Hevea brasiliensis* (Figures 3, 4 and 5). This is expected because high density wood performs low retention and low leaching loss of preservative component. This result is comparable with the findings of Cockcroft & Laidlaw (1978) and Wilson (1971). They pointed out that high density, lower permeability species of wood tend to be more resistant to chemical leaching. Also moisture content influences the rate of preservative diffusion within the wood. Low moisture content increases the diffusion and decreases the leaching (Kaldas & Cooper, 1996; Cockcroft & Laidlaw, 1978). Results are also consistent with the observation of Yap et al. (1990). Chung and Ruddick (2004) pointed out some factors that influence leaching are rainfall or water exposure, temperature, exposure time and sunlight. However, Kennedy and Palmer (1994) also noted that leaching of CCA may be greater from heartwood than sapwood, possibly because the heartwood extractives interfere with the fixation process. Salim et al. (2012) observed that the arsenic losses were significantly higher in some Malaysian tropical hardwoods (MTHs) than in *radiata* pine, except for *Acacia*. High amount of copper and chromium losses were also observed in Geronggang (*Cratoxylon arborescens*), Meranti rambaidaun (*Shorea acuminata*), Putat (*Barringtonia racemosa*) and Ramin (*Gonystylus bancanus*). The high amount of CCA components leached from the treated MTHs could be explained by the deposition of the products derived from

CCA fixation reaction in the cell lumens due to higher extractive levels where they would be more readily accessible to leaching (Cooper, 1991; Srinivasan et al., 1999; Stevanovic-Janezic et al., 2000; Kim et al., 2008). Differences of leaching losses among Malaysian tropical woods are possibly due to the differences in wood chemical composition such as extractive content and the ratio of major cell wall components including cellulose, hemicelluloses and lignin (Yamamoto & Rokova, 1991; Srinivasan et al., 1999). Generally, leaching of components from preservatives treated lumber is small and primarily occurs immediately after the initial exposure of the treated member as unfixed components are removed from the wood surface (Evans, 1987). Even fully-fixed CCA will leach to some degree, depending on the exposure conditions (Ruddick, 1993), but this process is slow. Organic solvent preservative relatively insoluble in water and their permanence depends on their degree of volatility (Kaldas & Cooper, 1996; Cooper & Stokes, 1993).

Moreover the type of grain exposed can also influence leaching characteristics. Although wood properties may affect leaching in different ways, comparative studies generally agree that CCA components are more leachable from hardwoods than softwoods (Cooper, 1990; Yamamoto & Rokova, 1991). This phenomenon has been attributed to the lower content and type of lignin in hardwoods (Butcher & Nilsson, 1982; Cooper, 1990; Gray, 1993). However, this trend is not always consistent and softwood species also differ in leach susceptibility. Slow fixation and high leaching rates have been noted for Japanese cedar (Yamamoto & Rokova, 1991), and Cooper (1990) reported that CCA leaching rates from small, red pine specimens were approximately double those from lodge pole pine, Douglas Fir, and red cedar.

Conclusions

This study showed that the newly synthesized organotin(IV) compounds successfully retained in *Alstonia scholaris, Macaranga triloba and Hevea brasiliensis*. *Alstonia scholaris* recorded the highest preservative retention followed by *M. triloba* and *H. brasiliensis*. Leaching test showed that the least tin was detected from MPT leachate followed by DBT and DMT. The lowest tin concentration in leachates was determined from *H. brasiliensis* followed by *M. triloba* and *A. scholaris*, and no tin was observed after 270 hours in leachates. However, this study was performed on cubes in laboratory test; hence field test should be conducted to more authenticate these results.

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