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Treatability of Tropical Wood Using Newly Synthesized Organotin(IV) Complexes

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Abstract: The treatability of three tropical wood species namely *Alstonia scholaris* (pulai), *Macaranga triloba* (mahang) and *Hevea brasiliensis* (rubberwood) was investigated. Wood species were chemically treated with five newly synthesized organotin(IV) complexes using full-cell treatment method. This study explored whether tropical wood species could be treated successfully with newly synthesized organotin(IV) complexes as wood preservatives. Ten 19×19×19 mm sized wood cubes of each species were treated with three levels of concentration (0.1, 0.5 and 1%) of monomethyltin(IV) (MMT) and monophenyltin(IV) (MPT) of monoseries and dimethyltin(IV) (DMT), diphenyltin(IV) (DPT) and dibutyltin (IV) (DBT) of diseries organotin(IV) complexes with 2-acetylpyridine-*N* (4)-cyclohexyl thiosemicarbazone ligand. The treated wood species were evaluated by chemical retention values and characterized by FT-IR (Fourier Transform Infrared) spectroscopy analysis. The highest retention (10.59 kg m^{-3}) was found in *Alstonia scholaris* treated with 1% DMT complexes and the lowest retention (0.47 kg m^{-3}) was found in *Hevea brasiliensis* treated with 0.1% DMT complexes. FT-IR spectra of treated wood showed new absorption bands in the range of 594-606 and 441-457 cm^{-1} due to Sn-C and Sn-N bonds, respectively. A newly formed absorption band at range 549-569 cm^{-1} due to Sn-O bond was also observed in the treated wood sample spectra. Chemical retention and FTIR spectra suggest tropical wood species are treatable with newly synthesized organotin(IV) complexes as wood preservatives.

Key words: Treatability, tropical wood, retention, FTIR, organotin(IV)

INTRODUCTION

Wood is one of the most attractive materials because of its complex structure and wide range of application in the world. Some wood species are naturally more durable which is preferred building and construction materials due to its physical, mechanical and aesthetically pleasing performance. Most of tropical wood species are non durable or less durable which limits its indoor and outdoor applications. Huge non durable tropical wood species are abundantly available in Southeast Asia (Chao and Lee, 2003; Brelid *et al*., 2000; Yalinkilic *et al*., 1999; Deka *et al*., 2002). 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. Quality forest resources are becoming scarce that can't meet the need of people resulted an imbalance between demand and supply of forest product (Tolunay *et al*., 2008). This has driven researchers to look for alternative low-quality resources for value-added applications. One way 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). Majority of commercial timbers worldwide need to be treated before they can be utilized for various purposes (Sotannde *et al*., 2011). Izreen *et al*. (2011) showed that low quality hardwood timbers can be converted to value added wood products through chemical impregnation which make them resistant to fungal decay.

Most conventional preservatives cause environmental pollution and a few of them are hazardous to animals and human beings (Onuorah, 2000). The toxicity of the conventional wood preservative Chromate Copper Arsenate (CCA) is higher prior to impregnation. Thus, CCA presents a high risk for workers exposed to liquid solutions (Eaton and Hale, 1993). 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). For instance, copper in water has detrimental effect on various fish tissues (Balambigai and Aruna, 2011). Suruchi and Khanna (2011) stressed that metals including cadmium, copper, lead, chromium and mercury may be adsorbed into vegetables tissues that pose danger to human health if regular monitoring is not done. Lead is toxic to fish like *Labeo rohita* even at low concentration thus pose a

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potential risk for human consuming the fish (Ghosh *et al*., 2010).

TBTO (tri-n-butyltin oxide) and TBTN (tri-n-butyltin naphthenate) are both trialkyltin compounds and are used as fungicides worldwide (Schweinfurth *et al*., 1991). Both of these compounds are liquid at room temperature. These compounds are most effective against wood decay fungi. Schweinfurth *et al*. (1991) observed that the undiluted active ingredient of TBTO was found to be severely irritating to the skin of rabbit and human. The application of the compounds onto the skin of human showed severe reddening and slight swelling. It was demonstrated that TBTO is toxic to fresh water fish tilapia, *Oreochromis niloticus* (Alkhail *et al*., 2004). This shows the danger in using these trialkyltin compounds as fungicides. The precautions and safety of usage cannot be guaranteed and strict supervision is needed if both these compounds were to be used. Although, it is very effective organotin to treat wood but there have several disadvantages. Therefore, TBTO is recommended only for above ground 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.

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 and Hale, 1993). For this reason, it is necessary to search for new wood 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 (Wong, 2006). 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. Dibutyltin(IV) and triphenyltin(IV) exhibit toxic effects against cancerous and normal liver cells (Awang *et al*., 2011). Dibutyltin(IV) di-*N*-maleoylglycinate has been shown to possess high toxicity against pathogenic bacteria *Staphylococcus aureus* and *S. pyrogenes* (Ashfaq *et al*., 2004). Thus, the main objective of this study was to determine the treating ability of newly synthesized two monoorganotin(IV) and three diorganotin(IV) compounds on *Alstonia scholaris* (pulai), *Macaranga triloba* (mahang) and *Hevea brasiliensis* (rubber) wood as new wood preservatives.

MATERIALS AND METHODS

Preparation of wood samples: Three nondurable wood species were chosen in this study. *Alstonia scholaris* and Macaranga *triloba* wood samples were collected from an old secondary forest in Sarawak, Malaysia. Trees of both species are can be found in abundant in secondary forests throughout Sarawak. *Hevea brasiliensis* was obtained from an old plantation adjacent to Universiti Malaysia Sarawak's arboretum. *Hevea brasiliensis* plantation is huge in Malaysia and the trees are felled after about 25 years of planting that is when its economic life has passed. The logs were quarter-sawed to 25×25×25 mm boards and kiln dried. Every effort was made to produce heartwood boards by removing the sapwood during cutting. Heartwood was selected because it is the most difficult to treat in most wood species. Thus, if heartwood of a species can be treated the sapwood is most likely treatable. The boards were further planed, ripped and cut into 19 mm cubes. The cubes were conditioned at 60°C and 70% relative humidity for four days until they reached a constant weight. The weights of sample cubes after conditioned (W_1) were recorded. The volume (V_1) of the wood cube was determined using water displacement method.

Preservatives-organotin(IV) complexes: Five newly synthesized organotin(IV) compounds (Affan *et al*., 2011) were used as wood preservatives. The compound methyltin [$MeSnCl_2$ (APCT)] (MMT) and phenyltin [$PhSnCl_2$ (APCT)] (MPT) of monoseries and dimethyltin [Me_2SnCl (APCT)] (DMT), dibutyltin [Bu_2SnCl (APCT)] (DBT) and diphenyltin [Ph_2SnCl (APCT)] (DPT) of diseries of organotin(IV) complexes were used in this study. 2-acetylpyridine-*N*(4)-cyclohexyl thiosemicarbazone (APCT) was used as ligand.

Three levels of concentration (0.1, 0.5 and 1%) of organotin(IV) complexes were prepared for treatment. The organotin(IV) complexes were dissolved in solution of 20% dimethyl sulphoxide (DMSO) and 80% distilled water. Twenty percent dimethyl sulphoxide (DMSO) and 80% distilled water were prepared without the organotin(IV) complexes as the treating solution for control treatments.

Treatment of wood cubes with organotin(IV) complexes: Ten replicates of wood cubes were used for each treatment. Treatments were carried out according to the AWPA standard E10-91 (American Wood-Preservers' Association Standard (AWPA) 1991) with slight modifications. All wood cubes were placed inside the beaker containing the treating solution and soaked for two h. The beakers containing the wood cubes were 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 h and a final

vacuum of 100 mm 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 with tissue paper and weighed (W_2).

Determination of preservative 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 according to AWPA (1991) standard:

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

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)

FTIR spectroscopy analysis: Fourier Transform Infrared (FTIR) analysis was performed on Perkin Elmer Spectrum GX Fourier-Transform spectrometer equipped with a micro sample holder. Potassium bromide (KBr) powder was used to establish the background. Wood samples were air-dried prior to mixing with KBr. Spectra of the samples were collected using diffuse Fourier transform infrared spectroscopic technique (DRIFT). Spectra were collected for a total of 64 scans on 370 to 4000 cm^{-1} wavenumber range with a resolution of 4 cm^{-1}. All spectra were displayed in transmittance and limited to 370-4000 cm^{-1} region.

Analysis of data: One-way analysis of variance was used to determine the differences between mean values of retention of different wood species using different concentrations of chemicals. Further analyses of mean comparisons were done using Tukey Multiple Comparison test.

RESULTS AND DISCUSSION

Preservative retention: The preservative retention of *Alstonia scholaris*, *Macaranga triloba* and *Hevea brasiliensis* wood using monoseries of organotin(IV) (like MMT and MPT) and diseries of organotin(IV) (like DMT, DPT and DBT) compound of each three levels of concentration (0.1, 0.5 and 1%) were statistically analyzed to determine the treatability of wood species shown in Table 1.

Table 1: Average retention values (kg m^{-3}) in *Alstonia scholaris*, *Macaranga triloba* and *Hevea brasiliensis* heartwood following treatment with monoseries and diseries of organotin(IV) at three levels of concentration

Treating chemicals	*Hevea brasiliensis*	*Macaranga triloba*	*Alstonia scholaris*
Monoseries organotin			
MMT (%)			
0.1	0.58* (0.04)^a	0.77 (0.02)^b	0.84 (0.02)^c
0.5	3.27 (0.17)^a	3.62 (0.14)^b	4.59 (0.10)^c
1	5.62 (0.13)^a	7.31 (0.14)^b	7.48 (0.17)^c
MPT (%)			
0.1	0.57 (0.04)^a	0.77 (0.02)^b	0.89 (0.02)^c
0.5	3.33 (0.14)^a	3.59 (0.18)^b	4.39 (0.02)^c
1	5.76 (0.12)^a	7.70 (0.11)^b	7.90 (0.14)^c
Diseries organotin			
DMT (%)			
0.1	0.47 (0.06)$_a$	1.00 (0.02)^b	1.00 (0.03)^b
0.5	2.80 (0.63)^a	4.62 (0.15)^b	5.65 (0.21)^c
1	5.65 (0.14)^a	9.58 (0.17)^b	10.59 (0.15)^c
DPT (%)			
0.1	0.52 (0.08)^a	0.97 (0.02)^b	0.96 (0.03)^b
0.5	2.58 (0.11)^a	4.52 (0.15)^b	4.62 (0.15)^b
1	5.34 (0.11)^a	7.61 (0.12)^b	7.94 (0.13)^c
DBT (%)			
0.1	0.62 (0.03)^a	0.83 (0.02)^b	0.81 (0.02)^b
0.5	3.35 (0.13)^a	4.37 (0.12)^b	4.91 (0.15)^c
1	6.05 (0.23)^a	8.06 (0.16)^b	9.49 (0.20)^c

Values in parenthesis is the standard deviation, MMT- Monomethyltin(IV) complex, MPT: Monophenyltin(IV) complex, DMT: Dimethyltin(IV) complex, DPT: Diphenyltin(IV) complex, DBT: Dibutyltin(IV) complex
*Means followed by a different letter within a row are statistically different at $p<0.05$ using Tukey multiple comparison test

The mean preservative retentions varied significantly between wood species (Table 1). *Alstonia scholaris* gained higher chemical retention uptake than *Macaranga triloba* and *Hevea brasiliensis* wood for all level of preservatives concentration. Differences in preservatives uptake indicated that treatability differ between wood species. The highest (10.59 kg m^{-3}) and lowest (0.47 kg m^{-3}) retention uptake was observed in *Alstonia scholaris* and *Hevea brasiliensis* wood, respectively.

Results also showed that increased in concentration from 0.1 to 1% resulted in increased retention in all wood species. The mean preservatives retention following increased in treating concentration varied from 0.81 to 10.59 kg m^{-3} in *Alstonia scholaris* for all treating chemicals. Greaves *et al.* (1982) observed that treating *Pinus radiata* using 0.1% TBTO resulted in retention of 1.2 kg m^{-3}.

The retention following treatment with 1% organotin(IV) versus wood species is illustrated in Fig. 1. In this study higher retention was observed with diseries preservatives (DMT and DBT) in *Alstonia scholaris* and *Macaranga triloba* than the monoseries preservatives. Among monoseries 1% MPT showed significantly higher retention than MMT in *Alstonia scholaris* and *Macaranga triloba*. However, in *Hevea brasiliensis*

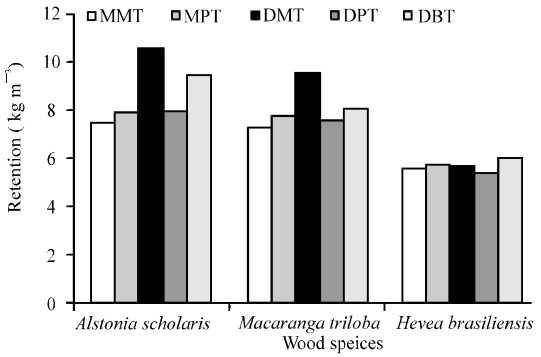

Fig. 1: Chemical retention of (1%) organotin(IV) treated wood species

the difference of retention between MPT and MMT was not significant.

Among diseries of 1% organotin(IV) DMT gave significantly higher retention than DPT and DBT in *Alstonia scholaris* and *Macaranga triloba*. But in *Hevea brasiliensis* DBT gave significantly higher retention than DMT and DPT.

Results presented in Table 1 and Fig. 1 suggest organotin(IV) can successfully treat *Alstonia scholaris*, *Macaranga triloba* and *Hevea brasiliensis*. Treatability results showed that preservative retention for *Alstonia scholaris* was almost two fold that of *Hevea brasiliensis*. Retention values suggest the treatability of wood is in the decreasing order of *Alstonia scholaris* and *Macaranga triloba* and *Hevea brasiliensis*. In Malaysia *Hevea brasiliensis* is a treatable timber (Dahlan *et al*., 1994; Hiziroglu, 1997). Retention in *Hevea brasiliensis* treated by 2% copper chrome arsenic type C (CCA-C) after two hours of pressure by full cell method was 13.0 kg m^{-3} (Jusoh and Kamdem, 2000). Retentions of 14 and 16 kg m^{-3} were observed in Elm and Oak, respectively following treatment with 5% CCA (Kazemi, 2007).

The Malaysian Standard SIRIM (1991) did not specify the use of organotin(IV) to treat wood. However, as a point of reference the specified retention of 8 and 12 kg m^{-3} of the CCA preservative is required in the treated timber for above ground and soil contact, respectively. The retentions obtained in this study suggest the newly synthesized organotin(IV) compounds successfully treat *Alstonia scholaris* and *Macaranga triloba* and *Hevea brasiliensis* for above ground exposure and ground contact.

Fourier Transform Infrared (FT-IR) spectroscopy analysis: *Alstonia scholaris*, *Macaranga triloba* and *Hevea brasiliensis* wood samples were analyzed by FT-IR spectroscopy to authenticate the treatability by determining whether the treating chemicals were incorporated within the wood cell. The main characteristics FT-IR absorption data of all treated wood samples are presented in Table 2.

The characterizations were performed on all species of wood samples, but similar results were obtained. Thus only the results corresponding to MMT and DPT, MPT and DBT, MMT and DMT treated and untreated *Alstonia scholaris*, *Macaranga triloba* and *Hevea brasiliensis* wood spectra are given as a representative case shown in Fig. 2 (a, b), 3 (a, b) and 4 (a, b), respectively. The IR spectra of the untreated wood clearly showed the absorption bands at 3406-3415, 2903-2917 and 1730-1742 cm^{-1} due to OH, CH and CO stretching vibrations, respectively. These absorption bands are due to the hydroxyl group in cellulose, carbonyl group in hemicellulose and carbonyl aldehyde in lignin (Ismail *et al*., 2002). Zhang and Kamdem (2005) also observed that hemicellulose and lignin are the bonding sites for copper. On the other hand the formation of new bonds like tin carbon (Sn-C), tin oxygen (Sn-O) and tin nitrogen (Sn-N) bond by the fixation of organotin(IV) compound within the wood cell was confirmed by the FT-IR spectroscopy analysis of treated wood. In the spectra of organotin(IV) treated *Alstonia scholaris*, *Macaranga triloba* and *Hevea brasiliensis* wood, a new absorption band at 594-606, 561-569 and 441-457 cm^{-1} are assigned to the stretching mode of ν(Sn-C), ν(Sn-O) and ν(Sn-N), respectively. A new ν(Sn-O) linkage indicating the tin(IV) coordinated with oxygen of OH after deprotination in wood cell which suggesting the fixation of organotin(IV) within the wood cell (Yin *et al*., 2007; Mendes *et al*., 2006). Moreover, IR spectra of treated wood clearly showed the presence of the characteristics of cyclohexyl, CH, C = N, C-S and N-N bond at 2929-2937, 1542-1559, 1242-1255 and 829-836 and 1011-1034 cm^{-1}, respectively (Rebolledo *et al*., 2005; Elvy *et al*., 1995; Haque *et al*., 2009; Covolan *et al*., 1997). The absorption band of OH group also shifted towards lower wave number (3415 to 3375-3395 cm^{-1}) with narrowed band intensity, which gives further evidence of the reaction of cellulose OH groups with organotin(IV) compound and formed Sn-O bond (Hortling *et al*., 1997; Tolvaj and Faix, 1995). Based on the IR spectra results, the organotin(IV) compound incorporated within the wood cell and tropical woods are treatable with synthesized organotin(IV) compounds.

Table 2: IR spectra data of organotin(IV) treated wood species

Wood species	Treating compound	Bonding: ν (NH) cm^{-1}	ν (cyclohexyl) cm^{-1}	ν (C = N-N = C) cm^{-1}	ν (N-N) cm^{-1}	ν (C-S) cm^{-1}	ν (Pyridine in plane) cm^{-1}	ν (Sn-C) cm^{-1}	ν (Sn-O) cm^{-1}	ν (Sn-N) cm^{-1}
Alstonia scholaris	MMT	3172	2930, 2854	1557	1028	1252, 831	649	603	568	441
	MPT	3178	2932, 2854	1556	1033	1255, 830	649	606	561	449
	DMT	3176	2936, 2851	1559	1034	1250, 831	622	596	567	449
	DPT	3176	2929, 2852	1542	1021	1251, 829	628	602	569	456
	DBT	3173	2936, 2851	1542	1018	1249, 836	634	596	565	448
Macaranga triloba	MMT	3179	2930, 2854	1557	1028	1252, 831	649	603	568	450
	MPT	3170	2932, 2853	1557	1020	1254, 830	650	605	567	448
	DMT	3177	2937, 2851	1542	1016	1250, 832	633	596	567	449
	DPT	3177	2930, 2852	1542	1021	1251, 829	636	596	569	451
	DBT	3175	2936, 2851	1542	1016	1249, 836	634	598	566	452
Hevea brasiliensis	MMT	3173	2930, 2854	1557	1027	1242, 831	649	603	568	451
	MPT	3179	2932, 2854	1556	1017	1254, 830	631	606	569	454
	DMT	3178	2936, 2851	1542	1013	1249, 832	622	596	567	450
	DPT	3171	2929, 2851	1542	1021	1250, 829	633	594	569	457
	DBT	3175	2936, 2851	1542	1011	1249, 835	621	595	561	453

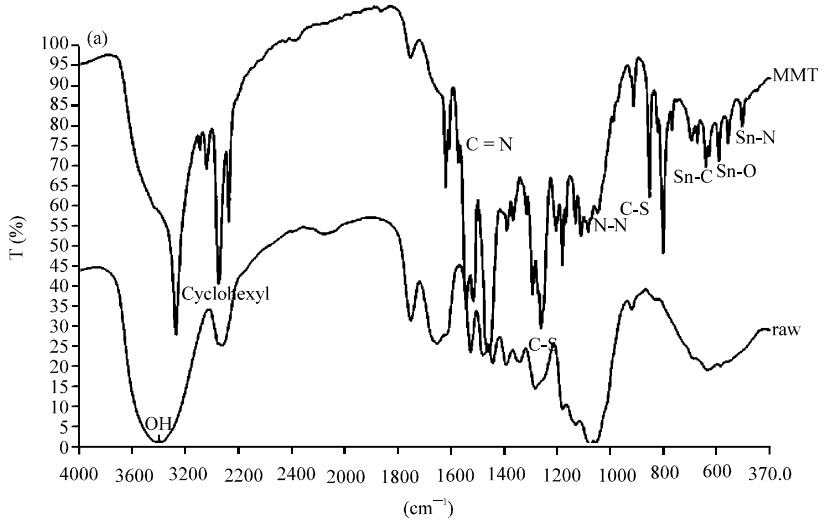

Fig. 2(a): IR spectra of untreated and treated *Alstonia scholaris* wood with monomethyltin(IV) complex

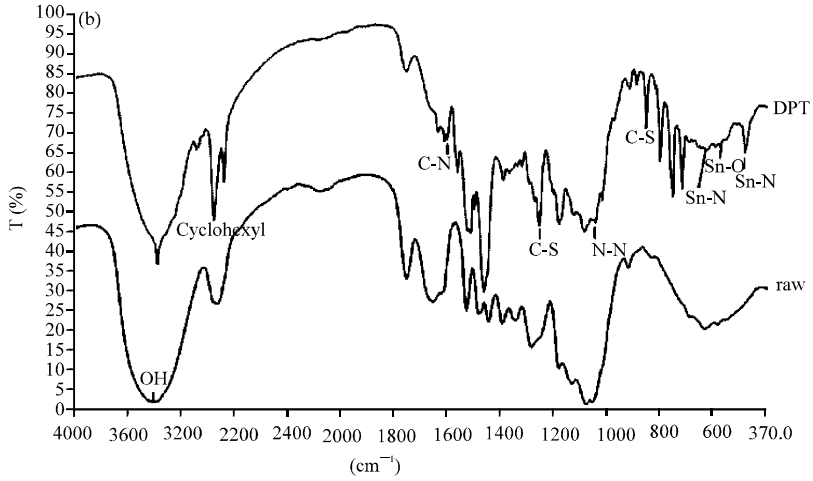

Fig. 2(b): IR spectra of untreated and treated *Alstonia scholaris* wood with diphenyltin(IV) compound

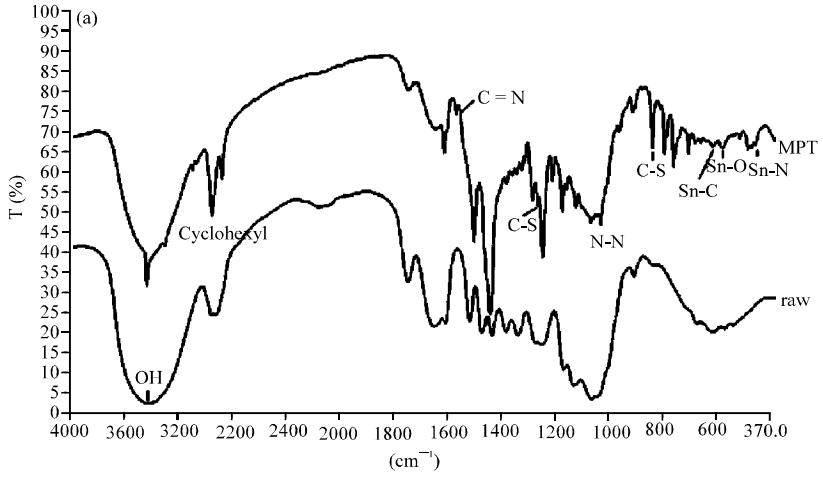

Fig. 3(a): IR spectra of untreated and treated *Macaranga triloba* wood with monophenyltin(IV) complex

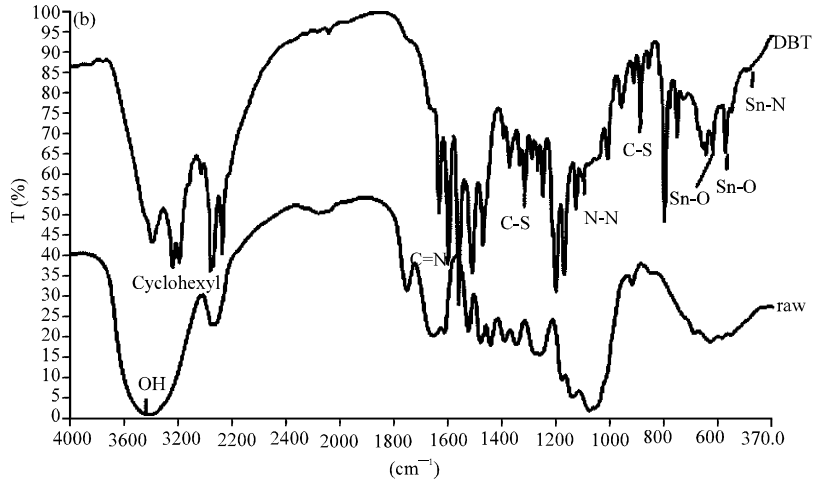

Fig. 3(b): IR spectra of untreated and treated *Macaranga triloba* wood with dibutyltin(IV) complex

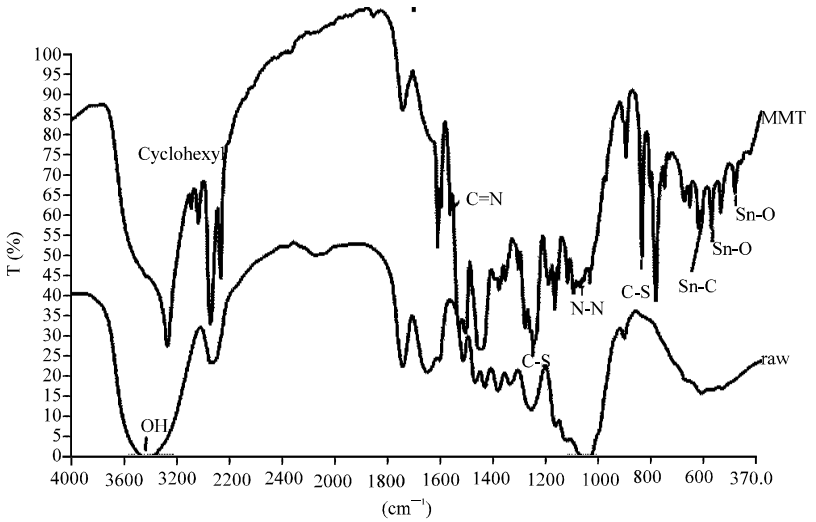

Fig. 4(a): IR spectra of untreated and treated *Hevea brasiliensis* wood with monomethyltin(IV) complex

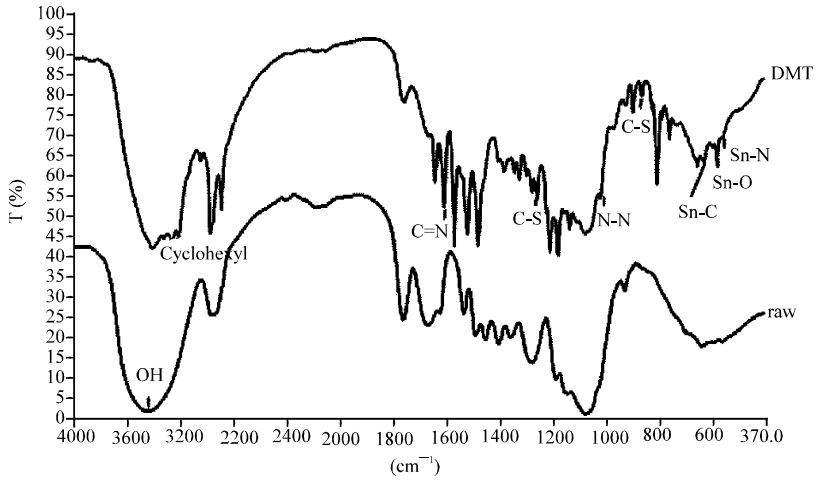

Fig. 4(b): IR spectra of untreated and treated *Hevea brasiliensis* wood with dimethyltin(IV) complex

CONCLUSION

This study showed that the newly synthesized organotin(IV) compounds successfully treated *Alstonia scholaris*, *Macaranga triloba* and *Hevea brasiliensis*. *Alstonia scholaris* recorded the highest preservative retention followed by *Macaranga triloba* and *Hevea brasiliensis*. Retention increased with the concentration increased in all wood species. A newly formed Sn-O bond was observed in all treated wood sample. FT-IR spectra showed tin compounds bind with wood cell suggest the treatability of tropical wood species with synthesized organotin(IV) compounds. Hemicellulose and lignin play the important role in bonding tin. Decay and field tests are underway in order to determine the efficacy of organotin(IV) as wood preservatives.

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