

## FIXATION AND LEACHING CHARACTERISTICS OF CCA-TREATED MALAYSIAN HARDWOOD

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**SABIHA S, SYAIDATUL S, ZAIDON A, MOHD HAMAMI S, CHOI YS & KIM GH. 2015. Fixation and leaching characteristics of CCA-treated Malaysian hardwood.** Effects of chromated copper arsenate (CCA) on fixation and leaching characteristics of eight Malaysian commercial hardwood, namely, acacia (*Acacia mangium*), bintangor (*Calophyllum* sp.), geronggang (*Cratoxylum arborescens*), medang (*Litsea* sp.), meranti rambai daun (*Shorea acuminata*), putat (*Planchonia* sp.), ramin (*Gonystylus* sp.) and simpuh (*Dillenia* sp.) were studied. CCA-fixation rate in wood was evaluated by monitoring the reduction of hexavalent to trivalent chromium in expressates. The amounts of copper, chromium and arsenic remaining in the unfixed state after 14 days were also determined. Acacia was the most suitable species for treatment with CCA. In comparison with radiata pine, leaching of CCA components from treated timbers was high and varied between species owing to their chemical composition especially lignin content. An additional study was conducted with geronggang and ramin to determine the effect of pre-extraction on fixation. Results indicated that differences in fixation and leaching characteristics of CCA-treated timbers (especially geronggang and ramin) were influenced by the extractive content of the wood.

Keywords: Commercial hardwood, reduction of hexavalent to trivalent chromium, pre-extraction

### INTRODUCTION

Malaysian tropical hardwood is of considerable interest to wood manufacturers because the timbers can be marketed for a wide range of industrial and domestic applications. Major importing countries such as Europe, Middle East, United States, Japan and Australia are also using Malaysian tropical hardwood for a wide variety of purposes (Cheung et al. 2007). In order for these timber resources to be used sustainably, they must be protected using efficient wood preservatives such as chromated copper arsenate (CCA). However, caution must be exercised when using CCA to avoid polluting the environment.

Currently, CCA is still being used for commercial and industrial products such as construction and other aboveground building components in several countries. This is because of its good performance against wood-decaying

organisms. However, CCA is water soluble and can be leached by rainwater and weathering. Leaching of toxic CCA components from treated wood has raised public concerns and decreased the service life of treated wood (Hingston et al. 2001, Lebow et al. 2008). Hazardous heavy metal components as well as contamination of soil and groundwater represent serious threats to humans and the ecosystem. Therefore, fixation of CCA-treated wood immediately after treatment is crucial. Although fixation and leaching characteristics of CCA-treated wood in temperate regions have been extensively investigated and the efficacy has been reported (Stevanovic-Janezic et al. 2000, Kartal & Lebow 2001, Guo et al. 2002, Radivojevic & Cooper 2007, Kim et al. 2008), information on fixation and leaching characteristics of CCA-treated tropical hardwood is lacking. A few species including

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sentang, rubberwood and kempas have been studied (Sulaiman et al. 2002, Wong et al. 2006). However, these studies focused on evaluating the efficacy of CCA in terms of durability and ability of CCA-treated wood to withstand biological decay, rather than quantifying their effects on leaching.

Hardwood is very complex and heterogeneous in terms of chemical composition and anatomical structure. Therefore, in comparison with softwood, hardwood shows different effects after treatment with CCA, especially in terms of fixation and leaching characteristics. Softwood is more readily fixed with this waterborne preservative because of higher lignin content. The diverse chemical wood constituents, celluloses, hemicelluloses and lignin react differently with components of CCA preservative. The wood extractive constituents of red maple and red oak (Stevanovic-Janezic et al. 2000) as well as Douglas-fir (Kim et al. 2008) were previously shown to influence the fixation of CCA. The amount, type and location of wood extractives may have important influence on variations in fixation and leaching between hardwood species. CCA is the most popular wood preservative in Malaysia. Therefore, identification of the fixation characteristics of tropical hardwood is crucial to produce environmentally-friendly products with increased service life at reduced cost.

In the present study, we evaluated the fixation and leaching characteristics of eight CCA-treated Malaysian tropical hardwood, namely acacia, bintangor, geronggang, medang, meranti rambai daun, ramin, putat and simpuh. We conducted an additional study to determine the effect of pre-extraction on CCA fixation. The effects of pre-extraction on fixation and leaching characteristics of eastern USA (Cooper et al. 1997), Canadian (Stevanovic-Janezic et al. 2000) and Appalachian USA (Dawson-Andoh et al. 2002) hardwood have been reported. To our knowledge, we are the first to evaluate the effect of pre-extraction on Malaysian tropical hardwood.

## MATERIALS AND METHODS

### Sample preparation and preservative treatment

Heartwood blocks (20 mm<sup>3</sup>) of eight Malaysian tropical hardwood species: acacia (*Acacia*

*mangium*), bintangor (*Calophyllum* sp.), geronggang (*Cratoxylum arborescens*), medang (*Litsea* sp.), meranti rambai daun (*Shorea acuminata*), putat (*Planchonia* sp.), ramin (*Gonystylus* sp.) and simpuh (*Dillenia* sp.) were cut from air-dried boards obtained from the Forest Research Institute of Malaysia and Kompleks Perakayan Terengganu, Malaysia. The blocks were conditioned at room temperature for a minimum of 2 weeks. The softwood radiata pine (*Pinus radiata*) was included for comparison. Air-dried heartwood blocks of the nine species were vacuum-pressure impregnated with CCA Type C (CrO<sub>3</sub> 47.5%; CuO 18.5%; As<sub>2</sub>O<sub>5</sub> 34.0%) solution to achieve target retention of 5.6 kg m<sup>-3</sup>, the minimum CCA retention for interior timbers such as roof trusses that are not in contact with the ground (MS 1991). The treatment schedule consisted of 30-min initial vacuum of 760 mm Hg, after which the cylinder was filled with CCA solution and subjected to pressure of 14 kg cm<sup>-2</sup> for 1 hour. The strength of CCA solution required to obtain the target CCA retention was calculated from results of initial water uptake trials on additional blocks using the treatment schedule described above. Actual CCA retentions were determined from the solution uptake and solution concentration. Treated blocks were immediately wrapped in aluminum foil and conditioned at 27 °C and 65% relative humidity (representing the annual average temperature and relative humidity of Peninsular Malaysia) to allow fixation without drying for different periods of time (0, 1, 3, 7, 10 and 14 days). Treated blocks conditioned without drying and prepared by drying without wrapping with aluminum foil at similar conditions and relative humidity were also included for comparison.

### Pre-extraction treatment

A number of geronggang and ramin samples were pre-extracted prior to CCA treatment by boiling submerged wood blocks in water for 8 hours per day for 2 days, with four water exchanges per day. The final extraction was conducted using water containing 5% aqueous ethanol to eliminate any phenolic compound present. Geronggang and ramin were selected for pre-extraction treatment because both species showed high unfixed amounts of CCA components (especially chromium) in the wood, even after 14 days.

## Determination of fixation

The fixation test was conducted according to the expressate method described by McNamara (1989). At appropriate fixation times, two blocks were randomly selected and squeezed in a hydraulic press to release unfixed treatment solution from void spaces of wood. The expressed solution was analysed for hexavalent chromium content using diphenylcarbazide colorimetric method (ASTM 2007), and for total chromium, copper and arsenic contents using inductively coupled plasma–optical emission spectroscopy (ICP–OES).

## Determination of leaching

Leaching test was performed according to the American Wood Preservers' Association standard E11-06 leaching procedure with some modifications (AWPA 2006a). Two 20-mm cubes were impregnated and leached with 110 mL of deionised water (volume was calculated using water-to-wood ratio specified by the AWPA E11-06 standard method). After 14 days of leaching, the accumulated leachate samples from nine sampling times (6, 24, 48, 96, 144, 192, 240, 288 and 336 hours) were analysed for total chromium, copper and arsenic contents using ICP–OES (AWPA 2006b). The total amount of leaching was expressed as percentage loss of each CCA component relative to the amount initially retained in treated samples. The amount of CCA retained in leached samples was determined by digesting treated samples according to the American Wood Preservers' Association standard A7-04 wet ashing procedure for preparing wood for chemical analysis (AWPA 2006c). The liquid samples obtained were analysed for total chromium, copper and arsenic contents using ICP–OES.

## RESULTS AND DISCUSSION

### Reduction of hexavalent chromium

In comparison with radiata pine, all eight hardwood species showed rapid reduction of hexavalent chromium to trivalent chromium (Figure 1). The chromium reduction rate varied markedly between the eight species. In acacia and geronggang, hexavalent chromium was

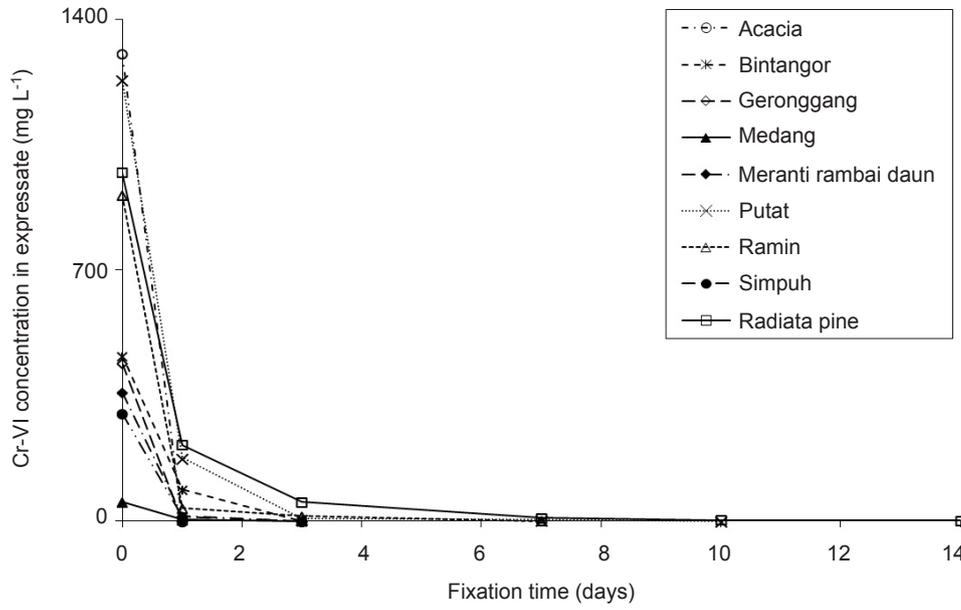
reduced approximately 1 day after treatment. In bintangor, medang, meranti rambai daun and simpuh, hexavalent chromium was reduced approximately 3 days after treatment. In ramin, hexavalent chromium was reduced approximately 7 days after treatment and in putat, 10 days after treatment. In radiata pine, hexavalent chromium was reduced only 14 days after treatment. The rapid reduction rates in tropical timbers may be caused by the reaction of hexavalent chromium with wood extractives (Kim et al. 2008).

### Fixation of CCA components

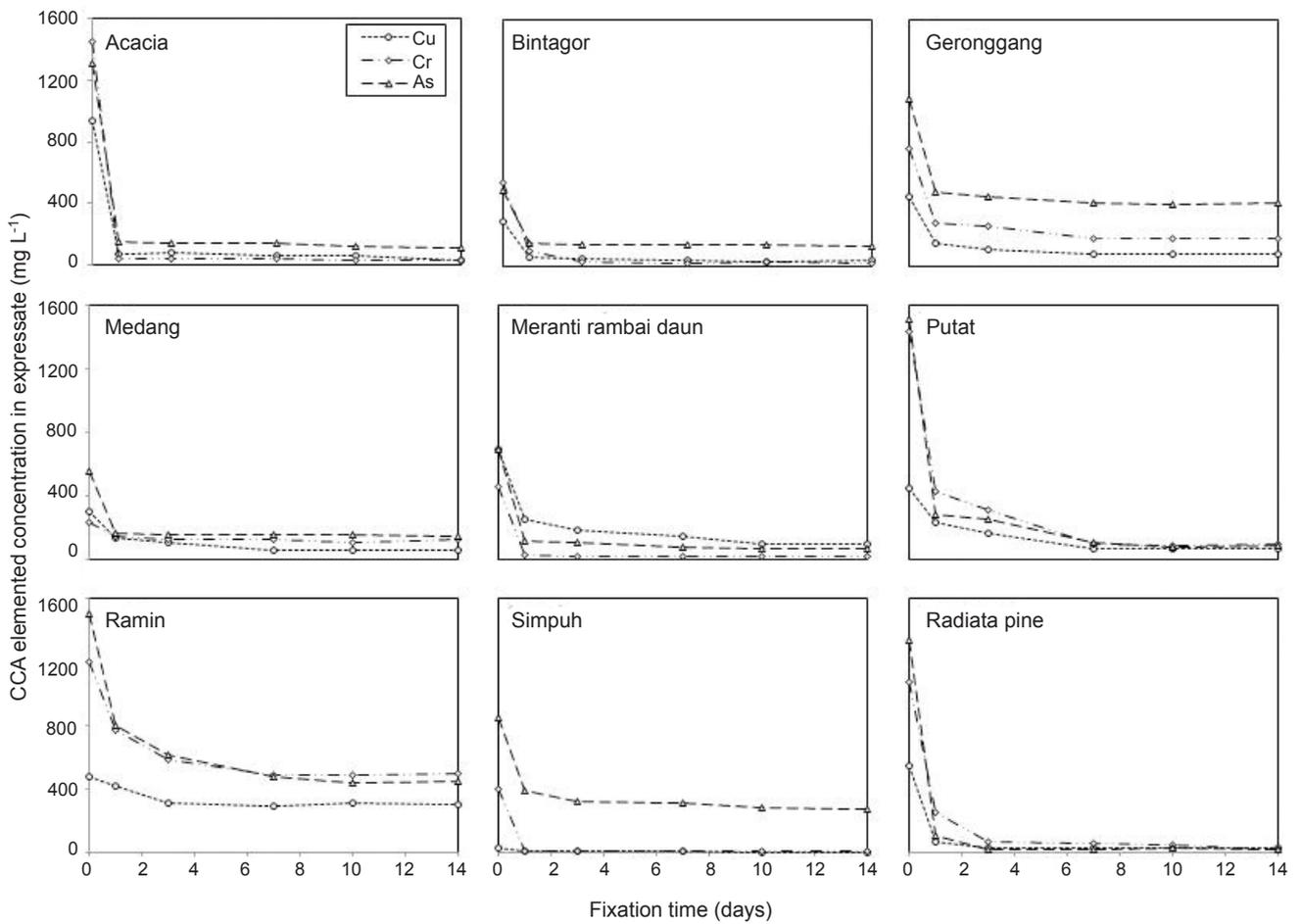
Despite the rapid reduction of hexavalent chromium (Figure 1), we found incomplete fixation of total chromium, copper and arsenic in all eight timber species, regardless of fixation time (Figure 2). The CCA components in the treated wood remained in an unfixed state, even after 14 days. In comparison with radiata pine, the amount of arsenic remaining unfixed in all eight timber species was considerably higher. The highest amount of unfixed arsenic ( $451 \text{ mg L}^{-1}$ ) was found in ramin, followed by geronggang, simpuh, medang, bintangor, acacia, putat and meranti rambai daun (Table 1). The highest amount of unfixed copper ( $306 \text{ mg L}^{-1}$ ) was found in ramin, followed by meranti rambai daun, geronggang, putat and medang. The highest amount of unfixed chromium ( $497 \text{ mg L}^{-1}$ ) was found in ramin, followed by geronggang, medang and putat. These results contradict the concept of CCA fixation, which indicates that copper and arsenic are stabilised in treated wood before all the hexavalent chromium is reduced to trivalent chromium. Therefore, monitoring of the reduction of hexavalent chromium to trivalent chromium may not be appropriate for confirming CCA fixation, at least for the eight timber species evaluated. Nonetheless, this might reflect the end of stabilisation reactions of CCA components because the amounts of unfixed arsenic and copper did not further stabilise with time.

### Leaching of CCA components

Incomplete fixation occurred in almost all timber species tested, probably because of the presence of extractives that could interfere with the fixation process (Dahlgren 1975, Kennedy



**Figure 1** Hexavalent chromium (Cr-VI) reduction in the expressate from treated Malaysian tropical hardwood and radiata pine, as a function of fixation time



**Figure 2** Fixation trends of chromated copper arsenate (CCA) components in treated Malaysian tropical hardwood, as a function of fixation time

**Table 1** Concentrations of unfixed chromated copper arsenate (CCA) components in expressates from treated timbers and percentage fixation after 14-day fixation

Timber	Amount of unfixed CCA components (mg L <sup>-1</sup> )		
	Cu	Cr	As
Acacia	33 (98.5)	33 (99.0)	112 (96.0)
Bintangor	34 (98.0)	18 (99.3)	130 (93.8)
Geronggang	79 (95.2)	176 (92.8)	410 (80.2)
Medang	59 (96.8)	124 (95.4)	150 (93.4)
Meranti rambai daun	148 (90.9)	24 (99.0)	72 (96.5)
Putat	65 (96.2)	98 (96.1)	91 (95.9)
Ramin	306 (77.7)	497 (75.5)	451 (73.2)
Simpuh	5 (99.6)	14 (99.2)	277 (82.7)
Radiata pine	32 (97.3)	23 (98.9)	25 (98.5)

Percentage fixation rate in parenthesis

**Table 2** Concentrations of chromated copper arsenate (CCA) components in leachates from treated timbers and percentage of total leached after 14-day leaching

Timber	Amount of leached CCA components					
	Cu		Cr		As	
	(mg L <sup>-1</sup> ) <sup>a</sup>	(%) <sup>b</sup>	(mg L <sup>-1</sup> )	(%)	(mg L <sup>-1</sup> )	(%)
Acacia	0.16 ± 0.04 ef	2.0	0.17 ± 0.01 c	0.5	2.15 ± 0.39 f	10.9
Bintangor	0.08 ± 0.01 ef	0.8	0.11 ± 0.01 c	0.4	3.88 ± 0.17 e	22.9
Geronggang	0.40 ± 0.07 e	4.3	1.11 ± 0.02 b	5.4	7.18 ± 0.14 c	46.5
Medang	0.30 ± 0.07 ef	3.1	0.73 ± 0.13 bc	3.0	9.45 ± 1.28 a	51.3
Meranti rambai daun	1.69 ± 0.15 b	15.5	0.34 ± 0.02 bc	0.9	6.30 ± 0.28 cd	29.5
Putat	0.83 ± 0.22 c	8.8	1.06 ± 0.31 b	4.7	2.24 ± 0.37 f	16.1
Ramin	3.37 ± 0.60 a	40.0	5.47 ± 1.75 a	27.1	6.15 ± 0.13 d	45.4
Simpuh	0.05 ± 0.00 f	0.4	0.17 ± 0.01 c	0.7	8.32 ± 0.62 b	47.6
Radiata pine	0.56 ± 0.09 c	4.9	0.14 ± 0.02 c	0.5	1.47 ± 0.20 f	9.0

<sup>a</sup>Cumulative concentration of CCA components in leachate; <sup>b</sup>amount of each preservative component leached is represented as percentage of the initial concentration (total retention) of the component in treated wood; values in each column followed by the same letter/s do not differ significantly ( $\alpha = 0.05$ ) according to Duncan's multiple range test

& Palmer 1994, Cooper et al. 1997, Stevanovic-Janezic et al. 2000). Except for acacia and putat, arsenic losses were significantly higher in all timbers tested than in softwood radiata pine (Table 2). High amounts of copper and chromium were lost from geronggang, medang, meranti rambai daun and ramin. Using radiata pine as reference, the eight species tested were categorised for leaching performance (Table 2)—high leaching: geronggang, medang, meranti rambai daun, ramin and simpuh; moderate

leaching: bintangor and putat and low leaching: acacia. The high amounts of CCA components leached from treated timbers can be explained by the deposition of products derived from CCA fixation in the cell lumen, where they are more readily accessible to leaching because of higher extractive levels (Cooper et al. 1997, Srivinasan et al. 1999, Stevanovic-Janezic et al. 2000, Kim et al. 2008). Variations in leaching losses between the eight timber species may be derived from differences in chemical composition of the wood,

e.g. extractive content and ratio of major cell wall components including cellulose, hemicellulose and lignin (Yamamoto & Rokoba 1991, Englund & Gardner 1993, Cooper et al. 1997, Srivinasan et al. 1999).

The highest amounts of unfixed arsenic were observed in geronggang and ramin (Table 1), possibly because of complex reactions between wood extractives and cations contained in the CCA preservative (Fengel & Wegener 1984, Stevanovic-Janezic et al. 2000). Therefore, we subsequently determined effects of pre-extraction on the fixation and leaching characteristics of geronggang and ramin.

### **Correlations between unfixed and leached amounts of copper, chromium and arsenic**

For the unextracted wood, positive correlations between unfixed and leached amounts of copper ( $r^2 = 0.968$ ) and chromium ( $r^2 = 0.965$ ) but no correlation between unfixed and leached amounts of arsenic were observed (results not shown). We performed an additional study to elucidate the effect of extractives on the lack of correlation between unfixed and leached amounts of arsenic. The results of our pre-extraction study showed positive correlations between unfixed and leached amounts of arsenic and chromium ( $r^2 = 0.998$ ) and also copper ( $r^2 = 0.977$ ). The presence of positive correlation between unfixed and leached amounts of arsenic indicated that the extractive content of geronggang and ramin contributed significantly to high unfixed and leached amounts of CCA components.

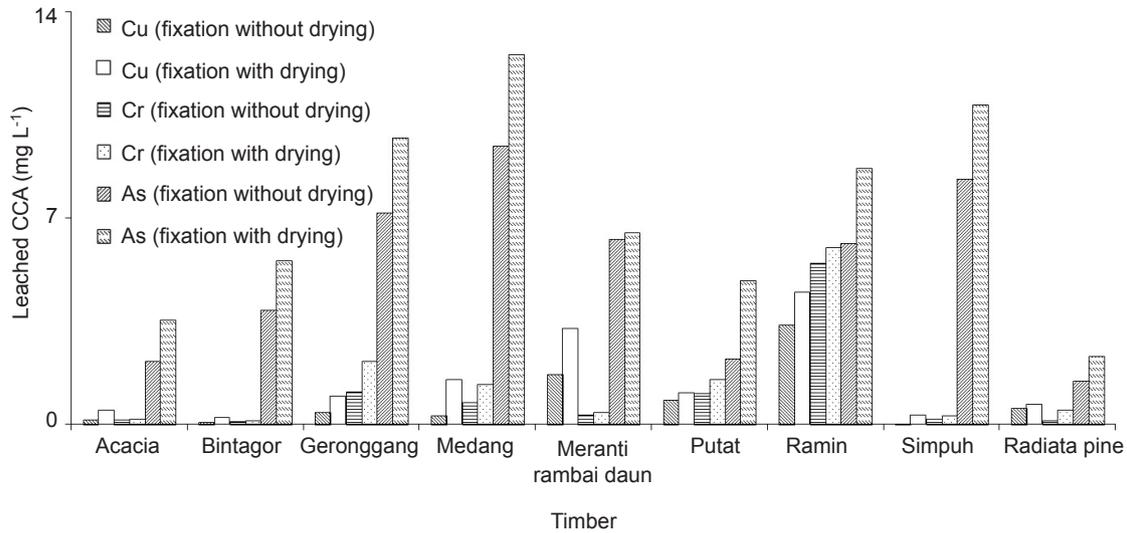
### **Effects of fixation with and without drying on leaching characteristics of treated wood**

After 14 days, amounts of CCA components leached from timbers that had undergone fixation without drying were lower than those from timbers that had undergone fixation with drying (Figure 3). These results were in accordance with those of previous studies, which reported a decrease in leaching of CCA when treated wood was subjected to higher moisture content or non-drying conditions during fixation (Lee et al. 1993, Kaldas & Cooper 1996, Ung & Cooper 1996).

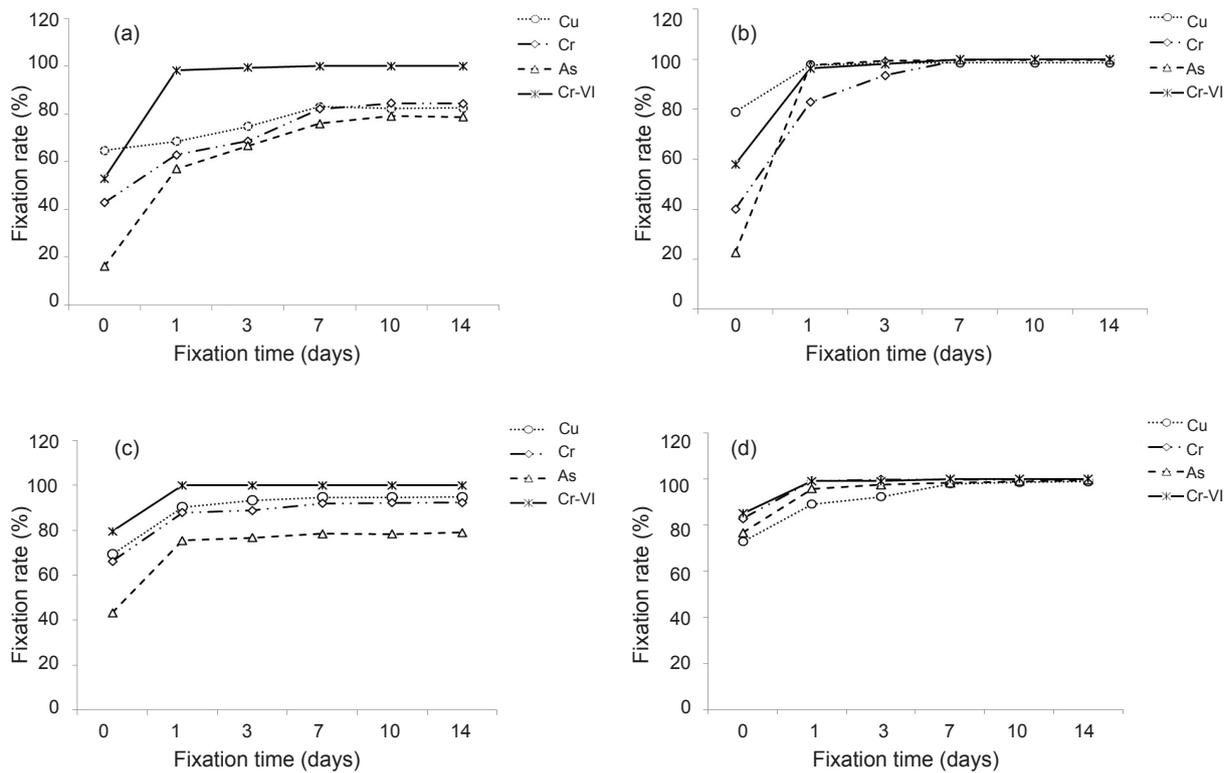
### **Effects of pre-extraction on fixation and leaching characteristics of geronggang and ramin**

In geronggang and ramin, we observed enhanced fixation rates of CCA components after pre-extraction (Figures 4a–d). Pre-extracted geronggang and ramin showed high fixation of copper and arsenic after reduction of hexavalent chromium to trivalent chromium (Figures 4b and d). The fixation time of geronggang was longer after pre-extraction than before pre-extraction (7 days versus 1 day); however, the fixation rate, particularly for arsenic, was higher (Table 3). On the contrary, the fixation time of ramin was 7 days, regardless of pre-extraction. This discrepancy may be caused by differences in extractive types and amounts in the two species (Carpenter & Gardner 1993, Englund & Gardner 1993, Cooper et al. 1997). In both species, pre-extraction led to decrease in the unfixed amounts of copper, chromium and arsenic in the expressate after 14 days. The fixation rates for chromium and arsenic were 99.7 and 99.4% respectively for geronggang, and 99.8 and 99.7% respectively for ramin. Prior to pre-extraction, the fixation rate of hexavalent chromium was rapid. However, arsenic and copper competed with tannins and soluble sugars for fixation. After pre-extraction with 5% aqueous ethanol solution, tannins in particular were removed from the wood blocks prior to CCA treatment. Thus, fixation of hexavalent chromium became slower. Moreover, extractives that had been removed via pre-extraction could have interfered with the reduction of hexavalent chromium to Cr (III) and complex Cu (II) (Radivojevic & Cooper 2007).

Our results indicated that wood extractives and other main chemical components of wood in ramin and geronggang interacted with CCA, thereby interfering with the fixation process. Our findings were in accordance with those reported for Appalachian hardwood by Dawson-Andoh et al. (2002). They reported that arsenic was frequently the most unfixed element in Appalachian hardwood because of insufficient availability of chromium for complexation. In contrast, in the present study, we observed a higher amount of fixed arsenic in pre-extracted wood, probably because sufficient chromium



**Figure 3** Effects of fixation conditions on chromated copper arsenate (CCA) leaching performance of different timbers



**Figure 4** Fixation rates of total chromium, copper and arsenic after reduction of hexavalent chromium (Cr-VI): (a) unextracted geronggang, (b) pre-extracted geronggang, (c) unextracted ramin and (d) pre-extracted ramin

was available for complexation during the fixation process, especially in ramin (Figures 4c and d). This is justified by the lower losses of

arsenic in geronggang and ramin after pre-extraction (Table 4). Further, copper becomes stabilised after arsenic because copper can be

**Table 3** Concentrations of unfixed chromated copper arsenate (CCA) components in expressate from treated geronggang and ramin and their % fixation after 14-day fixation

Timber	Pre-extract	Fixation time (days)	Unfixed amount of CCA components (mg L <sup>-1</sup> )		
			Cu	Cr	As
Geronggang	No	1	79 (95.2)	176 (92.8)	410 (80.2)
	Yes	7	20 (98.8)	7 (99.7)	13 (99.4)
Ramin	No	7	241 (82.5)	331 (84.4)	397 (78.7)
	Yes	7	19 (98.6)	4 (99.8)	5 (99.7)

Percentage fixation rate in parenthesis

**Table 4** Concentrations of chromated copper arsenate (CCA) components in leachates from pre-extracted treated wood and percentage of total leached after 14-day leaching

Timber	Pre-extract	Leached amount of CCA components <sup>a</sup>					
		Cu		Cr		As	
		(mg L <sup>-1</sup> )	% <sup>b</sup>	(mg L <sup>-1</sup> )	%	(mg L <sup>-1</sup> )	%
Geronggang	No	0.40 ± 0.07 c	4.3	1.11 ± 0.02 b	5.4	7.18 ± 0.14 a	46.5
	Yes	0.36 ± 0.04 c	4.0	0.10 ± 0.02 c	2.1	0.94 ± 0.02 b	3.3
Ramin	No	3.37 ± 0.60 a	40.0	5.47 ± 1.75 a	27.1	6.15 ± 0.13 a	45.4
	Yes	0.86 ± 0.03 b	5.2	0.14 ± 0.00 c	3.5	1.21 ± 0.00 b	4.7

<sup>a</sup>Cumulative amount of CCA components in leachate and leached blocks; <sup>b</sup>amount of each preservative component is represented as a percentage of the initial concentration (total retention) of the component in treated wood; Values in each column followed by the same letter do not differ significantly ( $\alpha = 0.05$ ) according to Duncan's multiple range test

fixed in wood in the absence of chromium. Our findings were in accordance with those of Hayes et al. (1994), who observed that changes in cell anatomy facilitated more rapid movement of preservative solution and therefore enhanced fixation.

Differences in chemical composition of wood especially the amount and type of lignin can affect the rate of fixation and preservative leachability (Pizzi 1990, Kartal & Lebow 2001). Bintangor, geronggang, medang, ramin and simpuh showed lower lignin content and recorded higher leaching compared with acacia (25.6%) and putat (28.8%) (Table 5). This finding suggested that, with some exceptions, lignin content affected the reduction of chromium for CCA fixation (Pizzi 1990). Reduction in leaching was previously reported to be caused by increased chromium hexavalent reduction to trivalent for arsenic precipitation and facilitated copper (II) oxidation (Radivojevic & Cooper 2007).

## CONCLUSIONS

Hexavalent chromium was rapidly reduced to trivalent chromium in the expressates of the timbers tested. Though reduction of hexavalent chromium was inappropriate for confirming CCA fixation, this could be used to measure the stabilisation of CCA components for indication of resistance to leaching. CCA fixation was confirmed using leaching tests. The fixation time for acacia and bintangor was 1 day, whereas that for putat, 7 days. Of the eight timbers investigated, acacia was most suitable for treatment with CCA because of its relatively high fixation rate and low leaching characteristics. Treatment of bintangor and putat in environmentally-sensitive areas was not recommended. Geronggang, medang, meranti rambai daun, ramin and simpuh were unsuitable for CCA treatment because of their high CCA leaching properties. The fixation of CCA components in geronggang and ramin

**Table 5** Lignin and pentosan contents of different timbers tested and radiata pine

Timber	Lignin content (%)	Pentosan content (%)	References
Acacia	25.6	17.4	Law & Wan Rosli (2000)
Bintangor	22.4	17.1	Peh et al. (1986)
Geronggang	23.1	13.6	Peh et al. (1986)
Medang	23.2	14.6	Peh et al. (1986)
Meranti rambai daun	26.3	9.0	Peh et al. (1986)
Putat	28.8	14.9	Peh et al. (1986)
Ramin	22.0	na	Zaihan (2012)
Simpuh	24.1	12.8	Peh et al. (1986)
Softwood (radiata pine)	27.0	14.7	Pettersen (1984)

na = not available

was enhanced by pre-extraction prior to CCA treatment. After pre-extraction, both species showed fixation time of 7 days, indicating that the fixation and leaching characteristics of geronggang and ramin were strongly influenced by extractive contents.

## ACKNOWLEDGEMENT

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