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Volatile Chemical Component Differences between Fully and Partially Dried Merbau (*Intsia* sp.) Wood Using Gas Chromatography-Mass Spectrometry (GC-MS) in Malaysia

(Perbezaan Komponen Kimia Meruap di antara Kayu Merbau (*Intsia* sp.) Kering dan Separa Kering Menggunakan Kromatografi Gas-Spektrometri Jisim (GC-MS) di Malaysia)

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ABSTRACT

Wood is one of the most common material found in homes and industrial fires, particularly in Malaysia. The abundance of different types of wood used to build structures, frames, furniture and others are important to take note as wood is a conducive material that can be used to initiate and propagate a fire, particularly in forensic fire cases. This study was conducted to provide the volatile chemical fingerprint of burnt Merbau (Intsia sp.) wood using gas chromatographymass spectrometry (GC-MS). The wood samples were prepared under two conditions; fully dried and partially dried. Six study replications of each condition were burnt outdoors and underwent an extraction process using activated carbon tablets. The tablets were incubated in an oven for 16 hours at 80°C. Next, the tablets were desorbed in two different types of solvent; pentane and ethanol. The pentane- and ethanol-desorbed volatile products were then introduced into the GC-MS. Volatile organic compounds obtained across both conditions were in the range of volatile acids, aromatics and its derivatives, p glucopyranose, phenolic derivatives and cycloalkane. The compounds emitted by fully and partially dried Merbau wood samples. Both solvents desorbed different types of volatile compounds. The burning behaviour of partially dried Merbau wood and is attributed to those documented from fully dried Merbau wood and is attributed to the composition of wood rather than the dry or wet conditions.

Keywords: Fire; Merbau wood; fully dried; partially dried; volatile products; activated carbon tablet; gas chromatographymass spectrometry (GC-MS)

ABSTRAK

Kayu adalah bahan yang biasa dijumpai di rumah dan industri, terutama di Malaysia. Pelbagai jenis kayu yang digunakan untuk membina struktur binaan, kerangka, perabot dan sebagainya adalah penting kerana kayu adalah bahan kondusif yang boleh memulakan dan menyemarakkan kebakaran, terutama dalam kes sains forensik. Kajian ini adalah bertujuan untuk menyediakan cap jari meruap kimia bagi kayu Merbau (Intsia sp.) menggunakan kromatografi gas-spektrometri jisim (GC-MS). Kayu telah disediakan dalam dua keadaan: kering sepenuhnya dan separa kering. Kayu-kayu ini telah dibakar secara terbuka sebanyak enam kali dan menjalani proses penyerapan menggunakan karbon aktif berbentuk tablet. Karbon aktif berbentuk tablet bersama sampel tersebut dimasukkan di dalam ketuhar selama 16 jam dengan suhu 80°C. Kemudian, karbon aktif tersebut dinyahjerap dengan pelarut pentana dan etanol, kemudian dianalisis menggunakan GC-MS. Sebatian organik meruap yang diperoleh adalah asid meruap, kompaun aromatik dan derivatifnya, _D-glucopiranosa, fenolik derivatif dan juga alkana siklik. Kompaun yang dihasilkan oleh kayu Merbau kering sepenuhnya dan kayu Merbau separa kering adalah hampir sama, kecuali ketiadaan asid malonik dan asid suksinik di dalam kayu Merbau separa kering. Kedua-dua pelarut menyerap jenis-jenis kompaun meruap yang berlainan. Keadaan kebakaran kayu Merbau separa kering adalah berbeza dengan kayu Merbau kering sepenuhnya dan dikaitkan dengan komposisi kayu Merbau, dan bukan keadaan kering atau basah.

Kata kunci: Kebakaran, kayu Merbau, kering sepenuhnya, separa kering, produk meruap, tablet karbon aktif, kromatografi gas-spektrometri jisim (GC-MS)

INTRODUCTION

Charred wood is commonly found in structural fires (The National Fire Protection Association 2017). This is because wood-based products are common materials encountered in

daily life as they appear in the form of furniture, bedding, flooring, structural support and others. In Malaysia, it is common for fire cases to involve some form of wood either in its natural state or that has been processed, as it is a common material found in structural buildings. Statistics released by the Fire and Rescue Department Malaysia showed an increase in the number of fire cases of different types of buildings, from 5447 cases in 2012 to 5817 cases in 2013 (Jabatan Bomba dan Penyelamat Malaysia 2012, 2013). As wood is one of the common materials present in a structural fire, the rise in the number of fire cases involving wood is alarming, particularly in forensic fire cases where fatalities are involved. Wood works as a very effective fire transfer material and with its good thermal conductivity (Suleiman et al. 1999), the transfer of heat and flames in high amounts either via conduction, convection and radiation is deemed to be very effective and rapid (Park et al. 2010). If present in sufficient amounts with a suitable initial heat source, the use of wood to conceal a crime can be very effective, particularly with the use of fires whereby the intention is to destroy and obliterate evidences in order to prevent identification and recovery, a description commonly referred to as arson (McMullin et al. 1983).

When wood is subjected to heat, it undergoes various physical and chemical changes, rendering it to different shades of yellow, brown, black and grey (Wakelyn et al. 2007) before finally reducing to ashes, if sufficient amounts of heat and oxygen are present (Hawley 1952). The first process that occurs, facilitating the ignition of wood is known as pyrolysis (Serban 1998). Pyrolysis refers to the "thermochemical decomposition of organic matter at elevated temperatures in the absence of oxygen" (The National Fire Protection Association 2017). During pyrolysis, large volume of volatile gases is released onto the surface above the wood. When these gases mix with oxygen in the presence of a heat source, combustion occurs, whereby flames are noted to be visually present. Gaseous products (benzene, carbon dioxide, methane and others), liquid products (tars, high molecular hydrocarbon and water) and solid products (char) are all by-products of pyrolysis and combustion (Vigouroux 2001). Along this process, a range of volatile products and substantial amounts of heat are released, that can cause the ignition of other items within the proximity, which in forensic fatality cases, would be that of human remains.

In Malaysia, one of the famous high profile cases involving wood and human remains was that of the murder of Datuk Sosilawati, whereby her body was burnt to ashes, in a bonfire, utilizing wood as a fuel (Sinar Harian Online 2015). This has also been the case in some parts of the world (Haglund & Sorg 2001; Schmidt & Symes 2015). In these cases, the detection and identification of human remains can be a very daunting task (Lim et al. 2019), particularly if human remains are not suspected to be present, or if the hard robust bone structures like the skull, teeth and femur, that are commonly used to indicate the presence of human remains, has undergone pre- or postmortem amputation and severe burning (Schmidt & Symes 2015). In addition, the physical appearance of burnt wood and burnt human remains in a fire would be somewhat similar; darkened and ashen structures, making it difficult to distinguish with the naked eye.

While pyrolytic data on the key indicators of human tissues have been studied and identified (Agu 2011; DeHaan et al. 2004; Gabriel 2015; Gabriel et al. 2017), there is lack in those of Merbau (Intsia sp.) wood. Merbau (Intsia sp.) wood is a coarse-textured, yellowish to orange-brown to reddish brown (Lumbermart 1999) hardwood species introduced from East Africa to Asia, the Tropical Islands and Northern Australia (Keong 2006). There are nine species of Merbau wood with genus Intsia spp. throughout the world but the most widespread are Intsia bijuga and Intsia palembanica (Tong et al. 2009). Other species that are not commonly encountered are Intsia amboinensis, Intsia bakeri, Intsia plurijuga, Intsia puberula, Intsia retusa and Intsia rhomboidea. Merbau (Intsia sp.) is a common wood used for structural furnishings such as windows, framing, flooring and doors in Asian countries, including Malaysia. The wood is an excellent timber sought after to make floorings and can also be used for furniture-making, truck bodies, stairs, shop fittings, turnery, fence posts and others. In Malaysia, it is one of the preferred wood of choice because of its excellent tensile strength in addition to its durability to termite attack (Chuan Soon Timber Trading 2018).

A number of studies on the pyrolysis of wood and volatile compounds emitted by wood in general, have been conducted by previous researchers (Branca et al. 2003; Jakab et al. 1997; Spokas et al. 2011). Branca et al. (2003) reported low-temperature pyrolysis products of wood to be in the range of furans, methyl acetate, 2propen-1-ol, ethyl acetate, 2-butanone, propionic acid, 3-pentanone, benzene, hydroxyacetaldehyde, formic acid, acetic acid, levoglucosan and other derivatives of these compounds. Levoglucosan, furan and its derivatives, hydroxyacetaldehyde and acetic acid were also identified as emission from thermal decomposition of white birch wood chips (Jakab et al. 1997). A study conducted by Spokas et al. (2011) recorded presence of acetone, benzene, methyl ethyl ketone, toluene, methyl acetate, propanal, octanal, pentanal and furan as volatile organic compounds from various wood species.

In terms of Merbau (*Intsia* sp.), Wencek & Prądzyński (2017) identified the presence of pentanal, hexanal and acetone from unheated Merbau wood. Risholm-Sundman et al. (1998) concluded that mainly carbonyl compounds such as aldehydes, ketone, carbonyl acids and their derivatives, together with pentanal and hexanal were present from hardwood samples. Witterseh (2004) identified aliphatic hydrocarbons, bromomethane, cyclododecane, acetic acid, ethanol, hexanal, octanal, pentane and pentanal in his volatile compound study on hardwood. While the volatile products generated under standard conditions are important, these studies on Merbau (*Intsia* sp.) wood and other hardwood did not cover aspects of thermal decomposition products of the said wood, particularly under dried and wet conditions. These conditions could alter the volatility of the

wood itself and in turn, the pyrolysis products generated during the process of burning. Thus, this work would like to look into generating a datasheet of knowledge of key pyrolytic indicators of Merbau (*Intsia* sp.) wood, in order to aid investigators and forensic scientist, to filter and extrapolate relevant pyrolytic data from fire samples that could potentially hold information on the presence of human remains and the identification of the type of wood used/present in a fire.

In this work, Merbau (*Intsia* sp.) wood samples were subjected to two conditions; fully dried and partially dried. The dry wood samples represent the condition of Merbau (*Intsia* sp.) wood in real fire scene situations where the wood is used to make furniture and other structural materials that would have undergone the drying process in the respective factories (Simpson 1999). The partially dried wood is intended to replicate water absorbed into the wood during the fire extinguishment process and for cases where wet wood is used to initiate or propagate the fire. The chemical fingerprints of Merbau wood samples in both fully and partially dried conditions were analysed, compared and reported.

MATERIALS AND METHODS

STANDARD TEST MIXTURE

Twelve high purity compounds; benzene, ethyl-benzene, butyl-benzene, toluene, octane, p-xylene, propyl-benzene, decane, tridecane, tetradecane, pentadecane and dodecane, were prepared in a 1 mg/mL standard solution (Sigma Aldrich, UK) and placed into six individual GC vials (Fisher Scientific). A total of seven analytical reagent grade solutions of acetic acid, ethanol, acetone, 2-butanone, propionic acid, formic acid and phenol compounds, were prepared in a 1 mg/mL standard solution and placed in six individual GC vials for each solution. These compounds were selected because they have been reported to be the type of volatile compounds emitted by wood, in general and from Merbau wood (Branca et al. 2003; Hiroyuki et al. 2012; Spokas et al. 2011). All the contents of the GC vials (Fisher Scientific) were injected into the GC-MS (Shimadzu, UK).

GC-MS SETTINGS

The instrument was set up with an injector temperature of 250°C and set to split mode with a split ratio of 20:1 and split flow of 32.7 mL/min. The column used was a DB-1MS (22 m length \times 0.2 mm inner diameter \times 0.33 mm film thickness) (Crawford Scientific, UK) column. The oven initial temperature was set at 40°C and held for five minutes. The temperature was ramped at 5°C/min until it reached 280°C which is the final temperature and held for two minutes. Data obtained from GC-MS were analysed based on their mass-to-charge ratio (m/z) against the National Institute of Standards and Technology (NIST)

library (NIST/EPA/NIH Mass Spectral Library) and data generated from the standard test mixtures.

SAMPLE PREPARATION

Untreated pallet of Merbau wood of less than 5 years old were purchased from Kean Guan (Lim Kee) Sdn Bhd, Salak Selatan, Kuala Lumpur. The wood pallets were not treated or layered with any chemicals, protectors or finishing. The pallets were only processed with sandpaper to smoothen out the wood's fibre.

FULLY DRIED

Fully dried refers to the stage of wood where it does not undergo anymore weight loss (loss of moisture) when subjected to extended heating. The Merbau (Intsia sp.) wood was cut into 12 individual pieces measuring 4 cm $(length) \times 4 cm (width) \times 1 cm (height) with fixed weight$ of 25 g \pm 1.5 g using a hacksaw and a bench-vice. The wood sample underwent the drying process in an oven with temperatures of 101-105°C. The wood sample was weighed every half an hour using an analytical balance to observe weight loss. When no further weight loss of the wood was observed, the sample has achieved its final weight (Simpson 1999), whereby the Merbau wood sample has now reached its fully dried stage and the time taken to reach this stage was noted. From the experiment, it was noted that the time taken to fully dry a Merbau wood sample was seven hours.

PARTIALLY DRIED

Partially dried refers to the stage of wood where half of the moisture content is still present in the wood upon heating and it is determined by halving the time taken to attain the fully dried stage. The time to attain partially dried stage for Merbau wood sample was half the time taken for the wood samples to reach the fully dried stage. Thus, the time taken to attain partially dried stage was at three and a half hours. The process of drying is similar to those applied in fully dried wood samples.

PYROLYSIS PROCESS

A retort stand was placed in an outdoor setting at the Forensic Science Simulation Site in Universiti Kebangsaan Malaysia, Bangi, Selangor. A tin can containing the wood sample with the lid removed was attached to the retort stand while a portable gas canister (Campingaz; International) was placed under the tin can at approximately 3 cm from the tip of the canister to the base of the tin as a heat source. The temperature of the experiment was monitored using a k-thermocouple (Lascar Electronics, UK) connected to a digital thermometer (Lascar Electronics, UK) which was placed in the headspace of the tin can. The Merbau wood sample was allowed to burn until the ignition of wood

occurred, and the burning process was continued for five minutes thereafter. If ignition did not occur, the Merbau wood samples were left to burn until it was two thirds burnt. This was determined based on the depth of char on the wood (two thirds of the entire thickness of the wood). The Bunsen burner and the k-thermocouple were rapidly removed ten seconds before the five-minute post-ignition burning time was attained to facilitate oxygen deprivation by placing an empty tin lid over the tin. If ignition did not occur, once the sample has reached two thirds burnt, the Bunsen burner and k-thermocouple were immediately removed and an empty tin lid was placed on the tin. The lid was then removed from the retort stand after it was left to cool for four minutes and immediately sealed with another lid holding an activated carbon tablet. The activated carbon tablet was attached on a paper clip and held by a magnet on the lid. The sealed tin can was incubated in an oven for 16 hours at a temperature of 80°C. After completing the incubation process, the activated carbon tablet was removed, broken into half and immediately desorbed respectively in 1 mg/mL pentane (Fisher Scientific) and 1 mg/mL ethanol (R&M Chemical), each containing 0.5 mg/mL of tetrachloroethylene (internal standard) in a 1 mL GC vial (Fisher Brand). Pentane was used as one of the solvent as it is a recommended solvent in fire debris analysis (Tranthim-Fryer 1990). Ethanol was chosen as another solvent because ethanol is semi-polar and has higher affinity towards potential polar volatile compounds (Harper 2000). The sample was then introduced into

the GC-MS (Shimadzu, UK) for the separation, detection and identification process. All steps in this process were repeated in six study replications of each condition; partially and fully dried. The data obtained for each condition were analysed and identified.

RESULTS AND DISCUSSION

Volatile organic compounds emitted from fully dried burnt Merbau wood samples across six study replications desorbed in pentane and ethanol are tabulated in Table 1 -4, alongside with their respective total ion chromatogram (TIC). Across six study replications desorbed in pentane (Table 1, Figure 1), nine compounds were detected and identified to be in the range of volatile acids and its derivative, as the main components of the volatile products generated, together with benzene derivative, phenol derivative and benzene sulfonamide. Majority of these compounds were present in more than half of the study replications.

Similarly, across the six study replications desorbed in ethanol (Table 2, Figure 2), volatile acids and its derivative were detected, although fewer in numbers (n = 5) in comparison to pentane (Table 1), together with the additional presence of graduate graduate and cyclododecane derivative, that were not detected in the pentane desorption. Only two of the five compounds were present in more than half of the study replications.

No	Retention time (min)	Compound	Study repl			dy replica	ication		
			1	2	3	4	5	6	
1	1.465	Malonic acid derivative		\checkmark		\checkmark		\checkmark	
2	5.046	Benzene derivative				\checkmark			
3	5.325	Propionic acid derivative		\checkmark					
4	5.647	Succinic acid derivative		\checkmark					
5	10.180	Propanoic acid derivative				\checkmark			
6	11.680	Fumaric acid derivative		\checkmark			\checkmark		
7	17.858	Benzene sulfonamide		\checkmark		\checkmark			
8	17.944	Phenol derivatives		\checkmark			\checkmark		
9	22.012	Acetic acid methyl ester derivative		\checkmark			\checkmark	\checkmark	

TABLE 1. Volatile compound emitted by fully dried burnt Merbau wood samples desorbed in pentane

TABLE 2. Volatile compound emitted by fully dried burnt Merbau wood samples desorbed in ethanol

No	Retention time (min)	Compound	Study replication			tion		
			1	2	3	4	5	6
1	12.369	Benzoic acid methyl ester derivative		\checkmark			\checkmark	
2	15.717	D-glucopyranose	\checkmark				\checkmark	
3	22.058	Cyclododecane derivative		\checkmark				
4	22.097	Acetic acid methyl ester derivative						
5	22.172	Thiopheneacetic acid	\checkmark		\checkmark	\checkmark		\checkmark

No	Retention time (min)	Compound	Study replication					
			1	2	3	4	5	6
1	1.465	Propionic acid derivative	\checkmark	\checkmark	\checkmark	\checkmark		\checkmark
2	5.046	Benzene derivatives						
3	5.325	Propanoic acid derivative						
4	5.647	Fumaric acid derivative						
5	10.180	Phenol derivative				\checkmark		
6	11.680	Benzene sulfonamide	\checkmark				\checkmark	
7	17.858	Acetic acid methyl ester derivative		\checkmark	\checkmark	\checkmark		

TABLE 3. Volatile compound emitted by partially dried burnt Merbau wood samples desorbed in pentane

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TABLE 4. Volatile compound emitted by partially dried burnt Merbau wood samples desorbed in eth	IIIIIIII

No	Retention time (min)	Compound	Study replication		tion			
			1	2	3	4	5	6
1	11.867	Cyclododecane	\checkmark					
2	11.958	Benzoic acid methyl ester derivatives			\checkmark	\checkmark	\checkmark	
3	15.150	D-glucopyranose						
4	21.126	Acetic acid methyl ester derivative	\checkmark	\checkmark		\checkmark		\checkmark
5	22.158	Thiopheneacetic acid		\checkmark				



FIGURE 1. Total Ion Chromatogram of study replication 2 of burnt fully dried Merbau wood desorbed in pentane

With reference to the overlay of time-temperature profile of fully dried burnt Merbau wood samples (Figure 3), all study replications did not auto-ignite during the burning process, hence they were burned until two-thirds burnt. The maximum temperature attained during the burning process were in the range of 200°C to 320°C. This auto-ignition is usually characterized by a sudden and sustainable spike in temperature above 400°C.

The volatile compounds emitted by partially dried burnt Merbau wood samples across six study replications have been identified and reported in Table 3, Figure 4 for samples that have been desorbed using pentane and in Table 4, Figure 5, for samples desorption in ethanol. Similar to the compounds present in fully dried Merbau samples in Table 1, the compounds generated by partially dried Merbau wood samples (Table 3) were volatile acids and its derivatives, with the absence of malonic acid and succinic acid in the partially dried Merbau samples. Also detected in the partially dried samples were benzene derivative, phenol derivative and benzene sulfonamide. A total of seven compounds were detected with majority being present in more than half of the study replications. The type and number of compounds detected in partially dried wood samples (Table 4) desorbed in ethanol were the



FIGURE 2. Total Ion Chromatogram of study replication 5 of burnt fully dried Merbau wood desorbed in ethanoll



Each colour refers to respective study replications.

FIGURE 3. Overlay of six time-temperature profiles of six study replications of fully dried burnt Merbau wood

same as those generated from the fully dried wood samples (Table 2), which were volatile acids and its derivatives, $_{\rm D}$ -glucopyranose and cyclododecane. Here, only two out of the five compounds were detected in more than half of the study replications.

The overlay of the time-temperature profile of partially dried burnt Merbau wood samples is illustrated in Figure 6. During the burning process, two out of the six study replications (study replication 2 and 6) ignited and sustained the flames upon ignition, attaining maximum temperatures of above 600°C. Study replication 3 and 4 experiences a short burst of auto-ignition but the flames

were not sustained for more than 15 seconds. Study replication 1 and 5 did not auto-ignite, and together with study replication 3 and 4, all four study replications were subjected to two-thirds burning. Maximum temperatures attained across these four study replications were in the range of 200°C to 450°C.

The pyrolysis product of burnt, fully and partially dried Merbau wood samples desorbed in pentane were noted to be similar in terms of the range of compounds present; volatile acids and its derivate together with benzene and phenol derivate (Table 1 and Table 3). The only differences observed were the absence of two volatile acids, i.e.



FIGURE 4. Total Ion Chromatogram of study replication 2 of burnt partially dried Merbau wood desorbed in pentane



FIGURE 5. Total Ion Chromatogram of study replication 1 of burnt partially dried Merbau wood desorbed in ethanol



Each colour refers to respective study replications.

FIGURE 6. Overlay of six time-temperature profiles of six study replications of partially dried burnt Merbau wood

succinic acid derivative and malonic acid derivative, in partially dried Merbau wood samples. In terms of the pyrolytic profile generated across the burning of fully and partially dried Merbau wood samples desorbed in ethanol (Table 2 and Table 4), a 100% match was noted in the type and number of volatile compounds generated. Compounds generated were of volatile acids and its derivate together with the presence of p-glucopyranose and cyclododecane derivate. The main differences noted across both desorbed solvents were that; 1) samples desorbed in pentane generated more volatile acid and derivatives than those desorbed in ethanol and, 2) samples desorbed in ethanol generated glucopyranose and cyclododecane that were not present in pentane desorption. A comparison between the products generated across both fully and partially dried Merbau wood samples was conducted and the results were tabulated in Table 5.

According to Laine (2005), wood is comprised of cellulose, hemicellulose, lignin and pectin. Pectin coats the outermost layer, followed by hemicellulose, cellulose

and lignin, impregnated within these structures (Cotton Incorporated 2014). These components will undergo decomposition as they are exposed to heat and release specific volatile compounds, with pectin and hemicellulose subjected to burning first followed by cellulose and lignin as heat penetrates the outer layer moving towards the inner layers (Wencek & Prądzyński 2017). With reference to Table 1 and Table 3, respectively, volatile acids were the main compounds formed across the six study replications of burning of fully and partially dried Merbau wood. This is not surprising as the productions of volatile acids during the initial stages of heating are mainly from the decomposition of hemicellulose as hemicellulose decomposes at 200-260°C (Mohan et al. 2006). Risholm-Sundman et al. (1998) also stated that volatile acids are present abundantly in hardwood species in comparison to softwood species. Balaban & Ucar (2003) commented that one of the main volatile acids that exist in wood is acetic acid, which were detected in both conditions of Merbau wood in this study, as displayed in Table 5.

TABLE 5. Comparison of pyrolysis product emitted between burnt, fully and, partially dried Merbau wood desorbed both in pentane and ethanol (No GC-MS output for this as it is a comparison of both conditions and solvents from the analysis above)

Compound		Fully dried	Partially dried
	Pentane		
Malonic acid derivatives		\checkmark	
Propionic acid derivatives		\checkmark	\checkmark
Benzene derivatives		\checkmark	\checkmark
Succinic acid derivatives		\checkmark	
Fumaric acid derivatives		\checkmark	
Phenol derivatives		\checkmark	
Propanoic acid derivative		\checkmark	
Benzenesulfonamide		\checkmark	
	Ethanol		
D-glucopyranose		\checkmark	
Cyclododecane		\checkmark	
Thiopheneacetic acid		\checkmark	\checkmark
Benzoic acid methyl ester		\checkmark	
*Acetic acid methyl ester derivative		\checkmark	

* present in both pentane and ethanol desorption

As the burning temperature increases above 300°C, cellulose is the second component that starts to breakdown (Bradbury et al. 1979; Jakab et al. 1997; Sinha et al. 2000). Cellulose is made up of _D-glucose chains that has underwent polymerisation to form repeatable units of _D-glucopyranose (Hedge et al. 2004; Matthews & Mauersberger 1947). Cellulose pyrolysis can occur via two different mechanisms that are temperature dependent; < 300°C (primary) or > 300°C (secondary; main and char decomposition) (Nimlos & Evans 2002; Price et al. 1997). During the initial stage, at temperatures below 300°C, physical changes occur within the amorphous region of cellulose, whereby the structure loses water and undergoes cross linking. If temperature

remains below 300°C, the main compound that is formed in this pathway is known as char (Nimlos & Evans 2002). When temperatures reach above 300°C, heat penetrates the crystalline region of the wood, activating random scission of the C-O bond, which in turn, unwinds the chain to form levoglucosan, p-glucopyranose and p-glucose (Beyler & Hirschler 2002). In line with the literature (Jakab et al. 1997), p-glucopyranose, were present in both fully and partially dried Merbau wood that has been burnt in this study (Table 5). The presence of p-glucopyranose proves that the decomposition of wood has reached the moderate pyrolysis stage (Sinha et al. 2000). With further heating (char decomposition), furans, organic acids, aldehydes, alkanes and alkenes, and other compounds are formed (Sundqvist et al. 2006), a number of which were documented in this study (Table 5). Benzoic acid and esters of higher fatty acids chains that were found in this study has also been corroborated in previous work (Pakdel et al. 1994). The presence of cyclododecane in both conditions of Merbau wood samples as shown in Table 5 indicates the uniqueness of Merbau wood, as it is a compound specifically emitted by Merbau wood. This is shown in the study conducted by Witterseh (2004) who stated that one of the volatile organic compounds released by Merbau wood to be cyclododecane.

Table 5 also shows the presence of benzene derivatives and phenol derivatives in both conditions of Merbau wood which are in agreement with previous studies as they were identified as products emitted by wood in general (Branca et al. 2003; Spokas et al. 2011). The production of phenol and its derivatives were primarily produced by the decomposition of lignin, a type of phenolic polymer, as lignin is reported to undergo pyrolysis at temperatures of 280-500°C (Liu et al. 2008; Mohan et al. 2006; Sinha et al. 2000).

In previous literatures, the emission of volatile compounds specifically n-aldehydes from Merbau (Instia sp.) wood samples have been reported (Risholm-Sundman et al. 1998; Wencek & Prądzyński 2017; Witterseh 2004). Wencek & Prądzyński (2017), who studied the emission of volatile compounds released from Merbau (Intsia sp.) wood at room temperature reported the presence of pentanal and hexanal. Risholm-Sundman et al. (1998) concluded that aldehydes, particularly pentanal and hexanal, were detected from Merbau wood when exposed to heat at 80°C. At temperatures of 40°C, volatiles such as hexanal, octanal and pentanal were detected by Witterseh (2004). However, in this current study, *n*-aldehydes were not reported to be present (Table 5). In previous literature, temperatures to which the wood was exposed to were below 80°C (Risholm-Sundman et al. 1998). In this study, the temperatures attained were in the range of 200-650°C. These high temperature ranges could have decomposed the unstable -CHO bond that form *n*-aldehydes, particularly above 160°C, in an uncontained environment. Any possible *n*-aldehydes generated could have underwent thermochemical breakdown or consumed by the flames at high temperatures and burning duration (Agu 2011; DeHaan et al. 2004; Gabriel et al. 2017).

Out of the six study replications, none of the fully dried Merbau wood underwent auto-ignition (Figure 3) whereas two out of six study replications of partially dried Merbau wood experienced auto-ignition, while the remaining four did not (Figure 6). The phenomenon of auto-ignition occurs when wood is heated and the volatile pyrolysis gaseous products are released from the wood surface that acts as a fuel and mixes with the ambient air. If the concentration of fuel and air mixture is sufficient enough and remains within the flammability limit of wood, in the presence of heat, auto-ignition occurs (McAllister 2013).

The ignitability of wood depends on several factors including density, thickness, distance from heat source, heat flux, intensity of heat, species of wood, thermal ignitability and moisture content (Babrauskas 2002; Shen et al. 2007). Thickness and species would not be an issue in this work as these factors were constant across study replications. The moisture content across both fully and partially dried Merbau wood samples are the most prominent differences across both conditions of wood. Moisture content is a known factor that effects the thermal ignitability of wood. When moisture content is high in a piece of wood, it decelerates the surface temperature due to large consumption of energy to evaporate water rather than generating volatiles. In other words, pyrolysis occurs rapidly in wood that is dried rather than wet (Yang et al. 2003). According to Shen et al. (2007), wet wood loses majority of water at 100°C, mostly from its outermost layer, however there will be some water that migrates deeper into the wood (Grønli & Melaaen 2000; Yang et al. 2003) creating a three zone wood system consisting of top pyrolysis zone, middle dehydrating zone and inner wet zone. Moisture in the wet zone eventually evaporates when temperature reaches 240°C (Schaffer 1973). Mild pyrolysis only begins at 240°C to 320°C whereas rapid pyrolysis occurs at temperatures above 320°C, facilitating the generation of volatile gases that in turn causes autoignition. In theory, given the lower moisture content in fully dried wood, it is the group of wood most likely to auto-ignite in comparison to the partially dried version. However, the results were the opposite in this work. This could largely be due to the chemical composition of the wood itself. Although the general composition is similar across any cellulosic material, each cut of the wood might comprise different composition with higher or lower levels of hemicellulose or cellulose or lignin or pectin or any combination of these components. White & Nordheim (1992) reported that cellulose is the main contributor to initiate flaming combustion (auto-ignition) in wood. More importantly, the percentage and deposition of chemical compounds such as triglyceride fats in specific cuts of wood would differ from one another (Chen 2014). These fat cells are the ones that are responsible for the auto-ignition of wood at the initial stages of pyrolysis when temperatures attain 315°C (Haessler 1969; Pakdel et al. 1994). These fat cells could have been higher in abundance in study replication 2 and 6 (Figure 6) compared to the rest of the study replications, be it in fully dried (Figure 3) or partially dried (Figure 6) conditions, facilitating auto-ignition. Babrauskas et al. (Babrauskas 2002) mentioned in their work when wood is heated for extended durations, there is higher changes of the wood to auto-ignite, hence the purpose of burning the wood until two thirds burnt. They documented the general auto-ignition of wood to be at 250°C (Babrauskas 2002). However, it was also mentioned that amidst glowing heating from a heat source, the wood may or may not follow flaming combustion and this varies from one piece of wood to another, as observed in this study. Ambient conditions and airflow velocity also affects the ignitability of wood samples since both can change the ignition temperature and ignition delay time of wood (McAllister 2013). In outdoor conditions as applied in this work, airflow and oxygen supply to the burning wood sample varied tremendously and hence could have altered the auto-ignition factors surrounding Merbau wood.

Two different solvents were utilized in this work in order to observe the desorption effects of these solvents towards the volatile compounds adsorbed onto the activated carbon tablet upon burning, and to ensure pyrolytic compounds generated during the burning process are not missed due to the selectivity of certain solvents. It is known that certain solvents desorb certain target compounds effectively in comparison to the rest and this is largely due to the chemical characteristic of the solvent and compound; polar, semi-polar or non-polar, volatility, molecular weight, lipophilicity and water solubility (Boman & Maibach 2000). As the target compounds in this work are not fully known, the use of two different polarity solvents such as pentane and ethanol were the logical choice in order not to discriminate any possible target compounds. Polar solvents were not utilized as the column used in this work is a nonpolar column and volatiles generated during pyrolysis are almost always non-polar or semi-polar. The differences detected in the desorption of both solvents were presented across Tables 1, 2, 3 and 4. The variation in the absence and presence of certain compounds detected between both conditions of Merbau wood desorbed in pentane and ethanol are largely due to the polarity and chemical characteristics of both solvents. While pentane is an organic solvent which has high affinity for non-polar, low and high molecular, weight compounds (PubChem Compound Database Pentane 2018), ethanol on the other hand, is semipolar and its hydroxyl group facilitates the solvent to be miscible in a range of compounds including cyclic alkanes (PubChem Compound Database Ethanol 2018), acids and glucopyranose, as reported in this study, in Table 2 and Table 4. The hydroxyl groups in the acids and pyranose chains facilitate these two groups of compounds to have higher affinity towards the hydroxyl group in ethanol, rendering desorption into the solvent (National Institute of Standards and Technology 2017a, 2017b).

Merbau wood pallets used in this work were untreated, free from any form of chemicals other than water. This is to ensure no additional volatiles were detected that were not from the wood itself. However, when wood is processed to make furniture, structural support and materials for buildings and furnishings, the condition of the wood at that stage is one that has been largely processed under various physical and chemical processes. These chemicals are usually added to strengthen the wood and to aid in correcting any unfavourable features of the wood such as ease to oxidation, resistance to water, resistance to fire, resistance to rotting and also for aesthetic appeal (Bartlett et al. 2019; Stamm & Harris 1953). The presence of any

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additional chemicals would indefinitely alter the burning profile of wood, either directly or indirectly, and in turn, potentially the chemical volatile products generated by wood. Friquin (2011) reported that virgin wood cracks easily upon heating, aiding in the rapid release of volatiles in comparison to treated wood. Wood that has been treated for enhanced fire resistance for instance, would have higher thermal resistance to heat, that in turn causes a delayed onset of pyrolysis or facilitates heat flux and thermal decomposition at higher than usual temperatures (Drysdale 2011; Moghtaderi et al. 1997). Torero (2015) reported that similar species of wood can experience auto-ignition at various temperature ranges, some differing up to 150°C from each other due to factors such as the chemical processing of wood and external factors such as thickness, density and moisture content. Permeability also differs from wood that is treated or untreated particularly in terms of moisture content (Friquin 2011). Wood that has been treated to enhance resistance to water are less permeable due to alteration in the grain direction that in turn impedes heat transfer and penetration of heat flux to the surface of the wood, causing lesser volatile chemical compounds to be formed (White & Dietenberger 2001). All these factors have to be taken into consideration when analysing wood from fire scenes, particularly in the generation of standard volatile chemical compounds data of specific wood species.

CONCLUSION

The pyrolysis products generated between fully and partially dried Merbau wood samples were mostly similar (volatile acids and its derivatives, benzene and phenol derivates, p-glucopyranose and cyclododecane) but differ in the absence of malonic acid and succinic acid in partially dried wood samples. This work also highlighted the effects of different polarity solvents towards the desorption of polar and semi-polar compounds. Ethanol desorped glucopyranose and cyclododecane that were not present in pentane, whereas pentane desorbed more volatile acids, phenolic and benzene compounds, that were not present in ethanol. No chemical profile differences were noted across both conditions of fully and partially dried Merbau wood when burnt. However, in terms of the physical properties, two out of the six study replications in partially dried Merbau wood auto-ignited whereas the remainder did not. Thus, supporting the fact that the chemical composition of wood, despite being from the same species and burnt under the same conditions, do differ from one another and can directly alter the burning properties of Merbau wood.

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