

Characteristics of Several Types of Palmyra Stem (*Borassus sp*) Extractives Based on GCMS (Gas Chromatography and Mass Spectroscopy) Analysis

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Abstract:- Palmyra palm extractives are one of the wood chemical components that can have a negative or positive effect on the utilization of palmyra palm stems. Analysis of the content of extractive compounds can be carried out by pyrolysis GCMS analysis. Samples in the form of fine powder of palmyra stems were used in the extraction process, using: A) cold-water extraction, B) hot-water extraction, and C) ethanol-benzene extraction. The results of the GCMS analysis showed that there were 35 cold-water soluble extractives (Method A), 15 hot-water-soluble extractives (Method B), and 30 ethanol-benzene soluble extractives components (Method C). In method B, the toxic compounds found were Phenol (4.06%) and Methane, tetranitro- (CAS) Tetranitromethane (1.24%), while in method A, the toxic compounds were Phenol (6.21%); Methane, tetranitro- (CAS) Tetranitromethane (3.19%); Guanosine (CAS) Guo (1.80%); 2-propenyl decanoate (2.46%) and Lactaropallidin (2.01%). The non-bioactive compounds, which are lignin derivative, detected in those three types of extractives, were Phenol, 2-methoxy- (CAS) Guaiacol; and 2,6-dimethoxy- (CAS) 2,6-Dimethoxyphenol. Beside these two compounds, there were also lignin derivative compounds such as Phenol compounds, 3,4,5-trimethoxy- (CAS) Antiarol; Phenol, 2,6-dimethoxy-4-(2-propenyl)- (CAS) 4-Allyl-2,6-dimethoxyphenol; Benzaldehyde, 4-hydroxy-3,5-dimethoxy- (CAS) Syringaldehyde; Phenol (CAS) Izal; Phenol, 4-(3-hydroxy-1-propenyl)-2-methoxy- (CAS) Coniferyl alcohol; and Phenol 2-methyl-5-(1-methylethyl)- Carvacrol. Each types of extractive from palmyra stem contained specific organic compounds. Therefore, in order to produce extractive-free palm stems, multilevel extractions were used, and to remove a certain compound, it should be extracted with an appropriate solvent.

Keywords:- Palm; Monocots; Maluku Indonesia; Sap; Parenchyma; Wood Chemistry.

I. INTRODUCTION

The decrease in the supply of timber forest products has caused the increase in the utilization of non-timber forest products such as monocot species that can become an alternative raw material for wood. Lontar or palmyra, one of

the monocot species, has been cultivated in the Southwest Maluku mainly for its sap. However, the bole of the palmyra tree also contains valuable wood materials.

The outer part of the palmyra tree trunk has a good quality wood. It is hard, heavy and black in color. Based on the strength classification of the Indonesian wood, these wood materials are classified as strength class II-III. This material had fracture at proportional limit for Modulus of Rupture (MOR) at 100.04 MPa. The MOR of the palmyra rod parallel to the grain was 49.68 MPa, perpendicular to the grain was 22.47 MPa, shear parallel to the grain was 9.22 MPa and side hardness was 48.87 MPa (Lempang et al. 2009).

For the preservative treatment properties of the palmyra wood materials, the results of the termite resistance test by Lestari (2019) showed that the palmyra stems lost 23.8% of their initial weight. The resistance to termite attack of palmyra stems is classified as class V, or weight loss after testing more than 18.9% (Indonesian National Standard 01-7207-2014). Wood materials with higher extractive content are more resistant to termite attack than the one with lower extractive content. However, the durability of wood materials are highly dependent on the bioactive compounds contained in those extractives (Lestari and Pari 1990). Beside their positive effects, extractive substances also have negative effects in the manufacture of artificial boards due to their inhibitory properties in the gluing process (Maulana et al. 2017).

There are different compounds of extractive constituent depending on the solvent used in the extraction process. In general, non-polar solvents can produce oil-soluble extractives, while polar and semi-polar solvents can produce oil-insoluble extractives. Extractive compounds that dissolve in semi-polar solvents consist of terpenoids and phenolic or almost all groups of compounds (Lukmandaru 2009). Meanwhile, polar solvents can produce extractives containing tannins, gums, sugars, dyes, and starch (Fengel & Wegener 1984). The composition of the compounds that make up each type of extractive can be analyzed using GCMS (Gas Chromatography and Mass Spectroscopy) pyrolysis analysis (Hotmian et al. 2021).

GCMS analysis is one of the most important chemical separation methods, which separates the extractive compounds in a chromatographic column. Pyrolysis GCMS analysis has the ability to provide complete information quickly when compared to the qualitative identification using the Meyer and Wagner method (Maakh et al. 2021). The GCMS analysis method works by reading the spectrum contained in the two combined methods. The number of compounds contained in the sample will appear based on the formed number of peaks. Furthermore, based on known retention time data from the literature, one can identify the compounds present in the sample. The most important information obtained from GC spectra is the retention time for each compound, and for MS (Mass Spectroscopy) spectra, one can obtain information regarding the relative molecular masses of those compounds. Using GCMS analysis, the objective of this research was to determine the composition of the extractive compounds of palmyra palm stems.

II. RESEARCH METHODS

A. Method

Palmyra palm stems were obtained from the community-owned forest in Southwest Maluku district. Based on the bole axial direction, samples were taken from the base, middle and top parts, while based on the radial direction samples were taken from the outer and inner parts. The samples of palmyra stem particles were dried and ground using a Willey mill and sieved using 40 and 60 mesh sieves to obtain the samples in the form of powder with size of -40/+ mesh for further extraction. The chemicals used in this research were obtained from PT Merck Chemicals and Life Sciences (MCLS) including ethanol and benzene.

B. Procedure

The extractives were obtained by extracting the powder samples and further dried to form a paste. The extraction process was carried out using three methods, namely, (A)

cold-water soluble extractive using TAPPI T 207 om-88, (B) hot-water soluble extractive using TAPPI T 264 om-88, and (C) ethanol-benzene soluble extractive using TAPPI T 257 cm-85. The extractives were dried at 103 ± 2 °C for one hour. About 0.1 g of extractives was placed in a pyrolysis tube and heated to release gas. Identification of organic compounds contained in the extractives was carried out by pyrolysis-Gas Chroma-tography Mass Spectrometry (py-GCMS). Observation of organic compounds was based on the gas produced from heating the extractive samples for 1 hour at 300 °C. The results obtained were in the form of spectrums. The peaks of the spectrum were then compared with that of the library data to determine the formed compounds.

III. RESULTS AND DISCUSSION

A. Results

The results of the GCMS analysis for the extractive organic compounds showed that there were 30 of the compounds from method A (Table I), 15 from method B (Table II), and 35 from method C (Table III). In method A (Figure 1), the phenol, 2-methoxy- (CAS) Guaiacol compound had the highest concentration (15.26%) detected at the retention time of 15.565 minutes. In method B (Figure 2), the carbamic acid, mono-ammonium salt compound, had the highest concentration (52.81%) detected at the retention time of 6,557 minutes. In method C (Figure 3), hexadecanoic acid (CAS) Palmitic acid compound had the highest concentration (12.25%) detected at a retention time of 22,411 minutes. Phenol, 2-methoxy- (CAS) Guaiacol and Phenol, 2,6-dimethoxy- (CAS) 2,6-Dimethoxy-phenol Phenol, 2,6-dimethoxy- (CAS) 2,6-Dimethoxyphenol were two organic compounds detected using those three methods but at different retention times. The highest concentrations of these two compounds were found in method A, i.e., Phenol, 2-methoxy- (CAS) Guaiacol (15.26%) and Phenol, 2,6-dimethoxy- (CAS) 2,6-Dimethoxyphenol (6.60%).

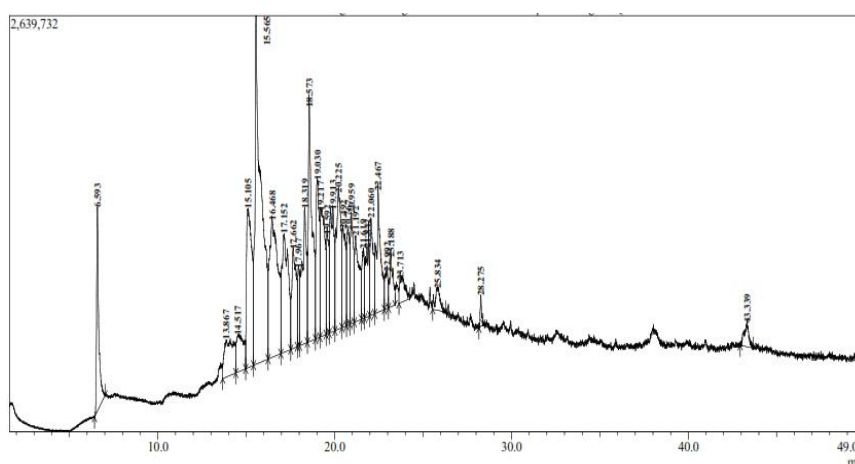


Fig. 1. Py-GC/MS trace of cold-water soluble extractive (Method A). Pyrolysis at 400 °C, for 60 minutes. Peak numbers and names refer to Table I.

TABLE I. IDENTIFIED MASS PEAKS IN PYROLYSIS OF COLD-WATER SOLUBLE EXTRACTIVE (METHOD A)

Peak	Product	Concentration (%)
1	Methane, tetranitro- (CAS) Tetranitromethane	3.19
2	Acetamide (CAS) Ethanamide	2.45
3	1,4-Butanediol, 2,3-bis(methylene)- (CAS) 2,3-Dimethylene-1,4-butanediol	1.90
4	Phenol (CAS) Izal	6.21
5	Phenol, 2-methoxy- (CAS) Guaiacol	15.26
6	1-Butanamine, N-ethyl-N-nitroso- (CAS) ETHYL BUTYL NITROSAMINE	8.40
7	Butanamide, N-[(acetylamino)carbonyl]-2-bromo-2-ethyl- (CAS) Acetylcarbromal	5.33
8	Oxirane, 2-butyl-3-methyl- (CAS) 2,3-Epoxyheptane	3.15
9	(+)-.alpha.-Cyperone	1.01
10	2-METHOXY-6-VINYLPHENOL	4.44
11	Phenol, 2,6-dimethoxy- (CAS) 2,6-Dimethoxyphenol	6.60
12	4-Methyl-2-hexanol	3.62
13	3,6,9, 12-Tetraoxahexadecan-1-ol (CAS) Tetraethylene glycol, monobutyl ether	4.31
14	Guanosine (CAS) Guo	1.80
15	Dodecanoic acid (CAS) Lauric acid	3.86
16	Phenol, 4-(3-hydroxy-1-propenyl)-2-methoxy- (CAS) Coniferyl alcohol	4.88
17	2-propenyl decanoate	2.46
18	.alpha.-D-Glucopyranoside, .beta.-D-fructofuranosyl (CAS) Sucrose	1.97
19	Dodecane, 1,1'-oxybis-(CAS) DIDODECANE ETHER	2.42
20	Tetradecanoic acid, 9a-(acetyloxy)-1a,1b,2,4a,5,7a,7b,8,9,9a-decahydro-4a,7b-dihydroxy-3-(hydroxymeth	2.71
21	3-HEXADECYLOXYCARBONYL-5-(2-HYDROXYETHYL)-4-METHYLIMIDAZOLIUM ION	1.18
22	Lactaropallidin	2.01
23	2-CYCLOHEXEN-1-ONE, 4-HYDROXY-3,5,5-TRIMETHYL-4-(3-OXO-1-BUTENYL)-	2.21
24	Hexadecanoic acid (CAS) Palmitic acid	3.70
25	1-Heptanol, 2-propyl- (CAS) 2-Propyl-1-heptanol	0.83
26	9-Octadecenoic acid, methyl ester, (E)- (CAS) Methyl elaidate	1.27
27	Octadecane (CAS) n-Octadecane	0.99
28	Lanost-7-en-3-one, (9.beta.,13.alpha.,14.beta.,17.alpha.)- (CAS) 9.BETA.-EUPH-7-EN-3-ONE	0.78
29	Oxacyclotetradecane-2,11-dione, 13-methyl- (CAS) .GAMMA.-TRIDECANOLACTONE, 13-METHYL	0.29
30	Stigmast-5-en-3-ol, (3.beta.)- (CAS) 24.BETA.-ETHYL-5.DELTA.-CHOLESTEN-3.BETA.-OL	0.80
		100.00

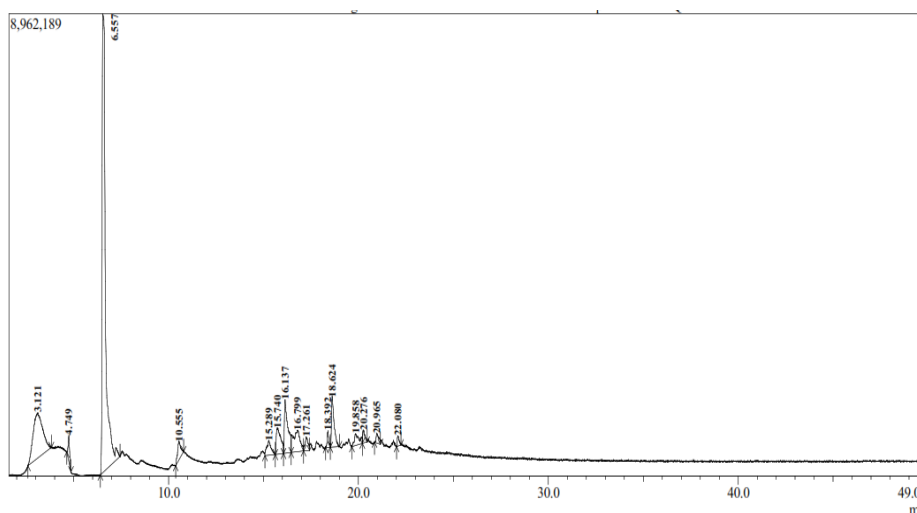


Fig. 2. Py-GC/MS trace of hot water-soluble extractive (Method B). Pyrolysis at 400 °C, for 60 minutes. Peak numbers and names refer to Table II.

TABLE II. IDENTIFIED MASS PEAKS IN PYROLYSIS OF HOT-WATER SOLUBLE EXTRACTIVE (METHOD B)

Peak	Product	Concentration (%)
1	Nitrogen oxide (N ₂ O) (CAS) Nitrous oxide	14.98
2	Methane, tetranitro-(CAS) Tetranitromethane	1.24
3	Carbamic acid, monoammonium salt (CAS) Ammonium carbamate	52.81
4	2-Propanone, 1-hydroxy- (CAS) Acetol	2.01
5	4-CYCLOOCTEN-1-ONE	2.10
6	Phenol (CAS) Izal	4.06
7	Phenol, 2-methoxy- (CAS) Guaiacol	6.48
8	Hexanal (CAS) n-Hexanal	5.11
9	5-METHYL-2-ISOPROPYL-2-HEXENAL	1.42
10	Phenol, 2-methyl-5-(1-methylethyl)- (CAS) Carvacrol	0.96
11	Phenol, 2,6-dimethoxy- (CAS) 2,6-Dimethoxyphenol	4.46
12	1-(3-methoxy-2-pyrazinyl)-2-methyl-1-propanol	1.86
13	2H-PYRAN-2-ON, 5,6-DIHYDRO-4-(2,3-DIMETHYL-2-BUTEN-4-YL)-	0.94
14	Dodecane, 1,1'-oxybis- (CAS) DIDODECANE ETHER	0.92
15	2-CYCLOHEXEN-1-ONE, 4-HYDROXY-3,5,5-TRIMETHYL-4-(3-OXO-1-BUTENYL)-	0.64
		100.00

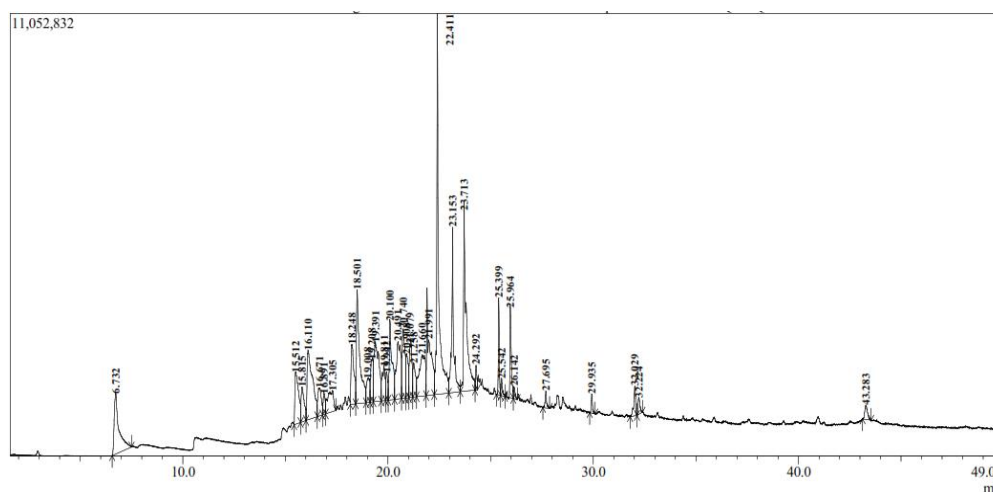


Fig. 3. Py-GC/MS trace of Alcohol-benzene soluble extractive (Method C). Pyrolysis at 400 °C, for 60 minutes. Peak numbers and names refer to Table III

TABLE III. IDENTIFIED MASS PEAKS IN PYROLYSIS OF ALCOHOL-BENZENE SOLUBLE EXTRACTIVE (METHOD C)

Peak	Product	Concentration (%)
1	Cyclopropane, 1,1-dibromo-2-chloro-2-fluoro- (CAS) 1,1-DIBROMO-2-CHLORO-2-FLUOROCYCLOPROP	4.81
2	Benzenesulfonic acid, 4-hydroxy- (CAS) Benzenesulfonic acid, p-hydroxy-	3.15
3	5H-1,4-Dioxepin, 2,3-dihydro-2,5-dimethyl- (CAS) 2,5-DIMETHYL-2,3-DIHYDRO-5H-1,4-DIOXEPIN	1.75
4	Phenol, 2-methoxy- (CAS) Guaiacol	6.21
5	Octanal (CAS) n-Octanal	1.56
6	Undecane, 2,4-dimethyl- (CAS) 2,4-Dimethylundecane	0.71
7	1-Decanol (CAS) Decyl alcohol	2.13
8	Ethanone, 1-(2-hydroxy-5-methylphenyl)- (CAS) 1-Hydroxy-2-acetyl-4-methylbenzene	2.96
9	Phenol, 2,6-dimethoxy- (CAS) 2,6-Dimethoxyphenol	6.19
10	Oxirane, [(tetradecyloxy)methyl]- (CAS) Myristyl glycidyl ether	1.28
11	Propanal, 3-ethoxy- (CAS) 3-Ethoxypropionaldehyde	1.86
12	Propanal, 3-ethoxy- (CAS) 3-Ethoxypropionaldehyde	4.80
13	Dodecanoic acid (CAS) Lauric acid	1.57
14	Benzoic acid, 4-hydroxy-3-methoxy-, methyl ester (CAS) Methyl 3-methoxy-4-hydroxybenzoate	0.91
15	methyl ortho-methoxybenzyl acetate	3.77
16	Nonanoic acid (CAS) Nonoic acid	4.22

17	Phenol, 3,4,5-trimethoxy- (CAS) Antiarol	3.15
18	ZINGERONE [4-(4-HYDROXY-3-METHOXYPHENYL)-2-BUTANONE	1.86
19	Phenol, 2,6-dimethoxy-4-(2-propenyl)- (CAS) 4-Allyl-2,6-dimethoxyphenol	2.36
20	Benzaldehyde, 4-hydroxy-3,5-dimethoxy- (CAS) Syringaldehyde	1.40
21	Ethanone, 1-(4-hydroxy-3,5-dimethoxyphenyl)- (CAS) Acetosyringone	4.18
22	2-CYCLOHEXEN-1-ONE, 4-HYDROXY-3,5,5-TRIMETHYL-4-(3-OXO-1-BUTENYL)-	5.26
23	Hexadecanoic acid (CAS) Palmitic acid	12.25
24	9,12-Octadecadienyl chloride, (Z,Z)-	5.45
25	9,12-Octadecadienoic acid (Z,Z)- (CAS) Linoleic acid	8.40
26	Hexadecanamide (CAS) Amide 16	0.54
27	Cyclohexane, decyl- (CAS) n-Decylcyclohexane	1.62
28	Cyclohexane, decyl- (CAS) n-Decylcyclohexane	0.37
29	9-Octadecenamide, (Z)- (CAS) OLEOAMIDE	2.24
30	9-Octadecenamide, (Z)- (CAS) OLEOAMIDE	0.27
31	1,2-Benzenedicarboxylic acid, dioctyl ester (CAS) Dioctyl phthalate	0.39
32	Tetracosanoic acid, methyl ester (CAS) Methyl lignocerate	0.39
33	2,6,10,14,18,22-Tetracosahexaene, 2,6,10,15,19,23-hexamethyl- (CAS) Squalene	0.85
34	9-Octadecenamide, (Z)- (CAS) OLEOAMIDE	0.51
35	Stigmast-5-en-3-ol, (3.beta.,24S)- (CAS) Clionasterol	0.63
		100.00

B. Discussions

The results of the GCMS analysis of palmyra stem extracts showed that the extractive constituent compounds were mostly different according to the type of solvent used. This was due to the dependence of dissolved extractive constituent compounds on the nature of the solvent. Cold water and hot water are polar solvents but their dissolved extractives were different. There were more cold-water extractive constituents compared to that of the hot-water soluble. The existence of high temperature treatment during hot water extraction was supposed to affect the amount of soluble extractive constituent compounds. An increase in water temperature up to 100 °C could cause volatile extractives to evaporate during the hot water extraction process. This phenomenon was different from the ethanol-benzene extraction process, which also used the temperature treatment. The increase of temperature during the ethanol-benzene extraction did not reach 100 °C because the boiling point of ethanol-benzene (78-80 °C) is lower than that of water. This was indicated by the presence of volatile compounds in ethanol-benzene soluble extractives such as Cyclopropane, 1,1-dibromo-2-chloro-2-fluoro-; Undecane, 2,4-dimethyl-; 2-Cyclohexen-1-One, 4-Hydroxy-3,5,5-Trimethyl-4-(3-Oxo-1-Butenyl)- and n-Decylcyclohexane. The lower boiling point of ethanol-benzene had no effect on the volatile ethanol-benzene soluble extractive compounds; therefore, these compounds remained in the extractives.

According to Roffael (2015), some types of extractive constituent compounds are volatile compounds. The extractive constituents of palmyra stems were different from the extractive constituents of other monocotyledon species such as sago. The number of types of compounds in the sago stem extractives was more than that in the palmyra stems extractives (Siruru *et al.*, 2019). Variations in the tree age, season and location and between trees in one stand had non-uniform extractives (Routa *et al.*, 2017).

Not all of the compounds detected by GCMS analysis were bioactive compounds. Compounds that are classified as bioactive compounds can be in the form of polyphenolic compounds, alkaloids, terpenes, and saponins (Zhao *et al.* 2015). The non-bioactive compounds detected in those three methods were Phenol, 2-methoxy- (CAS) Guaiacol; and 2,6-dimethoxy- (CAS) 2,6-Dimethoxyphenol. Both of these compounds were indication of lignin derivatives (Kuroda *et al.*, 2001; Ház *et al.*, 2013). In addition, the two compounds also contain lignin derivative compounds such as Phenol compounds, 3,4,5-trimethoxy- (CAS) Antiarol; Phenol, 2,6-dimethoxy-4-(2-propenyl)- (CAS) 4-Allyl-2,6-dimethoxyphenol; Benzaldehyde, 4-hydroxy-3,5-dimethoxy- (CAS) Syringaldehyde; Phenol (CAS) Izal; Phenol, 4-(3-hydroxy-1-propenyl)-2-methoxy- (CAS) Coniferyl alcohol; and Phenol 2-methyl-5-(1-methylethyl)- Carvacrol. The presence of lignin derivatives in extractives was due to the mechanical treatment of the biomass or palm stems. The mechanical process of sample powder caused the lignin bonds with polysaccharides to break so that the lignin dissolved during the extraction process. This was in accordance with the mechanical pulping method where lignin elimination was carried out by the mechanical pretreatment (Fengel and Wegner, 1995).

Based on the results of the GCMS analysis, it showed several toxic compounds. The toxic compounds in hot water-soluble extractives were Phenol (4.06%) and Methane, tetranitro- (CAS) Tetranitromethane (1.24%). In cold-water soluble extractives, the toxic compounds were Phenol (6.21%), Methane, tetranitro- (CAS) Tetranitromethane (3.19%); and Guanosine (CAS) Guo (1.80%) (The Metabolomics Innovation Centre; Pubcem). The concentration of toxic compounds in palmyra stem biomass was generally less than 5%. This was considered as the cause why those compounds did not significantly affect the resistance toward wood destroying agents. Types of biomass or wood that are durable generally have large bioactive content, i.e. > 50% (Chang *et al.*, 2000; Fendi and Kurniaty, 2016). In addition, regarding the resistance to wood destroying agents, the effectiveness of toxic compounds in

palmyra stem biomass was not available. The natural resistance of wood to destructive organisms varied depending on the type of wood extractive substance and the destructive organisms that attack it (Fernando et al., 2020).

IV. CONCLUSION

Three different methods used in GCMS analysis resulted in three types of extractives that mostly had different compounds. There were only two similar compounds found but with different concentrations.

There were several compounds with low concentrations, which are toxic. The three types of extractives had a medium concentration of two compounds that are similar to that of lignin compounds. Each types of extractive from palmyra stem contained specific organic compounds. Therefore, in order to produce extractive-free palm stems, multilevel extractions were used, and to remove a certain compound, it should be extracted with an appropriate solvent.

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