

The Role of Friedelin to Predict the Genesis of Muara Wahau Coal, East Kalimantan, Indonesia

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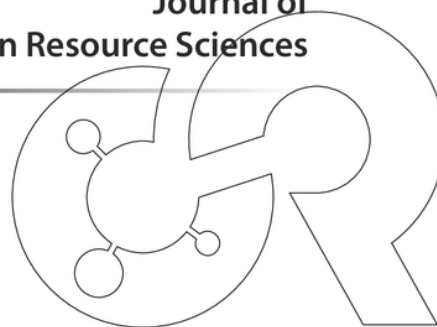
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The Role of Friedelin to Predict the Genesis of Muara Wahau Coal, East Kalimantan, Indonesia**Basuki Rahmad^{*1}, Sudarto Notosiswoyo^{*2}, Komang Anggayana^{*2},
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Muara Wahau's coal is the source of origin the organic element as a higher plants (angiosperm) within terrestrial area. It was characterized by long-chain *n*-Alkane carbon from C₁₄ - C₄₀. The objectives of the research is to predict the genesis of Muara Wahau's coal based on the role of friedelin. The Muara wahau's coal have been analyzed by coal petrographic and Gas Chromatography-Mass Spectrometry (GC-MS). Friedelin are dominating all of the coal samples which have value of 977 µg/g TOC. It was formed by bark of angiosperm and associated with suberinite maceral. The present of friedelin indicated the early transformation during the first stage of diagenesis which the Muara Wahau's coal also indicated the immature condition. The degradation of oxidative process on organic matter in tropical climate will produce friedelin.

1. Introduction

The region of Muara Wahau located in the Upper Kutai Basin of East Kalimantan, Indonesia. The coal samples have vitrinite reflectance values ranging between 0.44 to 0.46% which placing the coal into lignite rank. The coal type of Muara Wahau's coal is raised bog (high moor) which indicates the wet conditions (limnic). Limnic conditions with the high moor type is very for tropical peat typical, particularly in Muara Wahau's coal, East Kalimantan¹⁾. Muara Wahau's coal is the source of origin the organic elements as a higher plants within terrestrial area which characterized by long-chain *n*-alkane carbon from C₁₄ to C₄₀²⁾. It was consist of two series long chain *n*-alkane, that were : the series-1 has a carbon numbers of C₂₁ to C₃₅. The odd carbon numbers predominantly carbon exceeded even at the height of C₃₁ odd carbon, while the series-2 is the carbon number range C₃₆ to C₄₀ carbon numbers predominantly even-carbon peak exceeded even-odd carbon C₃₈.

Friedelin is a substance that derived from a "cork"³⁾. Friedelin is a compound containing a ketone³⁾. Friedelin including pentacyclic triterpenes³⁾, with a molecular formula C₃₀H₅₀O the orthorhombic crystal form³⁾. Friedelin more shows in the bark (maceral suberinite associated) and the leaves of angiosperm³⁾, especially bark/bark of the cork/corks oaks⁴⁾. Friedelin has been found in many barks of angiosperm³⁾. However, these compounds is also detected in the lichens, algae, mosses, peat, coal and mineral wax³⁾.

Friedelin have identified in the fluvial sediments of the Mahakam River, East Kalimantan, Indonesia and it was detected in the bark of *Ficus elastic*⁶⁾. Friedelin has been detected in all coal samples implies that the precursor

of coal in the Kutai Basin are occurred from angiosperm and has a long-chain *n*-alkane from C₃₄ to C₄₀⁶⁾.

The organic geochemistry of the low rank coal from Kutai Basin (Tanito Harum, East Kalimantan, Indonesia) are dominated by sesquiterpenoid derivatives picene was indicating the origin of angiosperm plants⁷⁾.

The objective of research is to verify how to predict the genesis of Muara Wahau coal based on the role of friedelin as the implications of the early formation of long chain *n*-alkane Muara Wahau coal in the Kutai Basin.

2. Geological Setting

Muara Wahau located in East Kutai regency, East Kalimantan Province (Fig. 1). In regional geology Muara Wahau is part of the Upper Kutai Basin⁸⁾ (Fig. 2), economically one of the sedimentary basins in Indonesia, most important, but rich in oil and gas, this region is also rich in coal deposits. Coal bearing strata of the Muara Wahau coal is Wahau Formation, which was deposited during Early Miocene in fluvial-deltaic environment⁹⁾ in which the deposition during a phase of regression along with the uplift process (Syn-orogenic Regressive Phase Deposition¹⁰⁾ process) (Fig. 3).

This formation was intruded by Sintang intrusion composed of andesite and diorite. Based on K-Ar radiometric dating. Sintang intrusion is 16-21 million years in age (Early Miocene)⁹⁾. Wahau Formation lithology in the study area consists of black claystone containing carbonaceous, claystone tuffaceous, fine sandstones, sandstones and interbedded thick coal¹¹⁾ (Fig. 4).

The pattern of distribution of the geological structure of coal (coal cropline) in the study area is trending north-

west of syncline-southeast and in general lithology of Muara Wahau is claystone (Fig. 5).

Commonly position of the main seam of coal seam and the seam-1-2 is a northwest-southeast with the dipping of the coal seam ranges from 8° to 12°. In general, the Muara Wahau coal thickness is in the range 8 to 66 meters.

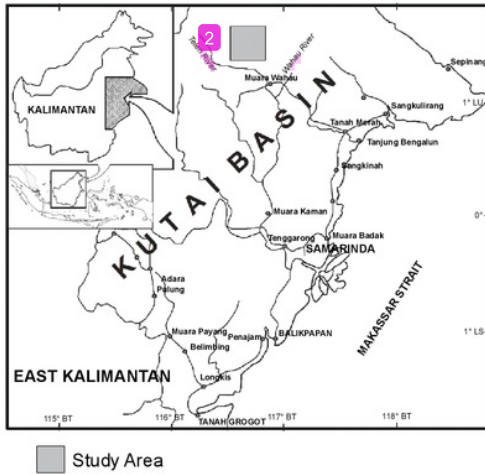


Fig. 1 Location map of Muara Wahau coal field in Kutai Basin, East Kalimantan.

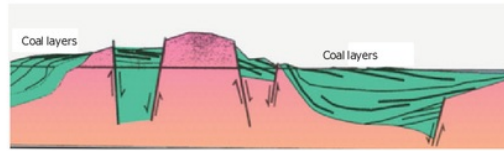


Fig. 3 Syn-orogenic Regressive Phase Coal Deposition¹⁰⁾.

PERIOD	AGE	SURFACE DEPOSITS	VULCANIC ROCK	FORMATION	DESCRIPTION
QUARTER	HOLOCENE	M M M M			Alluvium: pebbles, cobble, sand, mud and plant remains
	PLEISTOCENE				Merang Volcanic: andesite, basalt, lava, tuff, agglomerate breccia and lahars
TERTIARY	MIOCENE	LATE			Notung intrusion: stock & dyke of andesite and diorite
		MIDDLE			Upper Wahau Formation: intercalation of tuff, claystone, quartz sandstone, clayey sandstone, sandy claystone and lignite
		EARLY			Lower Wahau Formation: interbedded coal and algae limestone
EOCENE	LATE			Marah Formation: intercalation of marl, claystone, conglomerate and limestone	
	EARLY				
PALEOCENE					Metaphorphic rock and ultra basic rock

Fig. 4 Regional Stratigraphy of Muara Wahau area East Kalimantan¹¹⁾.

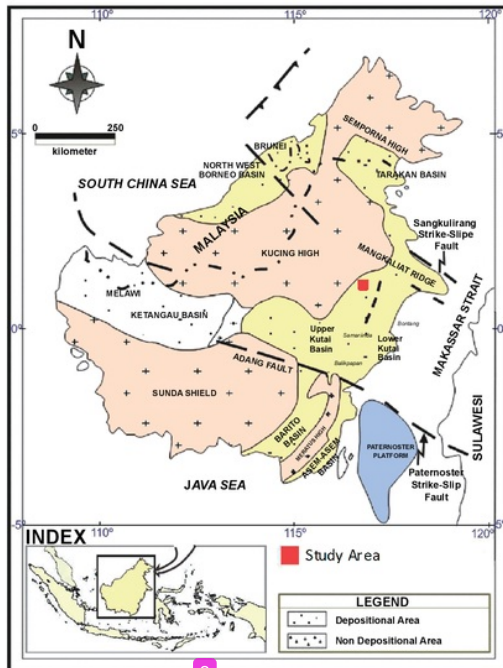


Fig. 2 Location of Muara Wahau coal field in Upper Kutai Basin, East Kalimantan⁹⁾.

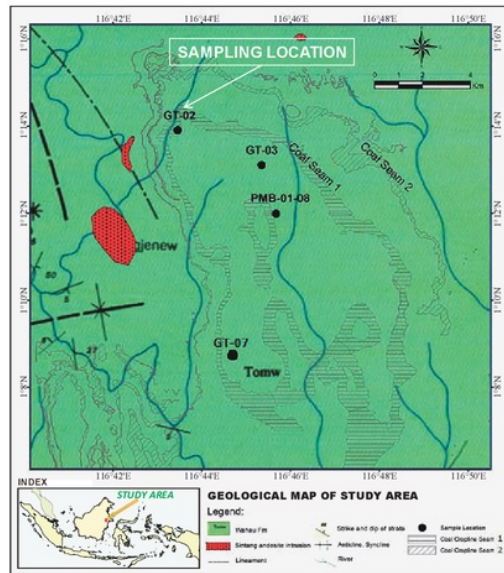


Fig. 5 Geological map of Muara Wahau coal field in Upper Kutai Basin, East Kalimantan.

3. Sampling and Methods

Muara Wahau coal samples were collected from coal seams 1 from one drill cores GT-02 (Fig. 5) and sampling was conducted ply-by-ply based on lithotypes along the coal cores. The samples were wrapped in aluminium foil, stored in plastic bags and transported to the laboratory within 60 days. Methods to coal analyses are consist of coal macerals analysis (15 samples) and organic geochemistry analysis with Gas Chromatography and Mass Spectrometry (GC-MS) method (4 samples); (Fig. 6).

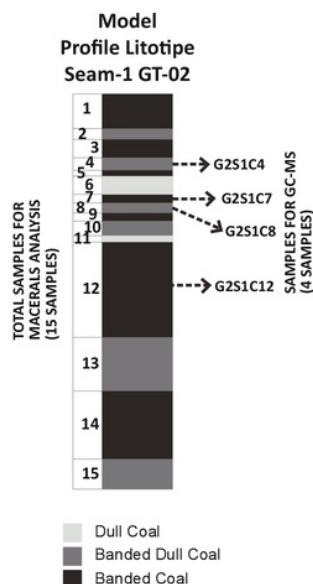


Fig. 6 Profile of lithotype coal seam-1 from 15 samples to macerals analysis (drill hole GT-02).

Coal macerals analysis were to know the presence of macerals composition of coal constituent. The coal samples were then crushed to a maximum size of 1 mm and placed in resin blocks. The sample blocks were polished with a specified polisher. Microscopic investigation was carried out with a Carl-Zeiss Axio Imager. A2m reflected-light microscope. During maceral analysis, 500 points with a minimum distance of 0.2 mm between each point were counted from the polished sections. The maceral composition is stated as percent (% volume). Maceral classification used in this study refers to Australian Standarts AS2856 (1986).

“Soxhlet” extraction is the first stage for organic geochemistry analysis, starting with coal crushed and sieved to obtain only particle size < 0.2 mm and this fraction about 3.25 grams was extracted for 24 hours in a soxhlet extraction apparatus using 200 ml dichloromethane (DCM) as solvent. The total hydrocarbon extract obtain from the soxhlet extraction was separated into different fraction by means of column

chromatography. The combined solvent extracts were filtered and concentrated by use of rotary evaporator and the under blow-down with dry nitrogen gas. Aliquots of the total extract were converted to trimethylsilyl derivatives by reaction with N,O-bis-(trimethylsilyl) trifluoroacetamide (BSTFA) and pyridine for 3 h at 70 °C. An aliquot of the total extract of coal sediment was also separated by use of flash column chromatography on silica into saturated hydrocarbon, aromatic hydrocarbons, and polar compounds to identify compounds with low concentration. The fraction were eluted with hexane, dichloromethane, and methanol, respectively, and dried under nitrogen.

Gas Chromatography - Mass Spectrometry (GC-MS) analyses of derivatives total extracts and separated fraction were performed using Thermo Scientific Ultra series gas chromatograph coupled to a Thermo Scientific DSQ II mass spectrometer. Separation was achieved on a fused silica capillary column coated with Thermo Scientific TR-5MS (30m × 0.25 mm ID × 0.25 µm film thickness). The GC operating conditions were as follows: temperature hold at 65 °C for for 2 min, increase from 65 to 300 °C at a rate of 6 °C min⁻¹ with final isothermal hold at 300 °C for 20 min. Slight differences of retention time are due to clipping of the column between samples. Helium was used as carrier gas. The sample was injected splitless with the injector temperature at 300 °C. The mass spectrometer was operated in the electron impact mode (EI) at 70 eV ionization energy and scanned from 50 to 650 dalton. Data were acquired and processed with the Xcalibur software. Individual compound were identified by co-injection of authentic standarts, comparison of mass spectra with literature and library data, and interpretation of mass spectrometric fragmentation patterns. Relative abundance were calculated from peak area in the total ion current (TIC) of the derivatized total extract and normalized to major peak as 100. Abundances of co-eluting compounds were calculated using the peak areas of characteristic fragment ions *m/z* in the ion chromatogram.

4. Result

Results of total extracts from GC-MS using Total Ion Current (TIC) of the obtained results of the identification of organic compounds (biomarkers) of 4 samples of Muara Wahau coal are Cadalene, TMS-Ester and Pentacyclic Triterpenoids derivatives. Concentrations of friedelin^{3,4,6} of 4 samples are 977 µg/g (G2S1C4); 234 µg/g TOC (G2SC7); 620 µg/g TOC (G2S1C8) and 861µg/g TOC (G2S1C12); (Figs. 7a; 7b; 7c; 7d; Tables 1a; 1b; 1c; 1d). Whereas result of suberinite maceral composition from 4 samples are 2.4 % Vol. (G2S1C4); 1.0% Vol. (G2S1C4); 1.6% Vol. (G2S1C4); 2.0% Vol. (G2S1C4); 12.4% Vol. (G2S1C4); (Fig. 8). Graphic of correlation between friedelin concentration with percent volume of suberinite (Fig. 9) showed a linear relationship (Fig. 10). The mass spectra of friedelin using *m/z* 191 can be see at (Fig. 11).

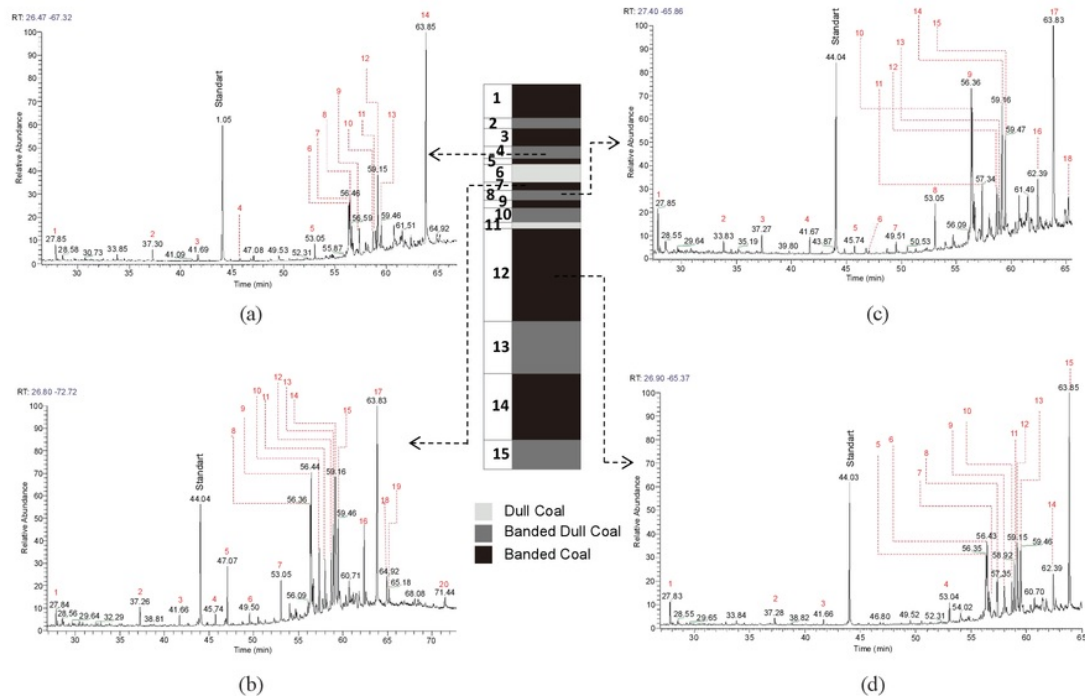


Fig. 7 GC-MS traces of the total extract Muara Wahau's coal: (a) GC-MS sample G2S1C4, (b) GC-MS sample G2S1C7, (c) GC-MS sample G2S1C8, (d) GC-MS sample G2S1C12. For peak annotation see table 1.

5. Discussion

The presence of TMS-ester possibly is methylation derivative of free acids in the sediments that occurred in terrestrial plant waxes⁴⁾. These conditions indicate that the Muara Wahau coal forming still in the early transformation of the during the first stage of diagenesis with the level of maturity is immature.

Friedelin dominating presence in all the coal samples of Muara Wahau with highest concentration (977 $\mu\text{g/g}$ TOC). Decrease of friedelin concentration indicates a over a low input of angiosperm bark material in the peat swamp⁴⁾. Graphic of correlation between friedelin concentration with volume percentage maceral of suberinite showed a linear relationship (Figs. 6 and 7), therefore a more specific biological source of friedelin is mainly from the bark higher plants of the angiosperm⁴⁾.

Friedelin concentration decrease may be caused by two factors, namely climate change and anthropogenic influence is indicated by the variation of biomarkers that will help to reconstruct both quantitative and qualitative changes in vegetation cover⁴⁾.

Therefore, by interpreting changes in the composition of the vegetation cover can be confirmed again that the distribution of biomarker-biomarkers such as: friedelin, β -amyrin (Olean-12-en-3-one) and α -amyrin (Urs-12-en-3-one) are able to demonstrate a more specific

biological sources⁴⁾. Thus a high concentration profile of a biomarker friedelin preliminary instructions for each source of autochthonous or allochthonous Muara Wahau coal, which is then followed by the presence of polar compounds β -amyrin triterpenoids such as Olean-13 (18)-ene; Olean-12-ene; Olean-18-ene, and α -amyrin such as Urs-12-ene.

Des-A-triterpenoids derivative and 3,4-secotriterpenoid acids of oleanane class, ursane, and is a biomarker that describes friedelane of Magnolia and Platanus leaves of Clarkia Formation sediments⁵⁾. The 3,4-secotriterpenoids interpreted as a product of photochemical or microbial degradation of biosynthesized triterpenes and triterpenols like friedelin or amyrin⁵⁾ which caused by oxidative degradation of organic matter in sediments Clarkia Formation, in the United States.

Steroid hydrocarbons not occurred in the coal samples Muara Wahau, this is consistent with previously been observed of some Tertiary coal in Indonesia, especially Kalimantan and Sumatra coal⁹⁾.

The presence of TMS-ester possibly is methylation derivative of free acids in the sediments that occurred in terrestrial plant waxes⁴⁾. These conditions indicate that the Muara Wahau coal forming still in the early transformation of the during the first stage of diagenesis with the level of maturity is immature.

Table 1 Compounds identified in the Muara Wahau coal sample

(a) G2SIC4					
Peaks	Ret. Time	Compound (4 TE)	Base Peak	M. W.	Concent. $\mu\text{g}/\text{g}$ TOC
1	27.85	Cadalene	183	198	81.69
2	37.3	Hexadecanoic Acid TMS Ester	73	313	59.41
3	41.69	Tetracosanoic Acid TMS Ester	73	440	32.75
4	45.74	Eicosanoic TMS Ester	73	384	8.08
5	53.05	Tetracosanoic Acid TMS Ester	73	440	82.9
6	56.35	TMS Ester of Tetracosanoic Ester	73	440	115.25
7	56.44	Olean-13(18)-ene	218	410	111.75
8	56.59	Olean-12-ene	218	410	110.52
9	57.34	Olean-18-ene	218	410	114.7
10	58.69	2,2,4a,9-Tetramethyl-1,2,3,4,4a,5,6,14b-Octahydronicene	218	342	152.31
11	58.93	1,2,4a,9-Tetramethyl-1,2,3,4,4a,5,6,14b-Octahydronicene	257	342	129.19
12	59.15	Urs-12-ene	218	410	332.83
13	59.46	Octadecanoic Acid TMS Ester	73	356	116.08
14	63.85	Friedelin	69	426	977.5

(b) G2SIC7					
Peaks	Ret. Time	Compound (7 TE)	Base Peak	M. W.	Concent. $\mu\text{g}/\text{g}$ TOC
1	27.84	Cadalene	183	198	16.28
2	37.26	Hexadecanoic Acid TMS Ester	73	313	18.47
3	41.66	Tetracosanoic Acid TMS Ester	73	440	10.46
4	45.74	Eicosanoic TMS Ester	73	384	14.55
5	47.07	Des-A-Arborane	95	330	75.91
6	49.5	Docosanoic Acid TMS Ester	73	412	17.27
7	53.04	Tetracosanoic Acid TMS Ester	73	440	49.02
8	56.36	TMS Ester of Tetracosanoic Ester	73	440	115.07
9	56.44	Olean-13(18)-ene	218	410	165.27
10	57.36	Olean-18-ene	218	410	106.01
11	58	5-Oxy-4-Cholestene-3-one	73	440	55.31
12	58.7	2,2,4a,9-Tetramethyl-1,2,3,4,4a,5,6,14b-Octahydronicene	218	342	97.94
13	58.93	1,2,4a,9-Tetramethyl-1,2,3,4,4a,5,6,14b-Octahydronicene	257	342	95.21
14	59.16	Urs-12-ene	218	410	161.61
15	59.46	Octadecanoic Acid TMS Ester	73	356	91.85
16	62.41	Octadecanoic Acid TMS Ester	73	356	113.08
17	63.83	Friedelin	69	426	234.53
18	64.92	9,12-Octadecadienoic Acid (Z,Z)-2,3-BIS(9-TMS)Oxypropyl Ester	73	498	40.7
19	65.2	9,12-Octadecadienoic Acid (Z,Z)-2,3-BIS(9-TMS)Oxypropyl Ester	73	498	18.78
20	71.44	9,12-Octadecadienoic Acid (Z,Z)-2,3-BIS(9-TMS)Oxypropyl Ester	73	498	8.87

(c) G2SIC8					
Peaks	Ret. Time	Compound (8 TE)	Base Peak	M. W.	Concent. $\mu\text{g}/\text{g}$ TOC
1	27.85	Cadalene	183	198	88.06
2	33.83	Octadecanoic Acid Methyl (TMS) Ester	73	400	26.92
3	37.27	Hexadecanoic Acid TMS Ester	73	313	34.91
4	41.67	Tetracosanoic Acid TMS Ester	73	440	34.06
5	45.74	Eicosanoic TMS Ester	73	384	18.19
6	47.07	Des-A-Arborane	95	330	10.29
7	49.5	Docosanoic Acid TMS Ester	73	412	33.09
8	53.05	Tetracosanoic Acid TMS Ester	73	440	130.91
9	56.36	TMS Ester of Tetracosanoic Ester	73	440	338.99
10	56.44	Olean-13(18)-ene	218	410	319.7
11	57.34	5-in-18-ene	218	410	170.43
12	58.68	2,2,4a,9-Tetramethyl-1,2,3,4,4a,5,6,14b-Octahydronicene	218	342	178.62
13	58.91	1,2,4a,9-Tetramethyl-1,2,3,4,4a,5,6,14b-Octahydronicene	257	342	151.69
14	59.14	Urs-12-ene	218	410	345.95
15	59.47	Octadecanoic Acid TMS Ester	73	356	256.23
16	62.39	Octadecanoic Acid TMS Ester	73	356	146.36
17	63.83	Friedelin	69	426	620.06
18	65.19	9,12-Octadecadienoic Acid (Z,Z)-2,3-BIS(9-TMS)Oxypropyl Ester	73	498	89.18

(d) G2SIC12					
Peaks	Ret. Time	Compound (12 TE)	Base Peak	M. W.	Concent. $\mu\text{g}/\text{g}$ TOC
1	27.83	Cadalene	183	198	67.81
2	37.26	Hexadecanoic Acid TMS Ester	73	313	19.98
3	41.66	Tetracosanoic Acid TMS Ester	73	440	16.4
4	53.04	Tetracosanoic Acid TMS Ester	73	440	59.01
5	56.37	Tetracosanoic Acid TMS Ester	73	440	183.31
6	56.43	Olean-13(18)-ene	218	410	238.22
7	56.58	Olean-18-ene	218	410	48.44
8	57.35	Olean-18-ene	218	410	142.5
9	58	5-acosanoic Acid TMS Ester	73	440	105.98
10	58.69	2,2,4a,9-Tetramethyl-1,2,3,4,4a,5,6,14b-Octahydronicene	218	342	132.91
11	58.92	1,2,4a,9-Tetramethyl-1,2,3,4,4a,5,6,14b-Octahydronicene	257	342	176.42
12	59.15	Urs-12-ene	218	410	270.44
13	59.46	Octadecanoic Acid TMS Ester	73	356	259.76
14	62.39	Octadecanoic Acid TMS Ester	73	356	104.75
15	63.85	Friedelin	69	426	861.27

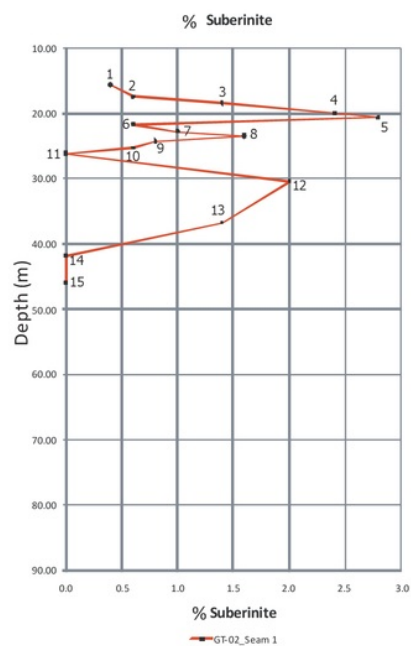


Fig. 8 Vertical variation of maceral suberinite from coal seam-1 (15 samples) from drill hole GT-02.

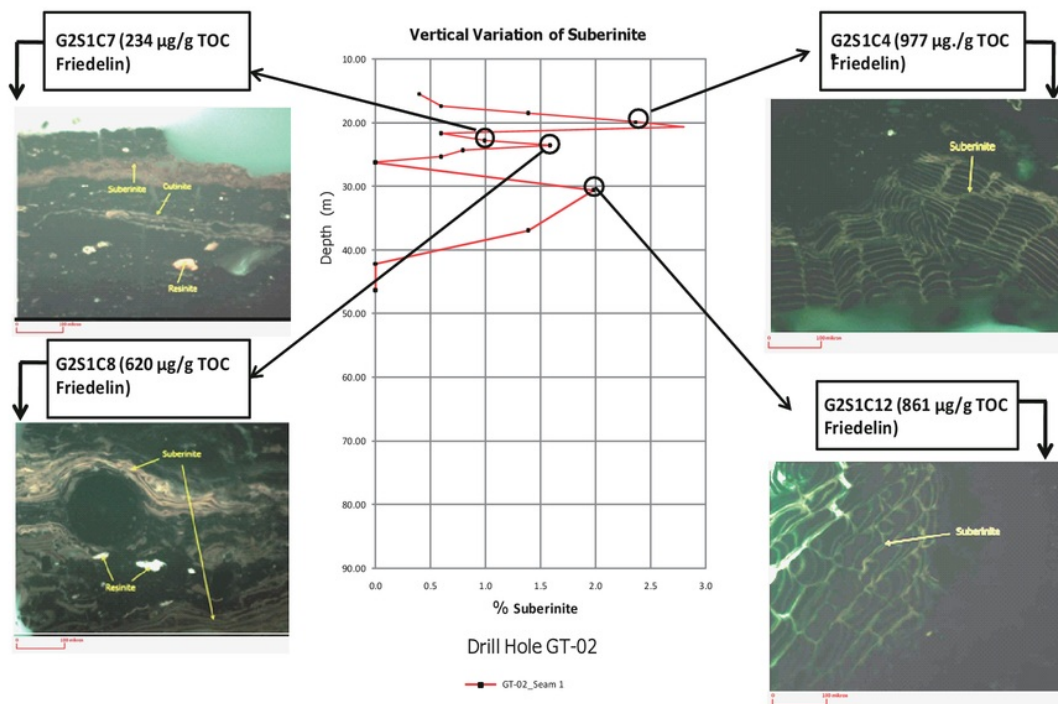


Fig. 9 Correlation friedelin and vertical variation of suberinite from 4 samples coal seam-1 drill hole GT-02 (samples number: G2S1C4; G2S1C7, G2S1C8 and G2S1C12).

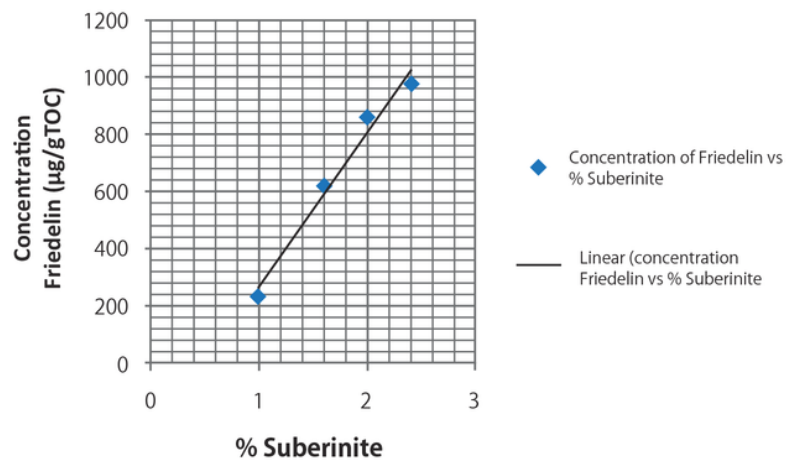


Fig. 10 Graphic which showed the linear relationship between friedelin to suberinite from 4 samples coal seam-1 drill hole GT-02 (samples number: G2SIC4; G2SIC7, G2SIC8 and G2SIC12).

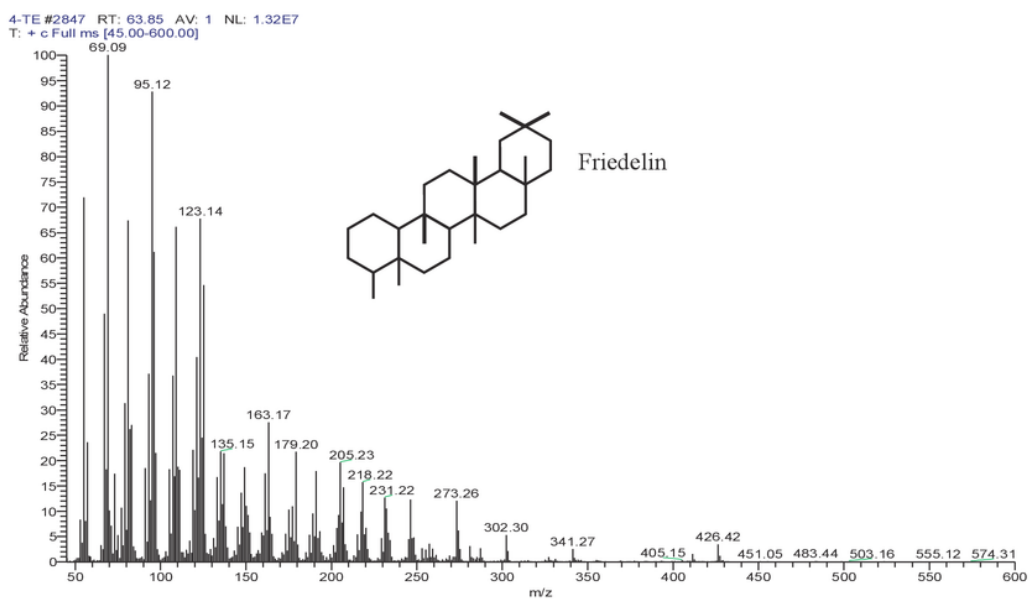


Fig. 11 Mass spectra and molecule structure of friedelin^{1,3,11} using m/z 191.

6. Conclusions

The present of friedelin^{3,4,6} are dominating in the Muara Wahau coal with the highest concentration (977 µg/g TOC). The present of friedelin also indicates the generated of Muara Wahau coal still in the early transformation during the first stage of diagenesis.

Source of friedelin is from the bark of higher plants (angiosperm). It is indicate that the consistency between the linear concentration of friedelin with the amount percent volume of suberinite maceral. The decrease of friedelin that follow the decrease of suberinite, and it was caused of influence higher plants (angiosperm) content.

The tropical climate condition in Indonesia contributed to the process of oxidative degradation of organic matter in the Muara Wahau coal which is a product of photochemical or microbial degradation which will result in friedelin.

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