Applications of Biomarkers to Determine Maturity Level of Coal from Tanjung Formation and Pamaluan Formation, Muser Area, East Kalimantan

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Abstract: The Tertiary coal from Tanjung Formation and Pamaluan Formation exposed in the Muser, East Kalimantan, were organic geochemistry analyzed, and their maturity level were interpreted. This is commonly provide information about the direction of migration, deposition environment, and level of maturity. The level of coal maturity of Tanjung Formation and Pamaluan Formation were distinguished by CPI value of 1, 22 - 3.53; C_{30} moretane/hopane ratio of 0.32 - 0.35; Tricyclic / 17 α -hopane (0.04 -0.12); Drimane /Homodrimane ratio of 0.27 - 2.19; C29aaa20S / C29aaa20R (1.21-0.59); MPI (0.55-0.43), TNR (0.67-0.53). All these biomarker maturity parameter data show that all samples are thermally early mature to mature. The coal of Tanjung formation was more mature than Pamaluan coal Formation.

Keywords: biomarker, maturity, coal, Tanjung Formation, Pamaluan Formation

1. Introduction

Biomarkers are molecular fossils of complex biochemical produced by lipid derived from living things and are very commonly used in determining the depositional environment or maturity level of sedimentary rocks; including coals (Peters and Moldowan, 1993; MacKenzie, A.S., Patience, R.L., Maxwell, J.R., 1981). This is quite important in the area that has entered the stage of further development because it can be a form of confirmation on the concept of the geological structure of coal that has been there before. For a company this could open up possibilities for exploration in a particular lithology and used analogies to learn other neighboring areas.

The aim of this study is to assessment of thermal maturity of coals from Tanjung Formation and Pamaluan Formation based on organic biomarker distributions

2. Geological Setting

The Muser Village is located in the vicinity of the Paser Regency, East Kalimantan Province (Fig.1). Geologically, the coal was situated in Pasir Basin. This basin is one of the Tertiary basin and well known as an area of major resources of oil, gas and coal in western Indonesia. The Pasir Basin is situated along the northwestern margin of the Paternoster Platform Shield in South Kalimantan.

The Pasir basin is defined by the Meratus Ophiolitic Complex to the west and separated from the Kutei Basin to the north by a flexure related to the Adang Fault. The basin has a narrow opening to the south towards the Asem-Asem basin. In the west bordering with Barito Basin. The Barito Basin is an asymmetric basin, forming a foredeep in the eastern part and a platform approaching the Meratus Mountains towards the west (Fig. 2).



Figure 1: Location of research area of Muser Village, Paser Regency, East Kalimantan

The Pasir Basin commenced its development in the Late Cretaceous, following a micro-continental collision between the Paternoster and SW Borneo microcontinents. Early Tertiary extensional deformation occurred as a tectonic consequence of that oblique convergence. In Muse Area, the Late Cretaceous uplifted caused displacement of Haruyan Formation which contains of ultrabasic rocks which were then intruded by granite, granodiorite and diorite; a NE-SW horst-graben structural was developed and became accommodation space for lacustrine sediment of the Tanjung and Pamaluan Formations.



Figure 2: A geological summary map of Pasir Basin (Darman H., 2014)

The Pasir Basin is underlain by Jurassic Cretaceous ultrabasic rocks and the Jurassic Pitap Formation. These are overlain by the Tanjung, Berai, Pamaluan, and covered by Quaternary alluvium. Research indicates that coal is formed mainly in the Tanjung, Pamaluan Formations within the basins. A generalised stratigraphic column is shown in Figure 3.



Figure 3: Geological Map and general stratigraphic column in Liburdinding and surrounding area.

The Tanjung Formation consists of intercalation of conglomerate, shale, sandstone and coals. The depositional environment of Tanjung Formation is lacustrine in the lower part and gradually change to shallow marine in the upper part. The alternation of quartz sandstone intercalation with claystone, shale, limestone and siltstone, well bedded quartz; ae characterized of Pamaluan Formation.

3. Methods

In this study, the coal samples have been taken of Tanjung and Pamaluan formation. Samples taken an outcrop. After the sample is extracted and then rock split into several factions, and it can be analyzed. After getting the data from the method, GC and GC-MS, it is done by the method of comparative analysis and plotting on tables that have been made by previous researchers.

3.1 Extraction Sample

An extraction is the first step process on this geochemical analysis. The extraction process useful for the conversion of coal samples from the solid phase into a liquid phase. **3.2 Liquid Chromatography (LC)**

Liquid chromatography is an analytical technique that is useful for separating ions or molecules dissolved in a solvent. If the sample solution in contact with the liquid phase, the different solutes will interact with another phase to differing degrees due to differences in adsorption, ion exchange, partition or size. These differences allow the mixture components to be separated from each other by the transit time of the solutes through the column.

Liquid Cromatography will be used to separate the extracted coal samples into 4 (four) fractions, namely aromatic, saturate, NSO/Resin and Asphalthenes. The saturate fractions and aromatic fractions will be used for biomarker analysis.

3.3 Gas Chromatography (GC)

Gas chromatography is a method of separating components of a substance based on the time interval (retention time) between the time of intake (injection) is a substance with a time of combustion in a gas chromatograph with an inert medium. Chromatograph equipped with a computer that records the data in the form of a chromatogram is done programmatically, recording the results of the known fingerprint (fingerprint).

Chromatogram is a series of straight lines that intersect at the top to form the peak - a pointed peak with a height and a certain density. The height of a peak indicates the relative amount of a component while the position on the horizontal line is the time interval (in minutes). The distribution pattern of the line - a line of very specific vertical angle between one substance to another due to the sensitivity of the ability of separation between the components of this chromatographs. Comparison (ratio) relative frequency of certain atomic number with the other atoms can explain many things, including the type of composition, the type of organic matter origin, depositional environment, thermal maturity and biodegradation process.

3.4 Gas Cromatography-Mass Spectrometry (GC-MS)

GC-MS is the method of separation of organic compounds using two methods of analysis of compounds which gas chromatography (GC) to quantitatively analyze the amount of compounds and mass spectrometry (MS) to analyze the molecular structure of the analyte compound.

Gas chromatography is a spectroscopic technique that uses the principle of the separation of a mixture based on differences in the speed of migration of its components. Gas chromatography is used to identify a compound contained in the gas mixture and also determine the concentration of a compound in the gas phase. While the mass spectroscopy is a method to obtain the molecular weight by finding the massto-charge ratio of ions that charge is determined by measuring the radius of its circular orbit in a uniform magnetic field. The use of gas chromatography can be combined with mass spectroscopy. Combination of the two can yield more accurate data in the identification of compounds that completed by its molecular structure.

Generally the mass spectrum obtained by converting a compound of a sample into ions that move quickly are separated based on their charge to mass ratio. Mass spectroscopy is capable of producing a beam of ions of a test substance, into a spectrum of sorting the ions corresponding to the mass-to-charge ratio and recording the relative abundance of each ion species that exist. Generally only positive ions are studied because negative ions generated from source generally slight collision.

4. Results

4.1 Extraction and Liquid Chromatography

Extraction is the process of sample preparation and the process is first conducted. The extraction process produces a

	Table 1. Extraction Data Worksheet												
Sample ID	Lithology	Rock	Initial	12020	1000100000	Empty	Vial +	20022	Total				
		Sample	Extract	Aliquot	Multi	Vial	Extract	Extract	Extract	EOM			
		(gr)	(ml)	(ml)	Factor	(gr)	(gr)	(mg)	(mg)	(ppm)			
Tanjung	Coal	3,2988	100	50	2	7,1024	7,2382	135,8	271,6	82333,0			
Pamaluan	Coal	4,1649	100	50	2	7,2005	7,3516	151,1	302,2	72558,8			

Table 1:	Extraction Data	Worksheet

Liquid Chromatography is a separation process saturated fraction, aromatic, asphaltene, and NSO / resin derived from samples that have been through the extraction process. The

results of Liquid Chromatography presented in Table 2.

		Table 2	. Liquid Chro	matograph Wo	rksheet	,		
Sample Depth	Charge (mg)	Empty Vial (gr)	Vial + Fraction (gr)	Wt of Fraction (mg)	Total (mg)	Norr (Total (%)	
		7,2189	7,2355	16,6		Sat	4,24	
Toniund		7,232	7,2955	63,5	391.7	Aro	16,21	100
Tanjung		7,1719	7,1868	14,9	391,7	NSO	3,80	100
		7,1089	7,4056	296,7	1	Aspl	75,75	

Table 2. Liquid Chromatograph Worksheet

Table	3	is	presenting	the	calculation	$C_{15^{+}}$	Extractable
Organi	ic N	Aatt	ter (EOM).				

Sample Depth (feet/metres)	Lithology	ЕОМ	HC	Composition of C ₁₅₊ Extractable Organic Matter (Normalised Percent)				Percent			Sat/Aro
		(ppm)	(ppm)	Sat	Aro	NSO	Asph	EOM/TOC	HC/TOC	HC/EOM	85
Tanjung	Coal	82333	16837	4,24	16,21	3,80	75,75	-	-	20,45	0,26
Pamaluan	Coal	72559	6683	2,17	7,04	18,42	72,37	-	12	9,21	0,31

Based on calculations C_{15} + extractable Organic Matter (EOM) of the samples, it can be seen that both samples have a high percentage of the content of asphaltene, i.e.72-75%. However, for NSO/Resin compounds there is a little difference prominently. Tanjung formation samples show the number of compounds NSO/Resin approximately 3%, is much smaller than Pamaluan formation, amounting to over 18%. In addition, both samples also contain aromatic fairly large, between 7-16%. Meanwhile, the content of saturated compounds in both samples is very small, about 2-4%. This indicates the characteristics of the coal samples which have undergone thermal degradation.

4.2 Gas Chromatography (GC)

N-alkane peaks can be identified through GC analysis of saturated fraction of each sample. In this study, the peak of n-alkanes are divided into three types, namely the n-alkanes short chain consisting of $nC_{13} - nC_{18}$, n-alkane chain being composed of $nC_{19} - nC_{28}$, and n-alkane long chain consisting of $nC_{29} - nC_{35}$, (Figure 4)



sample that is free from impurity content, so that pure samples containing compounds to be used in research. The following table presents the results of the extraction of the sample (Table 1) International Journal of Science and Research (IJSR) ISSN (Online): 2319-7064 Index Copernicus Value (2013): 6.14 | Impact Factor (2014): 5.611



Tanjung Formation (T) and Pamaluan Formation (P)

4.3 Gas Chromatography-Mass Spectrometry (GC-MS)

a) Saturated fraction

The GC-MS analysis of sample fractions of saturated, several biomarkers were identified, i.e., Triterpanes (m/z 191), steranes (m/z 217, 218), and a bicyclic alkanes (m/z 123).

Triterpenes (m/z 191)

Triterpenes identification is identified by MIC (Mass Ionic Carbon) ion m/z 191. The biomarker compounds contained in triterpenes include tricyclic diterpane, Tm, (or C_{27} 17a-trisnorhopane) and Ts (or C_{27} 18a-trisnorhopane), hopanes, moretanes, oleanane, and Gammacerane. The biomarker compounds were dispersed in retention time between 35-96 minutes to the formation of the Tanjung and 24-70 minutes on Pamaluan formation. (Figure 5)



Figure 5: Distribution of triterpane chromatogram (m/z 191) of coal samples from Tanjung Formation (T) and Pamaluan Formation (P)

Based on the picture, it can be determined that the peak (peak) A, B, C, D, E, F, G, and H on the tricyclic diterpane. Peak 1, 2, 4, 6, 8, 9, 11, 12, 13, 14, 15, 16, 17, and 18 is a class of compounds Hopane. Peak 5, 7, and 10 is a class of compounds Moretane. The culmination of oleanane and Gammacerane also be identified from the appearance of his peak. Chromatogram peaks that have been identified are then calculated to determine the value of the comparison between the biomarker compounds (Table 4)

Table	4:	Ratio	of	triterpane	biomark	cer
- aoie	••	1 carro	U 1	multipane	oronnan	

Well/Sample ID (feet/metres)	Tm Ts	Total %	Total %	% C30 Resins	C2901 B Hopane	C29 Moretane	C ₃₀ Moretane	C30 Hopane	22S	/22R	С300. ВНор	GMx100
		C19-C31 Tricy	Hopanes	+ OL	C ₃₀ αβ Hopane	C ₂₉ Hopane	C ₃₀ Hopane	C29+C30 Moretanes	C ₃₁	C32	OL	С300. ВНор
Type of Parameter	a, b	a, c	а	а	b	b	b	b	b	b	а	а
Tanjung	19,55	3,86	95,06	0,51	0,91	0,39	0,32	1,47	0,85	1,53	33,78	3,28
Pamaluan	1,58	10,73	88,14	1,13	2,64	0,15	0,35	1,33	0,18	1,61	10,59	0,00

Sterane (m/z 217)

Biomarker compounds sterane groups can be identified through ion chromatogram MIC at m/z 217. Some of the compounds can be identified on the ion m/z 217 was diacholestane and cholestane. The distribution of these compounds are in a span of 46-56 minutes on the formation of the Tanjung and 46-57 minutes on Pamaluan Formation. However, cyclic alkane and methylated sterane cannot be determined in both samples. (Figure 6)



Figure 6. Sterane Chromatogram (m/z 217) of coal samples. (T: Tanjung Formation; P: Pamaluan Formation)

Based on the above picture, it can be determined that the peak (peak) A, B, C, E, F, are diacholestane. Peak 1, 2, 3, 4, 5, 6, 7, 8, 9, 10, 11, 12, 13, 14, and 15 is a cholestane. The ratio of steranes biomarker were calculated and presented in Table 5.

Biomarker compounds that can be identified from the chromatogram MIC ion m/z 123 (Fig.7) is bicyclic, pentamethyldecahydronaphtalene, drimane, and homodrimane.





Figure 7. The bicyclic alkane chromatogram of coal samples. (T: Tanjung Formation; P: Pamaluan Formation)

Table 5:	Ration	of sterane	biomark	er

Well/Sample ID	Drimane	C15 Rearranged Bicyclics	Drimane	
(feet/metres)	Homodrimane	Drimane	C ₃₀ ab Hopane	
Type of Parameter	a, b	a, b, c	а	
Tanjung	0,27	2,03	0,10	
Pamaluan	2,19	3,21	0,04	

Bicyclic (m/z 123)

Abundance

Table 6: The bicyclic distribution in coals samples

Well/Sample ID (feet/metres)	<u>Total Hopane</u> Total Sterane	C30αβ Hop Total Des C ₂₉	6ааа 20 C ₂₇	OR Stera C ₂₈	anes C ₂₉	C27000020R C29000020R	Total C ₃₀ 4M Total Des C ₂₉	Total C ₃₀ 4M+Des C ₃₀ Total Des C ₂₉	C ₂₉ ααα.20S C ₂₉ ααα.20R	C ₂₉ βββ20R+20S C ₂₉ ααα20S+20R	C ₂₇ βααDaia20S C ₂₇ βααDia20R	C ₂₈ -C ₃₀ 4M Ster C ₂₇ -C ₂₉ Des	Total C ₂₇ -C ₂₉ Dia Total C ₂₇ -C ₂₉ Des
Type of Parameter	a	а	а	а	а	а	а	а	b	b	b	а	a,c
Tanjung	22,37	10,71	30.57	29,32	40,11	0,76	0,00	0,00	1,21	0,63	0,94	0,00	0,18
Pamaluan	8,72	4,40	32,95	21,33	45,72	0,72	0,00	0,00	0,59	0,64	6,38	0,00	0,42

b) Aromatic fraction

Analysis Gas Chromatography-Mass Spectrometry (GC-

MS) was also conducted on samples of aromatic fractions from a sample derived from Tanjung Formation and

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Trimethyl Naphtalene (m/z 170)

Trimethyl naphtalene a biomarker compounds naphthalene group legible on the chromatogram MIC ion m/z 170. Various arrangement trimethyl naphthalene with different branches of the chain can be seen in the chromatogram ion m/z 170. But in reality, not all the peaks of the sample chromatogram which can be identified as a class of compounds Trimethyl naphthalene (Figure 8).





Figure 8: Trimethyl Naphtalene chromatogram (m/z 170) (T : Tanjung Formation; P : pamaluan Formation)

Methyl Phenanthrene (m/z 192)

The biomarkers identified in MIC chromatogram phenanthrene ion m/z 178, next is identification Methyl groups Phenanthrene done on the chromatogram MIC ion m/z 192. A total of four compounds can be found in these ion chromatogram, which is 3-Methyl Phenanthrene, 2-Methyl Phenanthrene, 9-Methyl Phenanthrene, 1-Methyl Phenanthrene (Figure 9).

Furthermore, all biomarkers that can be identified from a sample of aromatic fraction is then inserted into the calculation to determine the abundance ratios of each of these biomarkers. The calculation result is then presented in tabular form. (Table 6).

Well/Sample ID (feet/metres)	DNR-1	DNR-6	TNR-1	TNR-3	TNR-4	MPR-1	MPR-2	MPI-1	MPI-2	Re	Rm		$\frac{C_{28}TA}{C_{29}MA + C_{28}IA} \%$ (% Aromatisation)
Tanjung	0,42	0,38	0,67	5,24	1,90	0,96	0,42	0,55	0,57	0,73	0,57	44,68	
Pamaluan	0,19	0,12	0,53	0,44	1,39	0,77	0,46	0,43	0,59	0,66	0,61	43,87	(4))





Fgure 9: Methyl Phenanthrene chromatogram (m/z 192) (T : Tanjung Formation; P : Pamaluan Formation)

5. Discussion

5.1 Extractable Organic Matter Maturity Parameter

In general, the results of extraction and Liquid Chromatography method, it can be seen that each sample has a very high content of compounds Asphaltene group, aromatic fractions in medium scale, whereas saturated and NSO/Resin very small. These things are the main characteristics seen in coal samples, biodegradable sustained increase levels Asphaltene and NSO (Resin), so that the aromatic compounds and saturated, too differentiated resulting in an abundance of smaller and smaller.

The fourth fraction which has been separated during the process of Liquid Chromatography, then plotted into Extract Composition plot diagram to see the distribution of data in specific zones predetermined (Figure 10)

The ternary diagram of extractable organic matter (EOM) shown all the data of Tanjung and Pamaluan Formation were concentrated on coal immature zone as with the NSO and Asphaltene concentration is very high. Therefore, based on the triangle of EOM, it can be stated that the coal of the Tanjung and Pamaluan Formation in immature stages.



Figure 10. Extract Composition Plot

5.2 n-alkanes and isoprenoids maturity parameter

The n-Alkanes are relatively abundant in the total ion current (TIC) of the saturated fraction. Based on the m/z 71 mass chromatogram of the saturated fraction, n-alkanes were identified in the range C_{17} to C_{35} . The n-alkane patterns of the coal samples are dominated by mid-chain homologues $(n-C_{21}-_{25})$ and long-chain $(n-C_{27}-_{31})$ with a maximum at $n-C_{29}$ and a marked odd over even predominance, indicating a significant contribution of terrestrial plant (Eglinton and Hamilton, 1967). The dominated mid-chain *n*-alkane in Tanjung Formation comparing in Pamaluan Formation; suggested that Tanjung coal formation is more mature than Pamaluan coal Formation.

The isoprenoid biomarkes which are frequently found in the sediments and oils are pristane (Pr) and phytane (Ph) are the most frequently occurring isoprenoids in sediments and oils. The variation of Pr/Ph, $Pr/n-C_{17}$ and $Ph/n-C_{18}$ in Tanjung and

Pamaluan Formation (Table 7) are related with the different source materials and redox potential of sediments. (Didyk et al., 1978; Connan and Cassou, 1980).

Lable 7. Ratio of isoprenoid in coal samples	Table 7: Ratio	of isoprenoid	in coal	samples
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Formation	Pr/Ph	Pr/C17	Ph/C18	nC31/nC19	CPI
Tanjung	8.27	74.49	10.12	3.8	1.22
Pamaluan	3.92	14.26	2.97	21.47	3.52

The ration of Pr/n- C_{17} and Ph/n- C_{18} ratios are considered sensitive to thermal maturity when organic matter types are constant, and decrease with increasing maturation (Connan and Cassou, 1980). However, in our research we found that ratio of Pr/Ph, Pr/n- C_{17} and Ph/n- C_{18} are not found suitable for determined the stage of maturation. The high ratio of Pr/Ph, Pr/n- C_{17} and Ph/n- C_{18} in Tanjung Formation were related the redox condition of sediments.

The values of the CPI (carbon preference index) and OEP 2 (odd–even predominance) of Tanjung Formation is 1.22; while the CPI value of Pamaluan Formation is 3.53. The CPI range between 1.22 - 3.53 which are in accordance with the sub-bituminous to bituminous coal rank. The value Carbon Preference Index (CPI) is strongly influenced by the source material and also of maturity levels (Bray and Evans, 1961). CPI large value (> 3, 0) indicates a low level of maturity and sediment material derived from the terrestrial plant. Due to high CPI value, the Pamaluan coal formations is in the immature stage.

5.3. Saturate Biomarker Maturity Parameter *C*₃₀ moretane / hopane

The ratio of C_{30} -moretanes/hopane can be used to measured thermal maturity of coal sample. Peters et al (2005) mentioned that, the C_{30} - moretane to hopane ratio for the C_{30} compound declines with increasing thermal maturity from \pm 0.8 in immature rocks to < 0.15 for mature rock. According to Peters et al (2005), these samples are in early mature. The moretane/hopane ratios thermal maturity parameter is also strongly influenced by source input, lithology, oxicity, and acidity of depositional environment (Moldowan et al., 1986)

Tricyclic / 17a-hopane

Seifer and Moldowan (1978) found that tricyclics/17 α hopane ration was increases for related oils samples due to increasing maturity level. The ratio of the tricyclics/17 α hopane of the Tanjung is 0.04, while the formation Pamaluan have a greater value is 0.12. This indicate that the Tanjung coal formation has a higher level of maturity than the coals from the Pamaluan Formation.

Drimane / Homodrimane

The measurement of ratio drimane / homodrimane identified from bicyclane (m/z 123) shown that Tanjung Formation is 0.27, while the formation Pamaluan 2.19. This value indicates that the formation of the Tanjung have a higher maturity level than Pamaluan formation.

C29aaa20S / C29aaa20R

This ratio of coal from Tanjung formation is 1.21, whereas for the formation Pamaluan is 0.59. This suggests that the formation of the Tanjung has a higher maturity level than Pamaluan formation.

5.4. Aromatic Biomarker maturity Parameter *MPI-1*

The value MPI-1 (Methyl Penanthrene Index) from both formations is 0.55 to 0.43 for the formation of the Tanjung and Pamaluan formation. The value of Rc (Calculated vitrinite reflectance) for both formations is 0.73 to 0.66 for the formation of the Tanjung and Pamaluan formation. Based on the diagram MPI vs Ro was appears that the formation of the Tanjung has a higher maturity level than Pamaluan formation. (Figure 11).



Figure 11: The crossplot MPI-1 and Ro of coal samples *Trimethylnaphthalenes (TNR-1)*

TNR values may determine the maturity level of a rock. The higher the peak of TNR showed higher levels of maturity. The value of TNR-1 formation Tanjung is 0.67 and 0.53, for the formation Pamaluan, respectively. This suggests that the formation of the Tanjung have a higher maturity level than Pamaluan formation.

6. Conclusion

The biomarker maturity parameter measurements of saturate fractions and aromatics fraction were shows that the Tanjung coal formation more mature than coal of Pamaluan Formation.

A combination of molecular maturation parameters, of the type considered in this study, is inferred to be necessary for a reliable delineation of the stages of organic matter maturation. However, in the selection of biomarker parameters have to be careful because some maturity parameter is also affected by the source organic materials, redox condition and their depositional environments.

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