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Biomarker Composition of Some Oil Seeps from West Sulawesi, Indonesia

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Abstract - A suite of oil seep samples collected from the Lariang and Karama Basins, West Sulawesi, Indonesia, have been analyzed using GC and GCMS techniques. Their biomarker compositions have been used to reveal the likely source rock that produces oil. These oil seep samples contain a range in both the concentration of hydrocarbons present and certain level of biodegradation experienced. All sterane m/z 217 data display a familiar pattern of steranes with carbon numbers from 27 to 29 together with pregnane and homo-pregnanes, including rearranged steranes. Triterpane (m/z 191) data show a moderate content of tricyclic terpanes (Tri/$C_{30}H$), medium to high ratio of norhopane/hopane (C_{29}/C_{30}), and medium to high abundant land plan marker such as resin bicadinanes(Bic/$C_{30}H$) and oleananes (Ol/$C_{30}H$). The presence of high abundant oleananes in these samples has been interpreted as the evidence for a marine influence during early diagenesis that may occur by marine or via re-deposition of peat organics in marine sediments. Hence, these results state that the organic matter facies for their source rock oil seep samples from the West Sulawesi region are detected as mixed between allochthonous macerals from typical terrestrial higher plants transported into the basin from distal swamps and soils and some marine organic matter.

Keywords: biomarker, oleananes, marine sediment, Lariang and Karama Basins, West Sulawesi

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INTRODUCTION

Background

The occurrence of oil and/ or gas seeps in an area may indicate the presence of active petroleum system in its subsurface. Therefore, the oil seeps provide significant data to build an integrated petroleum system analysis. Biomarker geochemistry is one of the best tools that can be used to establish petroleum systems to improve exploration success.

Biomarker composition in oil may be used to predict various geological and geochemical aspects of the oil corresponding source rock, including organo-facies and lithology. For example, the high of rearranged steranes relative to their steranes counterpart can be used to indicate petroleum derived from clay-rich environments for their source rocks (Peters *et al*., 2005). Biomarker parameters provide more detailed information needed to answer questions about the depositional environment of source rock, organo-facies, lithology, source rock age, thermal maturity, migration distance and relative direction, and the biodegradation of oils (Peters *et al*., 2005).

Numerous oil and gas seeps were found in the part of western and southern of Sulawesi, such as in Lariang and Karama Basins (Figure 1).These indicate that there is an active petroleum system in their subsurface, so these areas are feasible for further exploration activities. So far, there has been no discovery well in this area (Satyana *et al*., 2012). Therefore, some existing oil seeps provide important data to build an integrated petroleum system analysis.

The main purpose of this paper is to document the biomarker composition of the oil seep samples from both the Lariang and the Karama Basins. The biomarker composition characterizes the typical specific Southeast Asia crude oil, and infers that these oil seeps were possibly derived from mixed organic matter deposited in a marine deltaic depositional system.

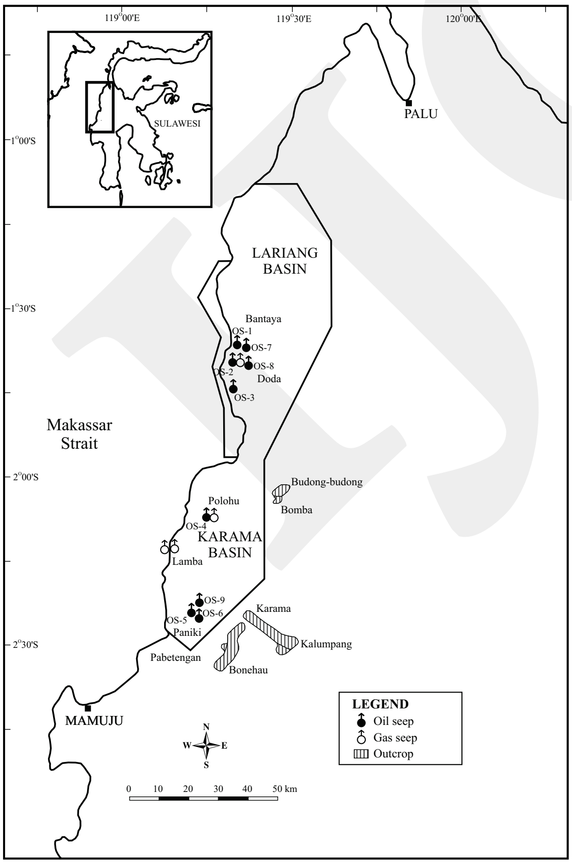

Figure 1. Location map of the study showing samples position.

Geological Settings

Western Sulawesi was separated from Kalimantan during the Cenozoic because of the opening of the Makassar Strait. This region was part of a single area in the Late Mesozoic. Western Sulawesi is believed to be the easternmost edge of the Sunda Craton. It is overlain by a sequence of Tertiary clastics, carbonates, and volcanics affected by complex tectonics as the Australian-Indian, Pacific, and Southeast Asian microplates interacted.

Figure 2 shows the tectonostratigraphy of Western Sulawesi region (Calvert and Hall, 2003), which separated Toraja Group into two formations, *i.e.* thicker marine/terrestrial sedimentary rocks of the Kalumpang Formation and marine sedimentary rocks of the Budong-Budong Formation. The Toraja Group is 3,500 m thick, and it is underlain unconformably by the Mesozoic basement, while the top of this group is assumed to be conformable with Lisu Formation.

The Kalumpang Formation is a sequence of shales, coal beds, and quartzose sandstones aged from Middle to Late Eocene, from terrestrial to transitional environment. It is approximately 3,200 m thick and vests unconformably on Late Cretaceous rocks. The formation interfingers with the Budong-Budong Formation.

The Budong-Budong Formation is a variable sequence of shaly mudstones, quartzose sandstones, limestones, and minor conglomerates aged from Middle Eocene to Late Oligocene, with the estimated thickness of 1,000 - 2,000 m. This formation is underlain unconformably by Late Cretaceous rocks. These phenomena can be seen in the Karama and Lariang regions.

The Lisu Formation is a sequence of interbedded mudstones, greywackes, and pebbly greywackes approximately 2,000 m thick and aged from late Early Miocene to Early Pliocene, based on nannofossil and foraminifera. The lowest part

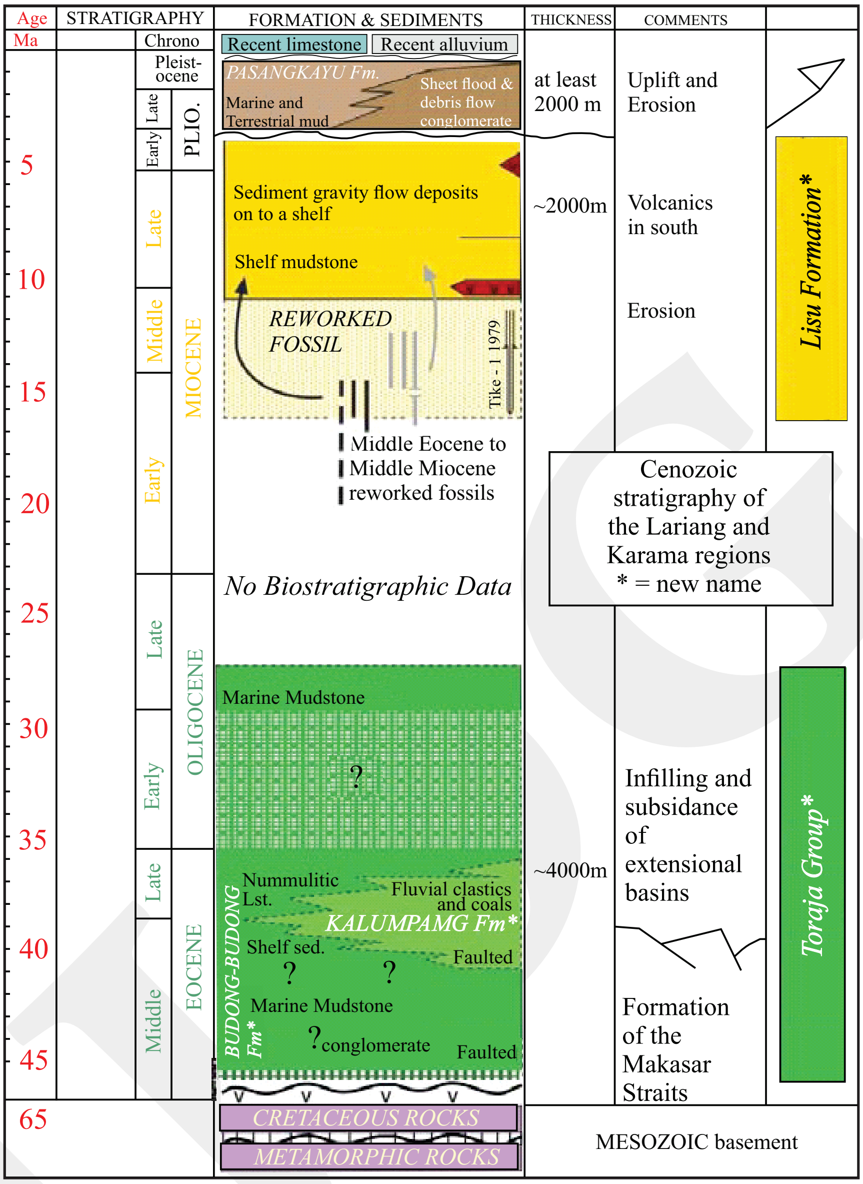

Figure 2. Stratigraphy of western Sulawesi (Calvert and Hall, 2003).

of the Lisu Formation is dominated by mudstone which is similar to the upper part of the Toraja Group. This similarity indicates that the contact between these two formations is conformable on gradual

The Pasangkayu Formation shaving age from Early Pliocene to Pleistocene, based on foraminifera and nannofossils is dominated by conglomerate, sandstone, and mudstone, with a thickness between 2,000 m and 3,500 m.

Sutadiwiria *et al.* (2017) stated that the source facies in western Sulawesi is proposed to be derived from Eocene coals or coaly shales of Toraja Group.

MATERIALS AND METHODS

A set of nine oil seep samples (OS-1 - OS-9) from both Lariang dan Karama Basins were analyzed (Table 1) using capillary gas chromatography (GC) and gas chromatography - mass spectrometry (GC-MS). The oil seep samples were chosen to represent crude oil biomarker composition from the regions, and their locations are shown in Figure 1. All the geochemical analytical works were carried out in the Geochemical Laboratory, CoreLab Dallas using a Hewlett Packard Technologies 5970B MSD

Table 1. Sample Identification of Oil Seeps

	Sample Identification	Location	Original Data/References
Oil seeps	OS-1	Bantaya, Lariang	(Robinson, 1990)
	OS-2	Doda, Lariang	(Robinson, 1990)
	OS-3	Lariang	(Davis, 1990)
	OS-7	Lariang	(Rachmalia and Jatmiko, 2011)
	OS-8	Lariang	(Rachmalia and Jatmiko, 2011)
	OS-9	Karama	(Rachmalia and Jatmiko, 2011)
	OS-4	Polohu, Karama	(Robinson, 1990)
	OS-5	Paniki, Karama	(Robinson, 1990)
	OS-6	Paniki, Karama	(Robinson, 1990)

mass spectrometer with a 5890 series gas chromatograph attached directly to the ion source (70 eV ionization voltage, 100 mA filament emissions current, 230°C interface temperature). The selected ion monitoring capabilities of the data acquisition system permitted specific ions to be monitored, such as tricyclic terpanes and hopanes (m/z 191), steranes (m/z 217), and methylated steranes (m/z 231, not shown in this paper) (Davis, 1990; Robinson, 1990; Rachmalia and Jatmiko, 2011).

The oil seep samples have been characterized and inferred the expected source rock facies in the Lariang and Karama Basins, West Sulawesi. The characterization of oil seep samples was determined by GC-MS chromatograms to conclude the input of organic matter and its depositional environment.

Results And Discussion

The oil seep samples were first analyzed using GC techniques, and their representative results are presented in Figure 3. As a result, visual inspection from the figure shows the GC traces of two representative biodegraded crude oils, each from the Lariang and the Karama Basins, representing level 5 corresponding to severe biodegradation (Wenger and Isaksen, 2002). The biodegradation rank is based on GC traces such as the absence of n-alkanes, and partial removal of isoprenoids. All the oil seep samples contain a complex mixture of unresolved branched/cyclic alkanes visualized by hump, typical biodegraded oils.

All the oil seep samples from the Lariang Basin also show abundant b/c-alkanes < C_{25} relative to that from the Karama Basin, and all samples

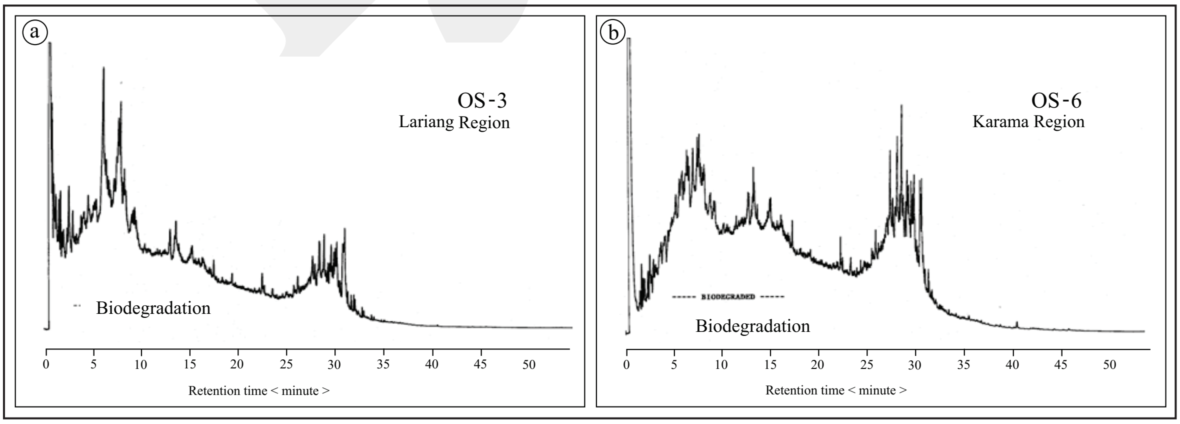

Figure 3. Whole oil chromatograms showing representative GC oil seeps from the Lariang (a) and the Karama Basins (b).

contain relatively high abundant of the three isomeric resin markers (> C_{25}). The oil seep samples from the Karama Basin show more abundant resin marker relative to that from the Lariang counterpart. In brief, the oil seep samples are classified as moderately to severely biodegraded oils. Therefore, the GC data are not reliable for further geochemical assessment.

The more detailed analysis of the branched and cyclic alkane fractions of the oil seeps, using GC-MS techniques was carried out in order to evaluate the composition of the high molecular weight hydrocarbon (sterane and triterpane) biomarkers. These biomarkers were monitored using m/z 217 and m/z 191 mass chromatograms for sterane and triterpane biomarkers respectively. Typical m/z 217 mass chromatograms of' the oil seep samples are shown in Figure 4, with peak assignments in Table 2. Based on these observations, the sterane distribution displays a familiar pattern of steranes with carbon numbers from 27 to 29 completed with the presence of pregnanes and homopregnanes together with rearranged steranes (diasteranes). The pattern of peaks from steranes that looks similar in all samples shows a common terrestrial origin which is complicated by co-elution of other compounds such resin bicadinanes.

The concentrations of rearranged steranes (diasteranes) in the samples were found to occur in all samples analyzed (see peaks A-F in Table 2). The transformation of sterols into rearranged steranes in the presence of clay which has been demonstrated in a laboratory (*e.g.* Sieskind *et al.*, 1979) suggests that rearranged steranes were formed in an acid condition. However, such composition has also been reported to indicate a high thermal maturity for some petroleum (Seifert and Moldowan, 1978). The abundance of these compounds in the oil seep samples has been interpreted as the evidence of a contribution from clay rich sediments to their source rock facies. Another indication is due to biodegradation.

Partial m/z 191 mass chromatograms for representative samples from the two locations of oil seep samples are shown in Figure 5, with peak assignments in Table 2. All the oil seeps have a distribution dominated by triterpanes of higher-plant origin. The 18_{α}(H)- and 18_{β}(H)-oleanane and other oleanoid compounds, labelled a, b, c, and d, previously reported (Ekweozor *et al.*, 1979; Czochanska *et al.*, 1988) are elsewhere together with C_{30} bicadinane resin markers (R) and tetracyclic terpanes (peak x) to be triterpanes of higher-plant origin.

The abundance of oleananes (18_{α}(H)- and 18_{β}(H)-) are higher than the C_{30} hopane in their source rock sediments, and are also presented in sediments from both Gumai Formation of the South Sumatra Basin and Baong Formation of the North Sumatra Basin (Robinson, 1990). The later is Miocene marine sediments with abundant allochthonous terrestrial higher plant markers.

The difference from the two locations can be seen from the abundance of bicadinanes relative

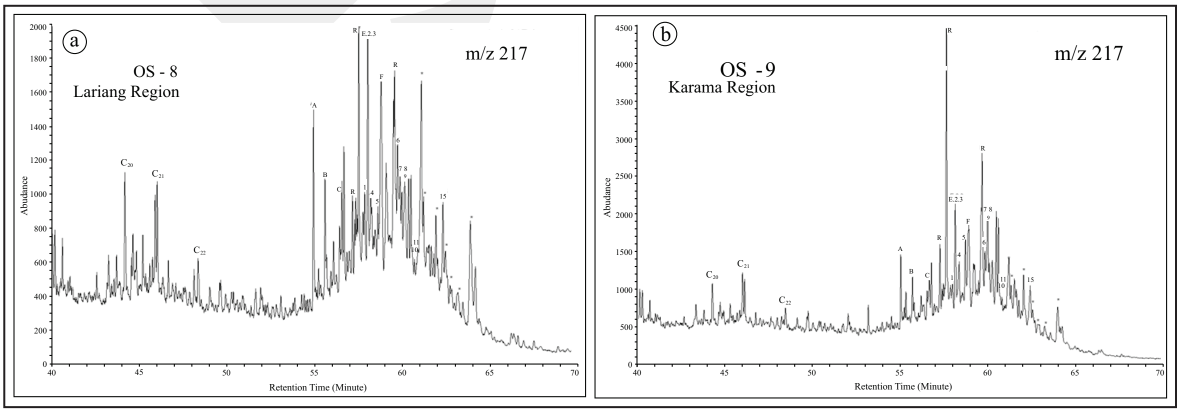

Figure 4. m/z 217 partial mass chromatograms showing sterane distribution in two different locations of oil seeps from the Lariang (a) and the Karama Basins (b). See Table 2 for key to labels.

Table 2. Labels Used for Peak Identification in Mass Chromatograms

Terpane Identification			Sterane Identification		
Peak ID	**Carbon Number**	**Compound Name**	**Peak ID**	**Carbon Number**	**Compound Name**
A	18	Tricyclic diterpane		20	5α, 14α, 17α(H)-Pregnane
B	19	Tricyclic diterpane		21	5α, 14α, 17α(H)-Homopregnane
C	20	Tricyclic diterpane		22	5α, 14α, 17α(H)-Bishomopregnanes
D	21	Tricyclic diterpane	A	27	13β(H), 17α(H)-diacholestane(20S)
E	22	Tricyclic diterpane	B	27	13β(H), 17α(H)-diacholestane(20R)
F	23	Tricyclic diterpane	C	28	24-Methyl-13β(H), 17α(H)-diacholestane(20S)
G	24	Tricyclic diterpane	D	28	24-Methyl-13β(H), 17α(H)-diacholestane(20R)
H	25	Tricyclic diterpane	E	29	24-Ethyl-13β(H), 17α(H)-diacholestane(20S)
I	26	Tricyclic diterpane	F	29	24-Ethyl-13β(H), 17α(H)-diacholestane(20R)
J	27	Tricyclic diterpane	a	27	13α(H), 17β(H)- diacholestane(20S)
K	28	Tricyclic diterpane	b	27	13α(H), 17β(H)- diacholestane(20R)
L	29	Tricyclic diterpane	c	28	24-Methyl-13α(H), 17β(H)- diacholestane(20S)
1	27	18α(H), 21β(H)-22,29,30-trisnorhopane(Ts)	d	28	24-Methyl-13α(H), 17β(H)- diacholestane(20R)
2	27	17α(H), 21β(H)-22,29,30-trisnorhopane(Tm)	e	29	24-Ethyl-13α(H), 17β(H)- diacholestane(20S)
M	30	Tricyclic diterpane	f	29	24-Ethyl-13α(H), 17β(H)- diacholestane(20R)
3	28	17α(H), 21β(H)-28,30-bishorhopane	1	27	5α(H), 14α(H), 17α(H)-cholestane(20S)
4	29	17α(H), 21β(H)- 30-norhopane	2	27	5β(H), 14α(H), 17α(H)-cholestane(20R)
5	29	17α(H), 21β(H)- 30-normoretane	3	27	5α(H), 14β(H), 17β(H)-cholestane(20R)
6	30	17α(H), 21β(H)-hopane	4	27	5α(H), 14β(H), 17β(H)-cholestane(20S)
7	30	17α(H), 21β(H)-moretane	5	27	5α(H), 14α(H), 17α(H)-cholestane(20R)
8	31	17α(H), 21β(H)- 30-hormohopane(22S)	6	28	24-Methyl-5α(H), 14α(H), 17α(H)-cholestane(20S)
9	31	17α(H), 21β(H)- 30-homohopane(22R)	7	28	24-Methyl-5β(H), 14α(H), 17α(H)-cholestane(20R)
10	31	17α(H), 21β(H)- 30-homomoretane	8	28	24-Methyl-5α(H), 14β(H), 17β(H)-cholestane(20R)
11	32	17α(H), 21β(H)-30,31-bishomohopane(22S)	9	28	24-Methyl-5α(H), 14β(H), 17β(H)-cholestane(20S)
12	32	17α(H), 21β(H)-30,31-bishomohopane(22R)	10	28	24-Methyl-5α(H), 14α(H), 17α(H)-cholestane(20R)
13	33	17α(H), 21β(H)-30,31,32-trishomohopane(22S)	11	29	24-Ethyl-5α(H), 14α(H), 17α(H)-cholestane(20S)
14	33	17α(H), 21β(H)-30,31,32-trishomohopane(22R)	12	29	24-Ethyl-5β(H), 14α(H), 17α(H)-cholestane(20R)
15	34	17α(H), 21β(H)-30,31,32,33-tetrahomohopane(22S)	13	29	24-Ethyl-5α(H), 14β(H), 17β(H)-cholestane(20R)
16	34	17α(H), 21β(H)-30,31,32,33-tetrahomohopane(22R)	14	29	24-Ethyl-5α(H), 14β(H), 17β(H)-cholestane(20S)
17	35	17α(H), 21β(H)-30,31,32,33,34-pentahomohopane(22S)	15	29	24-Ethyl-5α(H), 14α(H), 17α(H)-cholestane(20R)
18	35	17α(H), 21β(H)-30,31,32,33,34-pentahomohopane(22R)	R	30	Bicadinane
OL	30	18α(H)-Oleanane	*	30	C_{30}-Methylated Sterane
Gm	30	Gammacerane	*	30	C_{30}-Rearranged Methylated Sterane
R	30	Bicadinanes	16	30	24-Ethyl-4α-Methyl-5α(H), 14α(H), 17α(H)-cholestane(20S)
MH	30	Methylated Hopane	17	30	24-Ethyl-4α-Methyl-5α(H), 14β(H), 17β(H)-cholestane(20S/20R)
a	30	Higher-plant terpane	18	30	4α,23,24-trimethyl-5α(H), 14β(H), 17β(H)-cholestane(20R)
b	30	Higher-plant terpane	19	30	24-Ethyl-4α-Methyl-5α(H), 14α(H), 17α(H)-cholestane(20R)
c	30	Higher-plant terpane	20	30	24-Ethyl-4β-Methyl-5α(H), 14α(H), 17α(H)-cholestane(20R)
d	30	Higher-plant terpane	21	30	4α,23,24-Trimethyl-5α(H), 14α(H), 17α(H)-cholestane
MH	32	Methylated Hopane	22	30	Dinosterane Isomer
X	24	Tetracyclic Terpane	23	30	Dinosterane Isomer

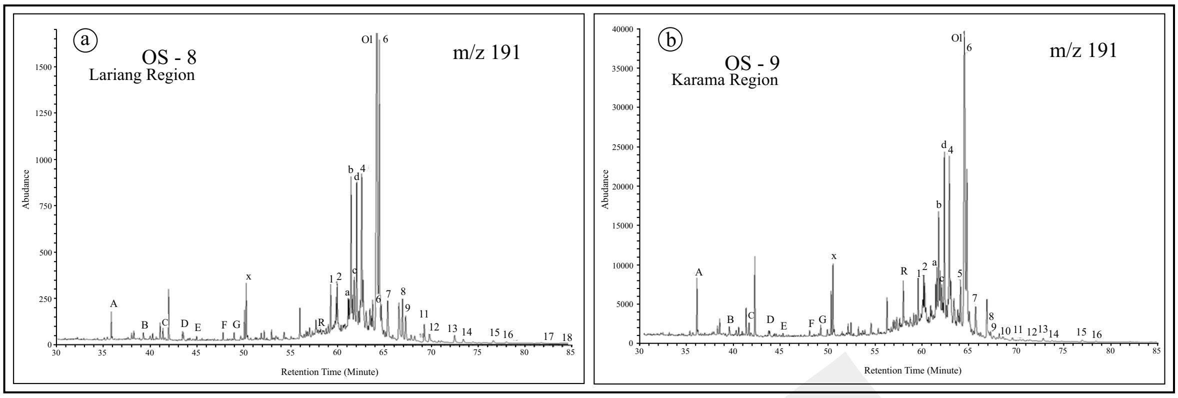

Figure 5. m/z 191 partial mass chromatograms showing triterpane distribution in two different locations of oil seeps from the Lariang (a) and the Karama (b) Basins. See Table 2 for key to labels.

to hopane (see Bic/C$_{30}$H in Table 3), where the Karama samples have relatively more abundant resin marker compared with that of the Lariang counterpart. The less abundance of the extended hopanes (HH Index)(see Table 3) in the samples from Karama compared with the Lariang counterpart provides supportive evidence that these source rocks have been deposited with the more oxic condition than that of the Lariang source rocks. Hence, the higher resin marker content in the Karama oil seep samples may be elsewhere that its source rock environment is located more distal than that for the Lariang oil.

Due to less documentation of biomarker data available in literatures for the Lariang and Karama Basins, the samples used in this study were selected to demonstrate the biomarker composition of the crude oil derived from the two basins. All the samples from both the Lariang and Karama Basins have been classified as a similar biomarker composition of mixed terrestrial higher-plants and marine origin, suggesting to derive from the same area of source rock. The interpreted source rocks for all the oil seep samples are typical marine deltaic sediments. Another report of similar crude oils suggested that the oil sources contained significant content of resin bicadinane, which also indicated the contribution of fluvio-deltaic setting (Sutadiwiria *et al.,* 2017).

The sterane composition could be used to give an indication of source differences (Seifert and Moldowan, 1978). Huang and Meinschein (1979)

Table 3. Biomarker Characteristics of the Oil-seeps from Both Lariang and Karama Basins, West Sulawesi

Sample Name	Location	C_{27}S	C_{28}S	C_{29}S	C_{27}S	Dia	S	Ts	S	Ol	Bic	C_{29}H	Tri	HH Index
			(%)		C_{29}S	Ste	(S+R)20S	Tm	(S+R)22H	C_{30}H	C_{30}H	C_{30}H	C_{30}H	
OS-1	Lariang Basin	60	21	19	3,07	0,97	0,76	2,33	0,57	1,85	0,44	0,59	0,64	0,09
OS-2		76	11	13	5,71	1,80	0,88	1,61	0,63	1,89	0,33	0,47	0,48	0,14
OS-3		51	31	18	2,77	1,08	0,80	1,47	0,66	1,47	0,34	0,51	0,46	0,19
OS-7		33	11	56	0,60	1,20	0,17	1,89	0,00	0,93	0,21	0,52	0,56	0,15
OS-8		31	10	59	0,52	1,20	0,16	1,89	0,00	0,91	0,19	0,52	0,54	0,16
OS-4	Karama Basin	79	8	13	6,33	1,50	0,92	1,43	0,67	4,17	0,89	1,11	0,82	0,03
OS-5		74	19	7	11,50	1,73	0,80	12,50	0,60	2,94	0,92	0,94	0,85	0,04
OS-6		74	12	15	5,00	1,57	0,69	2,56	0,57	2,27	0,92	0,8	0,87	0,05
OS-9		57	11	32	1,75	0,73	0,27	1,75	0,00	0,51	0,48	1,03	1,68	0,03

proposed that C_{27} sterols mainly derived from algae, while the C_{29} sterols are more typically associated with land plants. However, efforts to apply this concept to steranes (*e.g:* C_{27}, C_{28}, and C_{29}) in source rock and crude oil to describe the source-rock depositional environment only have limited success (Mackenzie *et al.*, 1983) due to so much overlap between the different oil source type limits (Moldowan *et al.*, 1995) with the possible exception of certain samples containing predominantly higher-plant organic matter. However, Peters *et al.* (2000) used sterane ternary diagrams to correlate oils and/or source rock bitumens from Mahakam Delta, Indonesia. Therefore, these biomarker interpretations should be used with caution.

Figure 6 shows a ternary diagram showing the relative abundances of steranes (C_{27}- C_{29}). Seep oil samples show a relatively higher proportion of C_{27} steranes (31 - 76% and 57 - 79%) compared to those of C_{28} (10 - 31% and 8 - 19%) and C_{29} (13 - 59% and 7 - 32%) sterane counterparts for Lariang and Karama Basins respectively (Table 3). This evidence reflects a high contribution of aquatic algal organic matter and a minor terrigenous organic matter input (Peters *et al.*, 2005). From the Figure 6, it is evident that the high abundance of C_{27}steranes for OS-1 – OS-3 samples combined with other biomarker data (see Table 3) is not easy to explain whether it is due to the geographic extent of the Karama Basin, or it reflects a mixture of samples from both locations. This is probably the lack of applying a sterane ternary diagram using data derived from a conventional SIM/GCMS.

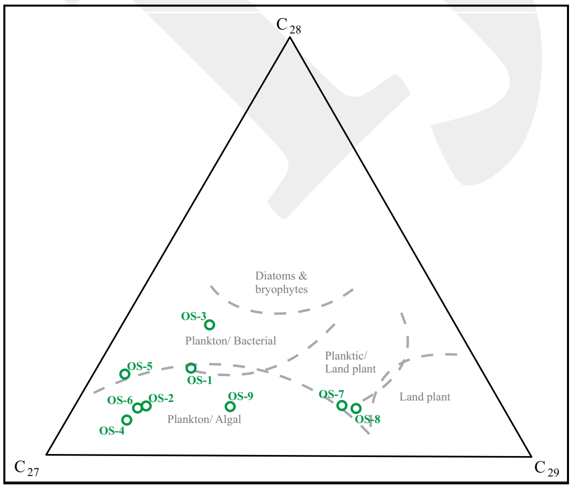

Figure 6. Sterane ternary diagram (C_{27}, C_{28}, and C_{29}) for oil seep samples from both the Karama and Lariang Basins (modified after Huang and Meinschein, 1979).

In general, steranes and hopanes are not biodegraded at lower ranks. At higher levels of biodegradation (level > 5), in some reservoirs hopanes can be demethylated at C-10 (*e.g.* Bennett *et al.*, 2006), resulting in abundant 25-norhopane series (C_{27}-C_{34}). However, the degradation of hopanes can occur without the formation of 25-norhopanes (*e.g.* Bost *et al.*, 2001). Regular steranes are also susceptible and are removed from extremely biodegraded oils. However, other triterpanes, such as C_{19} - C_{24} tricyclicterpanes (cheilanthanes), C_{27}Ts, C_{27}Tm, C_{29}Ts, gammacerane, 18_{α}(H)-oleanane, and 18_{β}(H)-oleanane, as well as C_{27}diasteranes can survive during microbial degradation (Connan, 1984; Peters *et al.*, 2005), leading to enrichment in these biomarkers at a high rank of biodegradation scale (extreme biodegradation).

Oleananes, as markers for the angiosperms, provide a valuable source and age information when occur in oil. The oleananes are formed in sediments through diagenetic and catagenetic alteration of various 3_{β}–functionalized angiosperm triterpenoids (Rullkotter *et al.*, 1994). The abundance of oleananes in terrestrial oils and sediments may be highly sensitive to changes in early diagenetic condition. The oleananes are absent or present at very low concentrations in samples from the base of an Eocene coal seam affected by post-depositional seawater intrusion (Murray *et al.*, 1997).

In addition, Murray *et al.* (1997) show a distribution of the 18α(H)-oleanane relative to C_{30}hopane (Ol/C_{30}H) in shale and coal from the South Sumatra Basin, Indonesia. Such feature has been observed previously for interbedded deltaic coals and shales from the Far East by Curiale and Lin (1991). Clearly, oleananes can only occur in sediments or oil and their abundance is likely to be highly sensitive to changes in Eh and pH during early diagenesis. Overall, it suggests that marine overprinting of coals increases the oleanane relative to C_{30}hopane of its source rock bitumen.

CONCLUSIONS

The oil seeps associated with both the Lariang and Karama Basins exhibited a wide range of concentrations of biomarker hydrocarbons and intensities of biodegradation. Their biomarker compositions have been used to infer the depositional environment of their source rock facies. All the oil seep samples from both the Lariang and the Karama Basins contain similar compositions of steranes with carbon number from 27 to 29 are present, together with pregnanes and homopregnanes, relatively high rearranged steranes, abundant oleananes, and other triterpenoids, bicadinanes of probable higher-plant origin along with series of hopane family. The abundance of biomarkers of terrestrial origin and the presence of saturated oleananes suggest the source rocks deposited in a deltaic or nearshore environment. All the seep samples have been inferred to be sourced from the same area. But the source rock for the Karama oil seep samples seems to be located more distal than that for the Lariang oil.

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