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Geochemical Characterization of Coal, Carbonaceous Shale, and Marine Shale As Source Rock in West Sulawesi, Indonesia

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Abstract - The eastern part of the Makassar Strait has not achieved significant success in the petroleum exploration stage. In general, exploration wells in this area have not found hydrocarbons to date. Most of the problems experienced by these wells are geochemical-related, including the absence of a source rock where the hydrocarbon is formed, immature source rock, and low volume of hydrocarbon. Therefore, this study aims to find potential Eocene source rock in West Sulawesi. It commenced by determining the paleofacies followed by the geochemical analysis of well cutting and surface samples. The geochemical analyses conducted are total organic carbon (TOC), rock eval pyrolysis (REP), kerogen type, vitrinite reflectance (VR), and gas chromatography/mass spectrometry (GC/MS). The samples that have a potential as an Eocene source rock were coal, as well as carbonaceous and marine shale. Based on the plotting of relative abundance for sterane (C_{27} - C_{29}), the coal and carbonaceous shale had the input from terrestrial organic material with a contribution from marine, while marine shale had the input from marine organic material and contribution from terrestrial. Furthermore, coal and carbonaceous shale had a relatively high proportion of C_{29} sterane compared to C_{27} and C_{28}, while marine shale had a relatively high proportion of C_{27} compared to C_{28} and C_{29}. The higher plants also contributed to the organic matter in carbonaceous shale and coal samples, as indicated by the abundance of oleanane/C_{30} hopane and the other significant content of resin biomarker. In accordance with the objectives of the study, it was found that during the Eocene, the type of source rock in the eastern side of the province varied, starting with coal and carbonaceous shale in the upper intertidal-fluviodeltaic environment, and marine shale sediments from the source facies of marine algal.

Keywords: coal, carbonaceous shale, marine shale, source rock, West Sulawesi

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Introduction

Background

There have been intense petroleum exploration activities on the western side of Makassar Strait since 1990, but on the eastern side, the exploration is limited. The activities on the western side are carried out using 2D - 3D seismic acquisition, collection, and analysis of other geological-geophysical data, but significant success has not been achieved. Despite several drilling activities, no hydrocarbon has been found in the Makassar Strait to date.

Calvert (2000) mentioned that during the Middle Eocene - Late Eocene, the opening

through extensional tectonics formed a system of spreading continental crust, creating a series of interconnected graben and half graben structures, with a northeast-southwest trending structural pattern. These structures are filled with fluvio-deltaic to marine sediments, which are included in the Toraja Group. Furthermore, oil seep analysis from Sutadiwiria *et al*. (2017 and 2018) suggests that the source rocks in the Lariang and Karama Basins, West Sulawesi, were deposited in a fluvio -deltaic to deltaic or nearshore environment in Toraja Group, due to the abundance of terrestrial biomarkers and saturated existence of oleanane in this area. All the seep samples were concluded to be sourced from the same area, but the source rock for the Karama oil seeps samples seems to be located more distal than the Lariang oil.

This study was conducted by analyzing the potential source rock in West Sulawesi, specifically on coal, as well as carbonaceous and marine shale, to determine the specific characterization of biomarkers. Coal and carbonaceous shale samples were taken from outcrop in Mamuju, while marine shale was taken from cuttings of KYS well, located on onshore of West Sulawesi (Figure 1). Meanwhile, all the samples have not

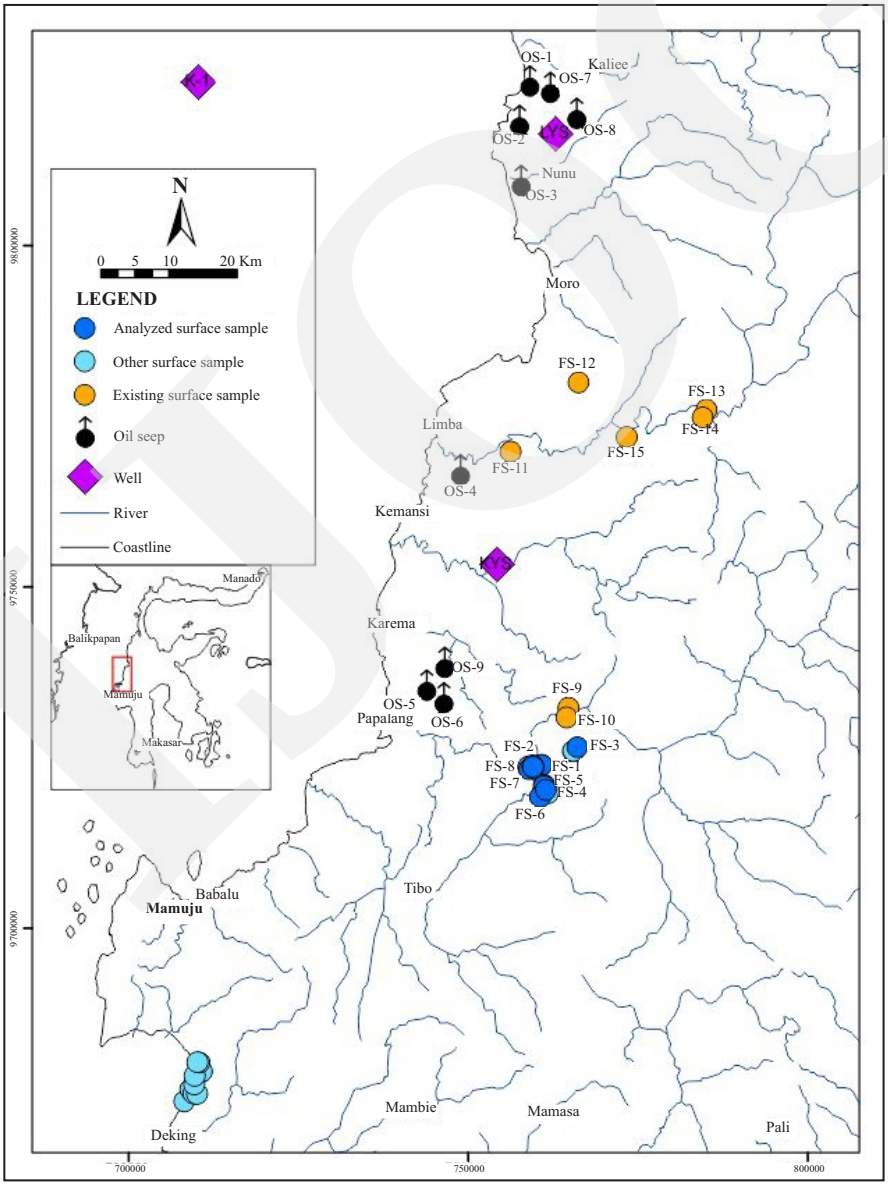


Figure 1. Locality map of the samples.

been investigated nor analyzed previously. The three samples were believed to be part of Kalumpang Formation of Toraja Group mentioned by Calvert (2000), and assumed to be the source rock in the eastern area of the province. Calvert (2000) stated that Kalumpang Formation consisted of carbonaceous claystone (shale) sequences, coal, siltstone, and quartz sandstone deposited in the terrestrial-marginal marine environment. It is expected that the results of geochemical analysis of coal, carbonaceous shale, and marine shale will increase the understanding of Eocene source rocks in the eastern area of the province.

Geological Settings

The formation of the Makassar Strait consists of six stages, which are based on seismic data along this area, namely the pre-rift stage of the Pre-Tertiary bedrock, the syn-rift in the Middle-Late Eocene, the post-rift in the Oligocene, Early-Middle-Late Miocene, and foreland basin stages in the Pliocene-Recent (Nur'Aini *et al*., 2005). Understanding the tectonostratigraphic sequence is linked to field data of the Lariang-Karama Subbasin in West Sulawesi by Calvert (2007), as well as the Kutai Basin in Kalimantan as an analog and approach.

The Kalumpang and the Budung-budung Formations belong to the Toraja Group, based on their lithological characteristics and the depositional environment (Calvert, 2000) as shown in Figure 2. The Kalumpang Formation is deposited in a terrestrial-marginal marine environment, consisting of carbonaceous claystone or shale sequences, coal, siltstone,

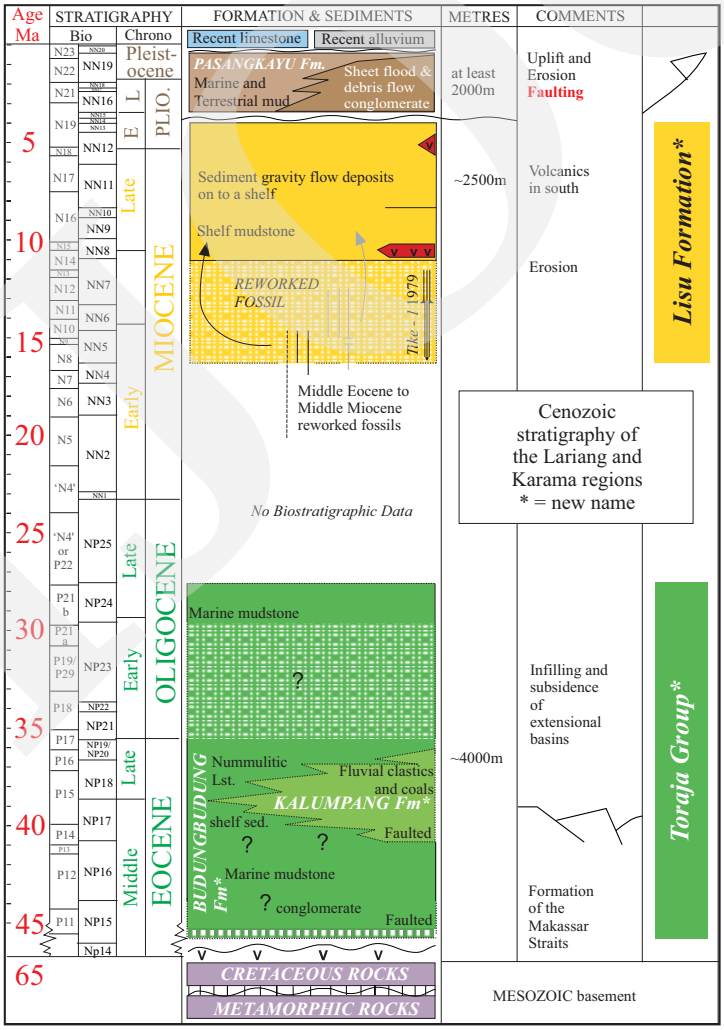


Figure 2. Stratigraphy of western Sulawesi (Calvert, 2000).

and quartz sandstone, with the thickness up to 3,200 m in several areas. The formation was deposited unconformably on Late Cretaceous rock, in a graben or half-graben oriented northeast-southwest, in the Karama Subbasin, West Sulawesi.

Meanwhile, the Budung-budung Formation was deposited in a shallow fluvial-marine environment, consisting of shale, shaly claystone, limestone, and a few conglomerates, incompatible, and deposited on metamorphic bedrock, black shale, and Late Cretaceous volcanic rocks. This formation also has an interfingering relationship with the Kalumpang Formation, with an estimated minimum thickness of 1,000 - 2,000 m (Calvert, 2000).

The Early Miocene-Pliocene Lisu Formation was deposited above the Toraja Group which comprises of the Kalumpang and the Budung-budung Formation (Figure 2). It is located at the Karama area, in the Budung-budung River. The thickness reaches 2,000 m. Furthermore, the characteristics of the facies at the bottom are similar to those of the upper Toraja Group, which are dominated by claystone during the Eocene-Oligocene. With no evidence of inconsistency in the field, the Lisu Formation contacts are assumed to be aligned above the Toraja Group.

In general, the Pasangkayu Formation is dominated by conglomerates and sandstone layers, as well as claystone. The conglomerate is formed from the deposition of alluvial fans that border and interfinger with the plains as well as the marine environment (inner-neritic). This formation is assumed to be an Early Pliocene-Pleistocene age, between 2,000 - 3500 m thick, deposited unconformably on Cenozoic or older rocks, and misaligned by Quaternary alluvials with limestone.

Methods and Materials

Several geochemical analyses were conducted on surface samples or outcrops in West Sulawesi, including analysis of total organic carbon (TOC), rock eval pyrolysis (REP), kerogen type, vitrinite reflectance (VR), gas chromatography (GC), and gas chromatography/mass spectrometry (GC/MS). The map of the sample locations is shown in Figure 1, and the data for all analyzed samples are presented in Table 1.

The peak identification on the chromatogram is shown in Table 2, and all geochemical analysis was conducted at the geochemical laboratory of P.T. Geoservices. The standard method used is internationally recognized according to ISO 9001:2015.

Table 1. Field Sample Identification and the Result of TOC, REP, and VR

	Sample Identification		Lithology	TOC	Tmax	HI	OI	PI	VR (%)
	Field ID	Name							
Surface Sample	1	FS-16	Black claystone	0.11	-	-	-	-	-
	7	FS-17	Black claystone	0.16	-	-	-	-	-
	8	FS-18	Black claystone	0.20	-	-	-	-	-
	9	FS-19	Black claystone	0.64	442	7	13	0.64	0.36
	12	FS-20	Carbonaceous shale	6.32	429	189	37	0.02	0.38
	25	FS-21	Claystone	0.10	-	-	-	-	-
	26	FS-22	Claystone	0.11	-	-	-	-	-
	31	FS-23	Red claystone	0.11	-	-	-	-	-
	4	FS-2	Coal	30.56	423	109	69	0.02	0.46
Well	4.310–4.400 m	KYS-6	Shale	2.49	416	122	88	0.17	0.77

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	A	27	13β(H),17α(H)-diacholestane(20S)
B	19	Tricyclic diterpane	B	27	13 β (H),17α(H)-diacholestane(20R)
C	20	Tricyclic diterpane	C	28	24-Methyl-13β(H),17α(H)-diacholestane(20S)
D	21	Tricyclic diterpane	D	28	24-Methyl-13β(H),17α(H)-diacholestane(20R)
E	22	Tricyclic diterpane	E	29	24-Ethyl-13β(H),17α(H)-diacholestane(20S)
F	23	Tricyclic diterpane	F	29	24-Ethyl-13β(H),17α(H)-diacholestane(20R)
G	24	Tricyclic diterpane	a	27	13α(H),17β(H)-diacholestane(20S)
H	25	Tricyclic diterpane	b	27	13α(H),17β(H)-diacholestane(20R)
I	26	Tricyclic diterpane	c	28	24-Methyl-13α(H),17β(H)-diacholestane(20S)
J	27	Tricyclic diterpane	d	28	24-Methyl-13α(H),17β(H)-diacholestane(20R)
K	28	Tricyclic diterpane	e	29	24-Ethyl-13α(H),17β(H)-diacholestane(20S)
L	29	Tricyclic diterpane	f	29	24-Ethyl-13α(H),17β(H)-diacholestane(20R)
1	27	18α(H),21β(H)-22,29,30-trisnorhopane(Ts)	1	27	5α(H),14α(H),17α(H)-cholestane(20S)
2	27	17α(H),21β(H)-22,29,30-trisnorhopane™)	2	27	5β(H),14α(H),17α(H)-cholestane(20R)
M	30	Tricyclic diterpane	3	27	5α(H),14β(H),17β(H)-cholestane(20R)
3	28	17α(H),21β(H)-28,30-bishorhopane	4	27	5α(H),14β(H),17β(H)-cholestane(20S)
4	29	17α(H),21β(H)-30-norhopane	5	27	5 α(H),14α(H),17α(H)-cholestane(20R)
5	29	17β(H),21α(H)-30-normoretane	6	28	24-Methyl-5α(H),14α(H),17α(H)-cholestane(20S)
6	30	17α(H),21β(H)-hopane	7	28	24-Methyl-5β(H),14α(H),17α(H)-cholestane(20R)
7	30	17β(H),21α(H)-moretane	8	28	24-Methyl-5α(H),14β(H),17β(H)-cholestane(20R)
8	31	17α(H),21β(H)-30-homohopane(22S)	9	28	24-Methyl-5α(H),14β(H),17β(H)-cholestane(20S)
9	31	17α(H),21β(H)-30-homohopane(22R)	10	28	24-Methyl-5α(H),14α(H),17α(H)-cholestane(20R)
10	31	17β(H),21α(H)-30-homomoretane	11	29	24-Ethyl-5α(H),14α(H),17α(H)-cholestane(20S)
11	32	17α(H),21β(H)-30,31-bishomohopane(22S)	12	29	24-Ethyl-5β(H),14α(H),17α(H)-cholestane(20R)
12	32	17α(H),21β(H)-30,31-bishomohopane(22R)	13	29	24-Ethyl-5α(H),14β(H),18β(H)-cholestane(20R)
13	33	17α(H),21β(H)-30,31,32-trishomohopane(22S	14	29	24-Ethyl-5α(H),14β(H),17β(H)-cholestane(20S)
14	33	17α(H),21β(H)-30,31,32-trishomohopane(22R)	15	29	24-Ethyl-5α(H),14α(H),17α(H)-cholestane(20R)
15	34	17α(H),21β(H)-30,31,32,33-tetrahomohopane(22S)	R	30	Cyclic Alkane
16	34	17α(H),21β(H)-30,31,32,33-tetrahomohopane(22R)	*	30	C30-Methylated Sterane
17	35	17α(H),21β(H)-30,31,32,33,34-pentahomohopane(22S)	*	30	C30-Rearranged Methylated Sterane
18	35	17α(H),21β(H)-30,31,32,33,34-pentahomohopane(22R)	16	30	24-Ethyl-4α-Methyl-5α(H),14α(H),17α(H)-Cholestane(20S)
OL	30	18?(H)-Oleanane	17	30	24-Ethyl-4α-Methyl-5α(H),14β(H),17β(H)-Cholestane(20S/20R)
Gm	30	Gammacerane	18	30	4α,23,24-Trimethyl-5α(H),14β(H),17β(H)-Cholestane(20R)
R	30	Cyclic Alkane	19	30	24-Ethyl-4α-Methyl-5α(H),14α(H),17α(H)-Cholestane(20R)
MH	30	Methylated Hopane	20	30	24-Ethyl-4β-Methyl-5α(H),14α(H),17α(H)-Cholestane(20R)
a	30	Higher-plant terpane	21	30	4 α,23,24-Trimethyl-5α(H),14α(H),17α(H)-Cholestane
b	30	Higher-plant terpane	22	30	Dinosterane Isomer
c	30	Higher-plant terpane	23	30	Dinosterane Isomer
d	30	Higher-plant terpane			
MH	32	Methylated Hopane			
X	34	Tetracyclic Terpane			

TOC measurements were conducted and continued with rock eval pyrolysis (REP) when the rock contained TOC more than 0.5, while other analysis included kerogen type and vitrinite reflectance (VR). Furthermore, the TOC, REP, and VR classifications used are consistent with Peters and Cassa (1994). The characteristics of the samples are shown on the GCMS profile, including the organic material content and the source facies.

The general standard used as an indication of source rock potential is the TOC value scale for sedimentary rocks from Peters and Cassa (1994), which was then modified as shown in Table 3. This TOC parameter is used in the initial stage to determine the potential for hydrocarbons in rock before proceeding to the following stage of analysis.

Table 3. Indications of Source Rock Potential Based on TOC Values (Peters dan Cassa, 1994)

Source Rock Implications	TOC Value (% weight)
Poor source rock	0 - 0.5
Fair source rock	0.5 - 1
Good source rock	1 - 2
Very good source rock	2 - 4
Excellent source rock	> 4

The quality of the organic material also analyzed is the kerogen type which is a determinant of the basic properties of petroleum and gas products. The concentration consists of five primary elements, namely carbon, hydrogen, oxygen, nitrogen, sulfur. The method used to classify the types is the graph value between hydrogen (HI) and oxygen (OI) known as the Van Krevelen diagram (Peters and Cassa, 1994), as well as a comparison between the value of hydrogen index (HI) and Tmax.

The maturity of the organic material can be determined from the value of Tmax and the reflectance of vitrinite (Ro). Tmax as a maturity indicator is the temperature at the maximum rate of pyrolysis. Moreover, the reflectance of vitrinite (Ro) can be used as a parameter of maturity because with the increasing temperature, the gloss or reflectance of vitrinite maceral also increases.

Aside from the primary data, secondary data were obtained from the existing well drilling results such as the biostratigraphic analysis including foraminifera, nannofossil, and palynology analysis by Muhamad *et al*. (2012). The well-cutting samples were obtained from existing well samples (K-1 well) located in the eastern Makassar Strait and KYS well located in West Sulawesi (Figure 1). Furthermore, the biostratigraphic data were obtained from the analysis carried out previously by the existing contractor. All study results on primary and secondary data samples are comprehensively correlated and integrated as shown in Figure 3.

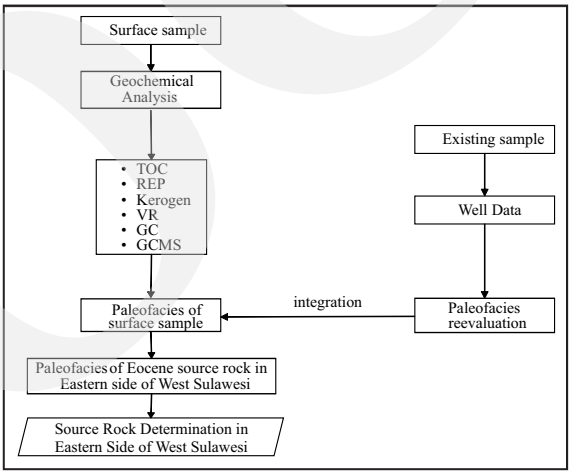


Figure 3. Flow chart of the study.

Results and Discussion

Based on the TOC value of the FS-20 sample namely 6.32%, which is considered to be good, REP analysis was then conducted with S2 result of 11.94 mgH /gOC, hydrogen index (HI) of 189 mg/C, and Tmax of 429°C. The results showed that the FS-20 sample tends to form gas prone - Type III kerogen, as shown by the VR value of 0.38%.

The characteristics shown by the FS-20 sample are similar to the existing coal sample (FS-2), having TOC of 30.56%, HI of 109 mg/C, Tmax of 423°C, and VR of 0.46%. This indicates

that the two samples namely FS-20 and FS-2 have high to very high TOC. Based on the value of HI, they also tend to form gas prone - Type III kerogen.

In the GCMS profile of FS-20 and FS-2 samples, specifically on the mass fragmentogram ion m/z 191 saturated hydrocarbon fraction, the ratio of oleanane to C_{30} hopane (oleanane/C_{30} hopane) were 0.05 and 0.93, respectively (Figures 4 and 5). Furthermore, several other terrestrial plant components such as resin compounds were also found, for example, ion bicadinane appeared in both FS-20 and FS-2 samples, while gamacerane was only found in FS-20.

The biomarker ion of bicadinane appeared as components of W, T, R, a, b, c, and d, which came from higher plants namely terrestrial or HPT. The biomarker index as the same terrestrial component, namely taraxastana (Tx), also appeared together with oleanane (Figure 4 and 6).

In the GCMS profile of mass fragmentogram ion m/z 217, the saturated hydrocarbon fraction shows that FS-20 and FS-2 samples have percentages of C_{27} sterane with 10.73% and 26.5%,

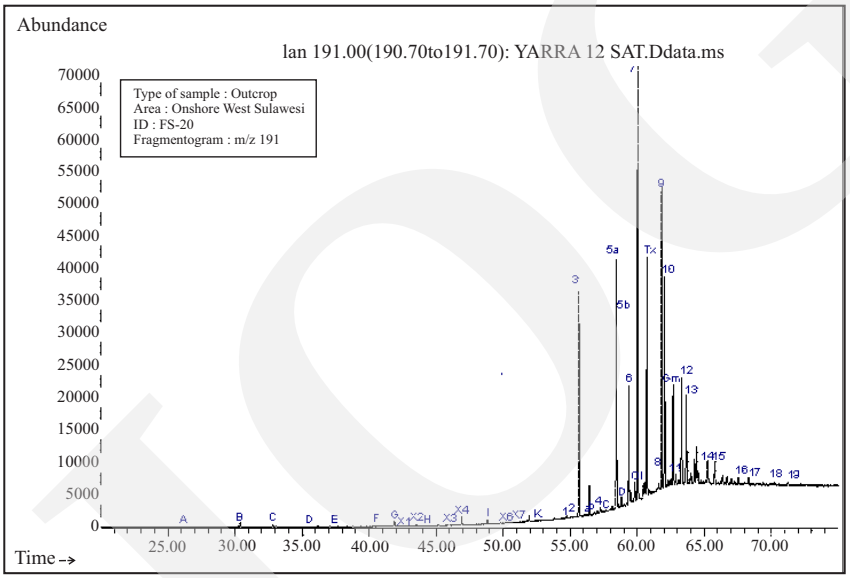


Figure 4. m/z 191 partial mass chromatograms showing triterpane distribution of FS-20. See Table 2 for key to labels.

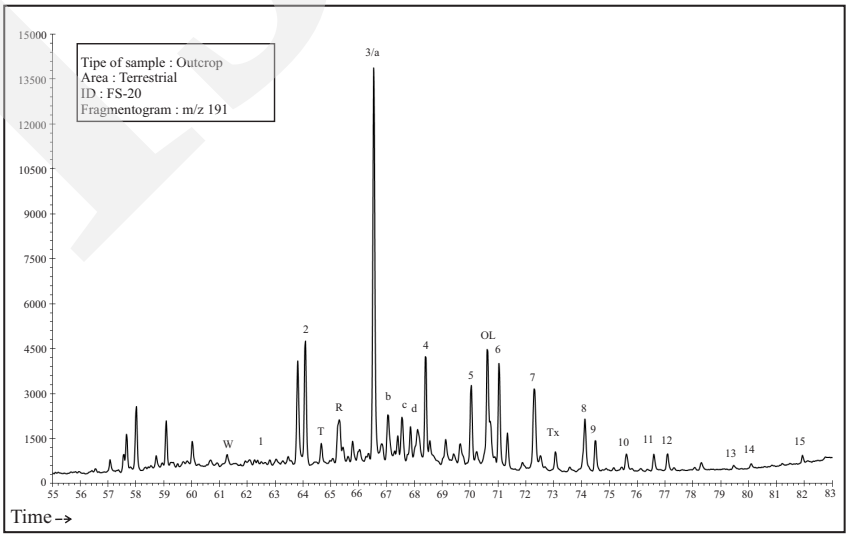


Figure 5. m/z 191 partial mass chromatograms showing triterpane distribution of FS-2. See Table 2 for key to labels.

C_{28} sterane of 34% and 25.21%, and C_{29} sterane of 55.27% and 48.29%, respectively (Figures 6 and 7).

Based on the plotting of the relative abundance of sterane (C_{27} - C_{29}) on the ternary diagram of the FS-20 and FS-2 samples (Figure 8) from Peters *et al*. (2005) and Huang and Meinschein (1979), a relatively high proportion of C_{29} sterane namely 55.27% - 48.29% was shown, compared to C_{27} of 10.73% - 26.5% and C_{28} of 34% - 25.21%. This suggests that the surface samples of carbonaceous shale (FS-20) and coal (FS-2) were deposited in the upper intertidal -fluviodeltaic environment, with the input of terrestrial and marine organic material. This is consistent with the palynology analysis results of these two surface samples, namely the upper intertidal-fluviodeltaic environment (Harsanti, 2017; Geoservices, 2020). It also supports a study which stated that significant content of resin biomarkers in the source rock

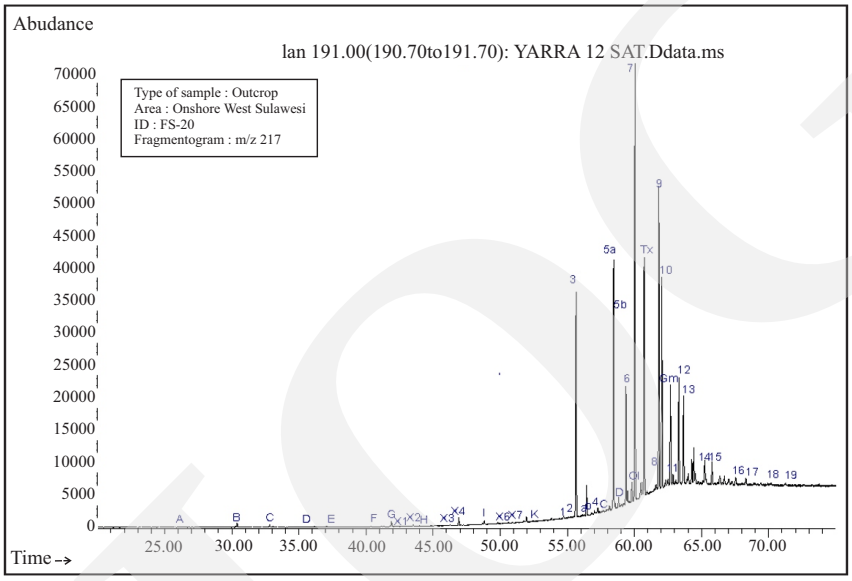


Figure 6. m/z 217 partial mass chromatograms showing sterane distribution of FS-20. See Table 2 for key to labels.

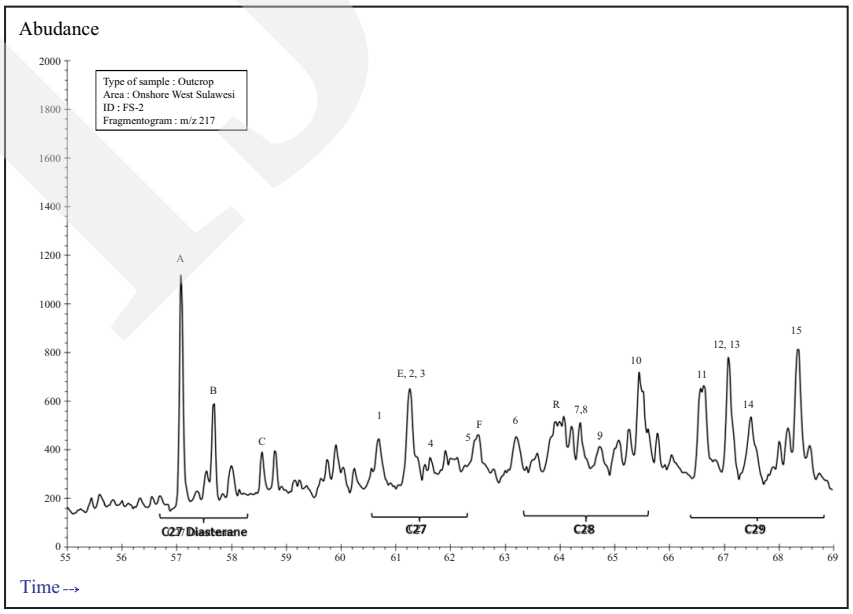


Figure 7. m/z 217 partial mass chromatograms showing sterane distribution of FS-2. See Table 2 for key to labels.

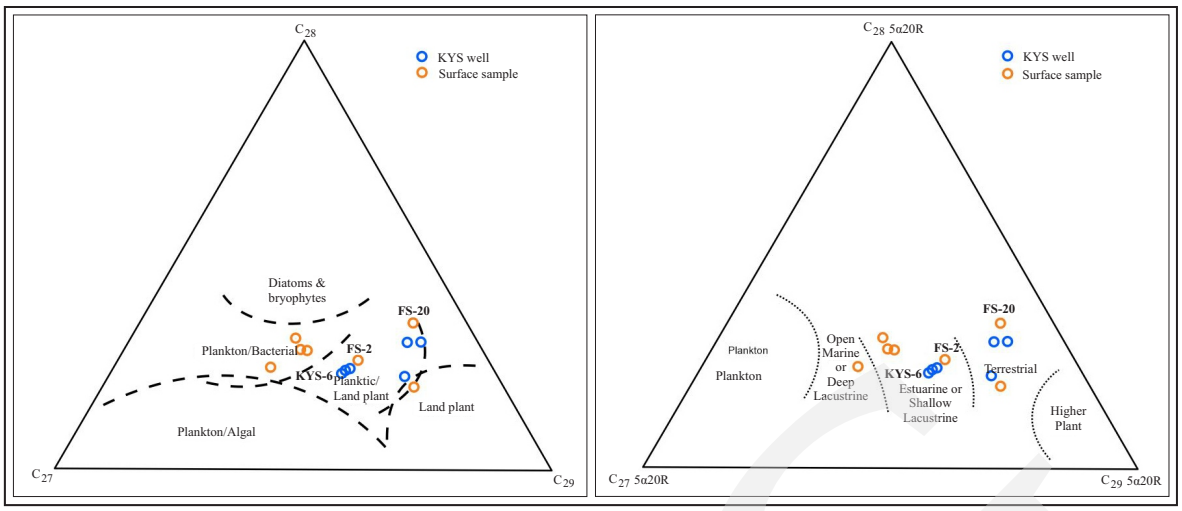


Figure 8. Saturated fraction of the surface sample extract plotted on the ternary diagram according to the (a). Huang and Meinschein, 1979 and (b). Peters *et al*., 2005.

indicates input from fluviodeltaic environment (Sutadiwiria *et al*., 2017).

Based on the geochemical analysis results from the KYS-6 sample, the values of TOC, HI, Tmax, and VR were 2.49%, 122 mg/C, 416°C, and 0.77%, respectively. The HI value of 122 mg/C indicates that the KYS-6 sample has a tendency to form gas Type-III kerogen (Peters and Cassa, 1994).

In the GC/MS profile of KYS well sample shown in Figures 9 and 10, specifically on the mass fragmentogram ion m/z 191 saturated hydrocarbon fraction, certain terrestrial materials appear including oleanane (OL), bicadinane, taraxastane, and gamacerane, while the relative abundance of sterane (C_{27} - C_{29}) is depicted by the GC/MS profile of the m/z 217 ion mass fragmentogram of the saturated hydrocarbon fraction. The relative abundance plot of sterane (C_{27} - C_{29}) illustrates a relatively high proportion of C_{29} compared to C_{27} and C_{28} (Figure 8). This shows that the KYS sample has an input from

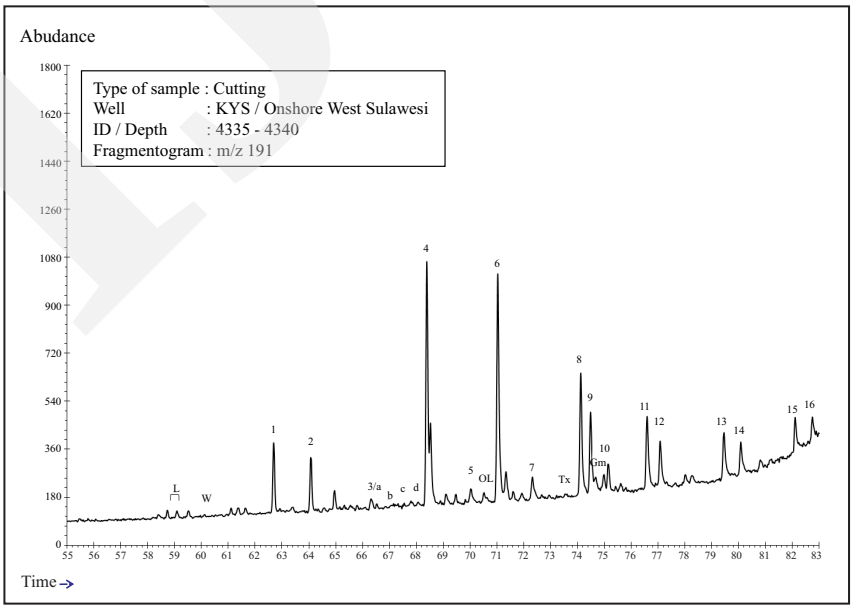


Figure 9. Partial mass chromatograms showing sterane distribution of KYS-6. See Table 2 for key to labels.

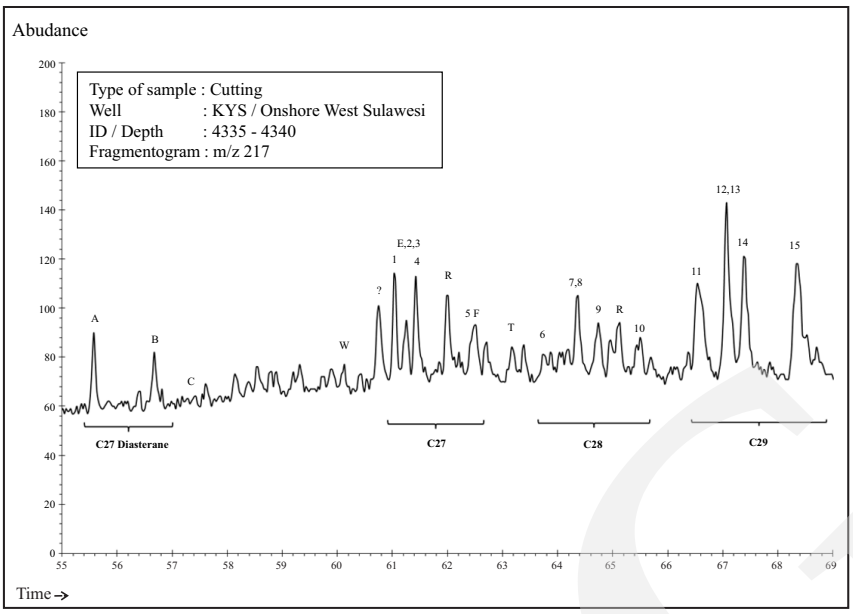


Figure 10. Partial mass chromatograms showing sterane distribution of KYS-6. See Table 2 for key to labels.

terrestrial and contribution from marine organic material.

Several surface samples were also found in the form of shale and shaly claystone with an age of Late Cretaceous-Middle Eocene, and paleofacies of intertidal, outer neritic to upper bathyal. The proportion of C_{27} sterane for KYS-6 and surface samples were also relatively high ranging 30.85 - 45.02% compared to the proportions of C_{28} and C_{29} (Figure 8).

Compared with the existing results of the K-1 well in the eastern Makassar Strait, in the form of biostratigraphic, petrographic, and radiometric dating analysis, at depth of 16,910 - 17,300 ft for Early Paleocene-Eocene (Harsanti *et al*., 2013), the lithologies are shale and shaly claystone, with paleofacies of marginal lacustrine, with volcanic materials at depth of 17,340 - 17,360 ft, and age of Late Cretaceous (Geoservices, 2012).

Based on the shale and shaly claystone lithology on surface samples and well data, with upper intertidal-bathyal paleofacies, old age of Early Paleocene-Late Eocene, and a relatively high proportion of C_{27} sterane compared to C_{28} and C_{29}, a new conclusion can be made, namely the discovery of marine shale source rocks in West Sulawesi and lacustrine shale source rocks in the eastern Makassar Strait (Figure 11). This

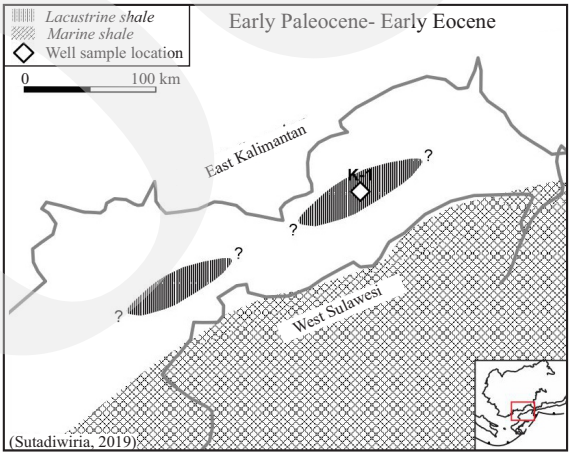


Figure 11. Paleogeographic map of Middle Eocene Marine shale source rock in West Sulawesi.

conclusion is consistent with the nannofossil analysis of KYS samples (Muhamad *et al.*, 2012) as well as the biostratigraphic analysis from surface samples (Harsanti, 2017), with the environment of marine from the middle to outer neritic. Furthermore, the results are in line with Sutadiwiria *et al.* (2018), which stated that the source rocks in the province were deposited in a deltaic or nearshore environment.

From the biostratigraphic analysis and dating of samples obtained in the well and on the surface, a new interpretation was made, namely the splitting and structuration of graben, at least not

earlier than the Early Paleocene. This inference differs from previous studies which stated that the expansion of the Makassar Strait and the formation of graben occurred during the Middle Eocene (Situmorang, 1982; Hall, 1996; Guntoro, 1999; Calvert and Hall, 2007; and Satyana, 2015).

Conclusions

There are three source rocks and two source facies in West Sulawesi, namely:

Coal and carbonaceous shale which are the source rock of the upper intertidal–fluviodeltaic source facies of the Toraja Group.

Marine shale, which is the source rock from marine algal source facies.

The petroleum system in West Sulawesi still has the potential to be explored, specifically with the discovery of coal, as well as carbonaceous and marine shale source rock in this area. The comprehensive evaluation of post-drilling laboratory analysis adapted to geological-geophysical-geochemical (3G) data will enhance the assessment of opportunities and risks to exploration activities in the area.

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