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Petroleum source rock characteristics of marine versus coastal settings: A comparative study between Madbi Formation of Masila Basin, Yemen and Nyalau Formation of Sarawak, Malaysia

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Abstract: Two sets of contrasting oil-prone source rocks have been evaluated and compared - marine shales of the Jurassic Madbi Formation from Masila Basin, Yemen and Cenozoic coals and organic-rich sediments of the Nyalau Formation from Sarawak, Malaysia. An assessment based on organic facies characteristics, has been carried out on these sediments to distinguish, characterise and evaluate source rocks deposited in marine versus coastal plain depositional settings. Good source rock potential is suggested by the high TOC values of Madbi shales and organic-rich sediments of the Nyalau Formation. This is supported by high hydrocarbon indices of 450-752 mgHC/gTOC for the Madbi shales and 295-516 mgHC/gTOC for the Nyalau Formation coaly shales and coals. The Madbi shales have vitrinite reflectance values ranging from 0.74-0.88 Ro% indicating an early mature to peak mature stage, while the Nyalau sediments have vitrinite reflectance values of 0.50-0.66%, suggesting early maturity. The Madbi shale is expected to be a better source rock for oil as indicated from its higher abundance of Types I and II kerogen, compared to the Nyalau Formation which is dominated by Types II-III and III kerogen. On the other hand, good oil/gas generating potential is anticipated from the coals and carbargilite/coaly shales of the Nyalau Formation, based on their liptinite-rich nature and the predominance of n-alkene/alkane doublets and aromatic compounds. Samples from Madbi and Nyalau Formations were characterized by a variety of biomarker parameters based on GC and GC-MS techniques. Distribution of organic facies parameters such as Tm/Ts, Pr/Ph, pristane/n-C_{17}, phytane/n-C_{18}, oleanane/C_{30} hopane ratios and abundance of regular sterane (C_{27}, C_{28} and C_{29}) appear to reflect variations in depositional conditions and/or source input. Although there is a mixture of land-derived and marine-derived organic matter in both sediments, the depositional conditions of these formations can be distinguished based on these biomarker parameters, whereby the Madbi shales display organic facies characteristics of suboxic marine depositional conditions while the Nyalau sediments are consistent with paralic deposition within suboxic to oxic conditions, typical of terrestrial coastal plain environments.

Keywords: petroleum source rocks, biomarker parameters, organic facies, marine condition, coastal terrestrial environments

INTRODUCTION

This study highlights the distinct differences that are observed in the organic geochemical characteristics of marine and terrestrial-derived sediments and the petroleum generating potential of these distinct source rock types. Environment-related organic geochemical characteristics, commonly referred to as organic facies (e.g. Huc, 1990; Thomas *et al*., 1985), define the quality of source rocks present in a basin, whether they are capable of generating oil or gas, and if they are mature enough to have expelled hydrocarbons (Hunt, 1996; Tissot & Welte, 1984). The depositional setting in which source rocks were deposited is also of interest to petroleum explorationists, whereby for this purpose molecular-level organic facies parameters based on biomarker compounds have been extensively applied as reviewed by Peters *et al*. (2005).

The main objectives of this study are to evaluate and compare the oil-generating potential of source rock intervals within the Madbi Formation in the Masila Basin, Yemen with those of the Nyalau Formation of Sarawak, Malaysia, as well as to relate their organic matter source input with depositional conditions. The main applications of such a comparison being that (a) an understanding of depositional setting can provide some important indications of the quality of potential source rocks that may be present, and (b) in situations where only oil is available then considerable detail regarding the source rock can be ascertained by study of the biomarkers present in the oil. This study is based on organic geochemical and petrographic characteristics of borehole cutting samples from the Madbi Formation and of outcrop samples from the Nyalau Formation.

GEOLOGICAL BACKGROUND

Masila Basin was chosen for this study because it contains one of the most attractive and productive oilfields in the Republic of Yemen. The basin is located within the Hadramawt region of East Central Yemen (Figure 1). The origin and stratigraphy of the basin has been studied by many authors (e.g. Hatitham & Nani, 1990; Bosence *et al*., 1996; Putnam *et al*., 1997; Beydoun *et al*., 1998; Beydoun & As-Saruri, 1998; Watchorn *et al*., 1998; Cheng *et al*., 1999). The basin consists of a Mesozoic and Cenozoic sequence

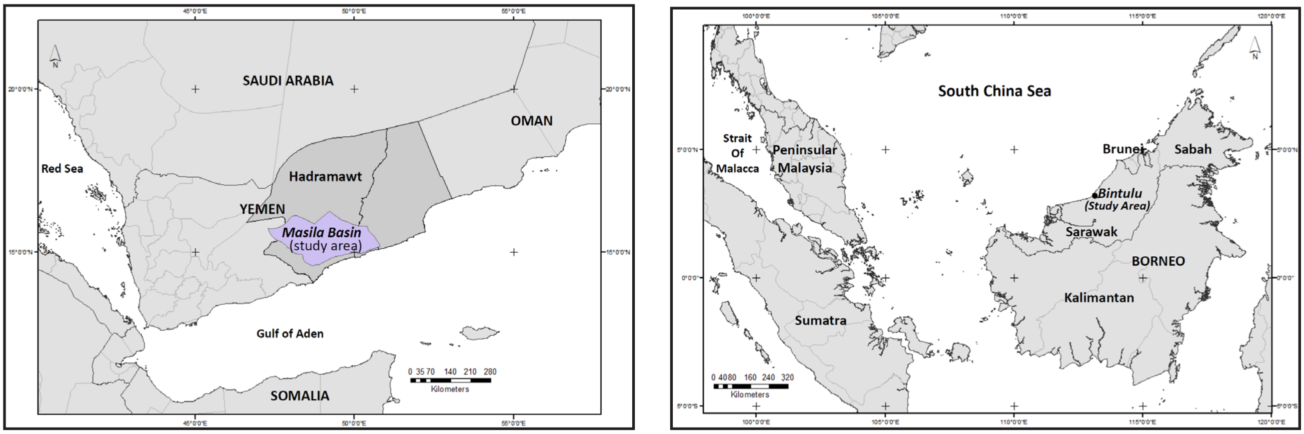

Figure 1: Location map of the study are in the Masila Basin, Yemen (left) and Bintulu, Sarawak, Malaysia (right).

unconformably overlying Pre-Cambrian basement (Figure 2a). The Jurassic interval is composed mainly of carbonates that were deposited in a shallow marine environment. However, important clastic intervals of shales and sandstone also occur (Beydoun *et al*., 1998). The Madbi Formation is of Upper Jurassic age and is part of the Amran Group (Beydoun *et al*., 1998). The Madbi Formation is generally composed of porous lime grainstone to argillaceous lime mudstone. The lithofacies of this formation reflects an open marine environment of deposition. The Madbi is subdivided into two members. The lower member is commonly an argillaceous lime with a basal sand, and forms a good reservoir in some oilfields of the Masila Basin. The upper member of this formation is composed of laminated organic rich shales, which is a prolific source rock in the Masila Basin (Mills, 1992; Hakimi *et al*., 2010).

In marked contrast, the Nyalau Formation is of Oligocene-Middle Miocene age (Liechti *et al*., 1960) and is an entirely clastic sequence deposited in a lower coastal plain setting in the Sarawak Basin of Northwest Borneo (Figure 1, right). Around Bintulu area, the Nyalau Formation is mainly composed of fine to coarse grained sandstone, often calcareous, alternating with shale and claystone all of which are predominantly terrestrial-derived. Coal seams are also common (e.g. Abdullah, 1997; Wan Hasiah, 1999; Shushan, 2006). The Nyalau Formation can be divided into two members, namely the Biban sandstone Member and the Kakus Member (Lee, *et al*., 2004). The base of Nyalau Formation is distinguished as the Biban sandstone Member and it is composed of consolidated, well bedded sandstone with subordinate sandy shale and shale (Shushan, 2006). The Kakus Member is the upper part of the Nyalau Formation and its top generally represents an erosional surface and probably marks the end of infill and cessation of deposition (Kho, 1968). It contains massive sandstone, laminated clays, brackish shales and lignites, deposited in inter-distributary deltaic environments (Shushan, 2006).

Selected outcrop samples of the Nyalau Formation were studied. The Nyalau Formation is of particular importance as it is an onshore equivalent of Cycles I and II in the Balingian Province of onshore Sarawak (Figure 2b) which is known to contain important source and reservoir rocks for oil and gas (Du Bois, 1985; Mazlan & Abolins, 1999; Wan Hasiah, 1999; Amir Hakif *et al.,* 2013).

SAMPLES AND METHODS

In this study, a total of sixteen samples (6 from Madbi and 10 from Nyalau) were analysed. The Madbi samples are well cuttings while the Nyalau samples are from outcrop exposures. Much care was taken during sampling of these Nyalau samples to avoid contamination and weathered outcrops.

Samples for petrographic study were prepared by mounting whole rock fragments in slow-setting polyester (Serifix) resin mixed with resin hardener and allowed to set. They were then ground flat on a diamond lap and subsequently polished on silicon carbide paper of different grades using isopropyl-alcohol as lubricant for organic-rich sediments and water for coal samples. Finally, the samples were polished to a highly reflecting surface using progressively finer alumina powder (5/20, 3/50 and Gamma). Measurements of mean random vitrinite reflectance (%Ro) were carried out using a x50 magnification oil immersion objective. Petrographic studies, consisting of quantitative analyses of vitrinite, liptinite, and inertinite maceral groups and mineral matter (Figures 3 and 4: Table 2), were performed with a Leica microscope using a combination of reflected white light and ultraviolet (UV) light fluorescence modes.

Bitumen extraction, gas chromatography (GC) and gas chromatography–mass spectrometry (GC-MS) analyses were conducted on eight samples (four from Madbi and four from Nyalau) of various lithologies. Samples were extracted in a Soxhlet apparatus for 72 hours using an azeotropic mixture of dichloromethane and methanol (93:7). Extracts were then separated into saturated hydrocarbon, aromatic hydrocarbon and NSO-asphaltene-compound fractions by liquid column chromatography. Saturated fractions were dissolved in hexane and analysed using a gas chromatograph. An FID detector was used for GC analysis (using HP-5MS column, temperature programmed from 40–300°C at a rate of 4°C/minute, and then held for 30 minutes at 300°C).

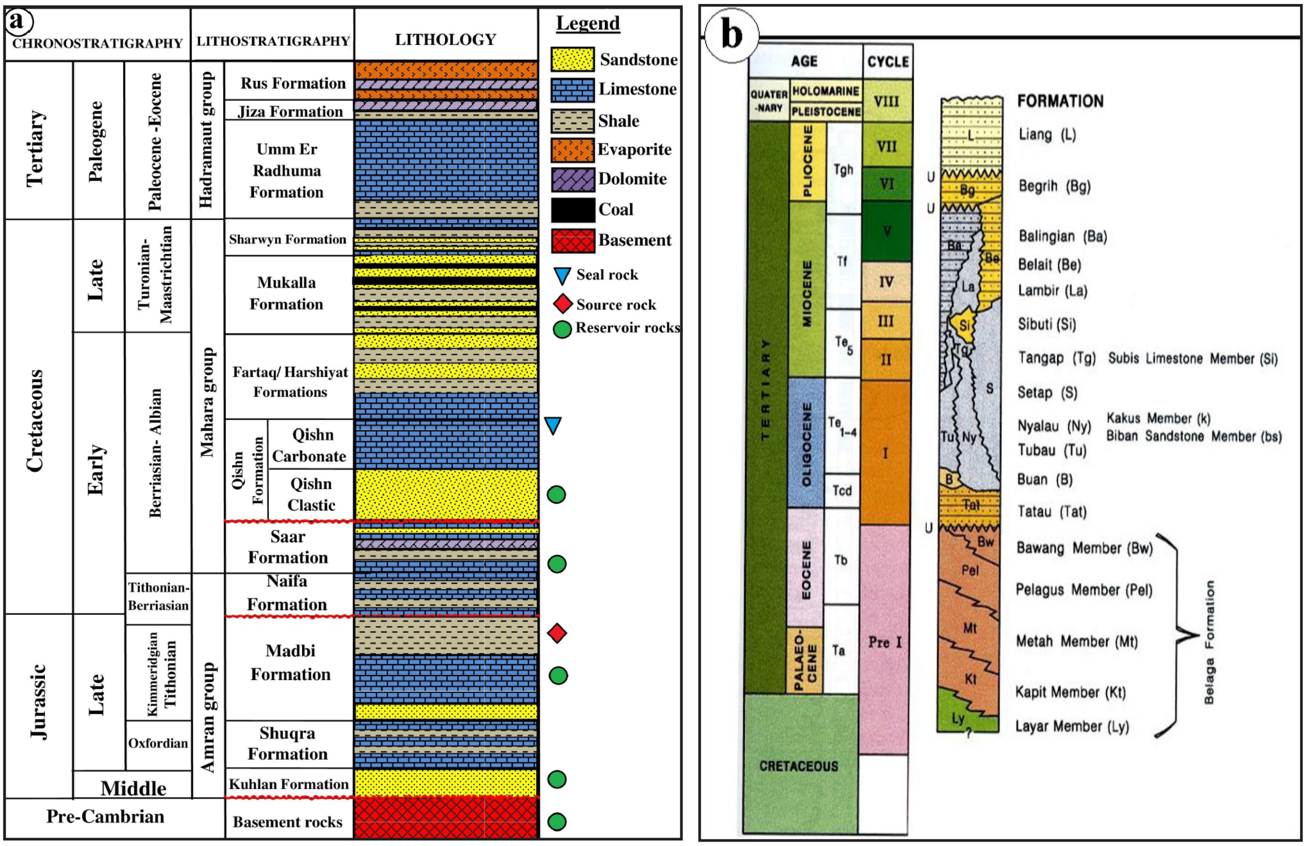

Figure 2: (a) Generalized stratigraphic column for the Masila Basin (after As-Saruri *et al*., 2010) and (b) the onshore Nyalau Formation of offshore stratigraphic equivalent in cycle I-II of Balingian-province (after Mohd Idrus & Redzuan, 1999).

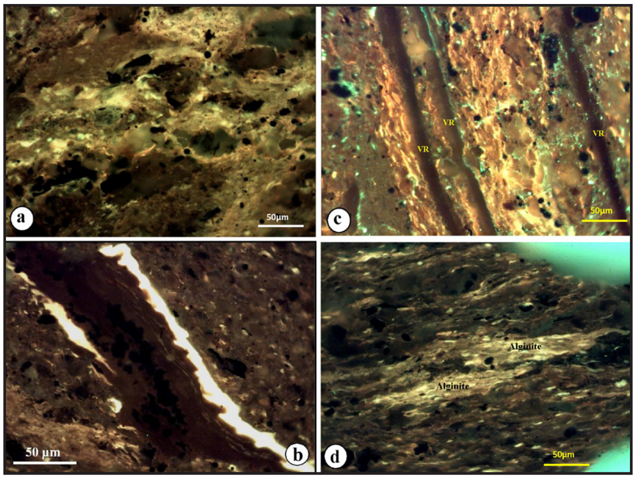

Figure 3: Photomicrographs of a shale sample from the Upper Jurassic Madbi Formation, under oil immersion, UV light examination: (a) Alginite occurs as bright fluorescing laminae and amorphous cluster; (b) large size alginite fluorescing bright whitish-yellow; (c) High concentration of fluorescing sporinite and liptodetrinite associated with vitrinite (VR); (d) High concentration of intense fluorescing bands of alginite.

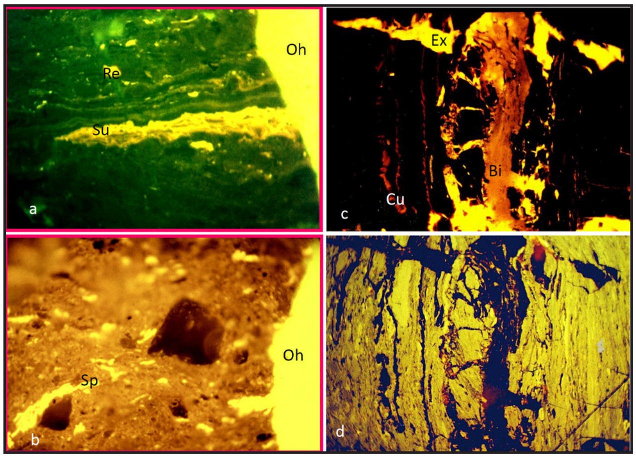

Figure 4: Photomicrographs of macerals from the Nyalau Formation of Sarawak Basin, under oil immersion, reflected white light and UV light: (a) Bright yellow fluorescing resinite (Re), suberinite (Su) and oil haze (Oh); (b) Oil haze (Oh) and Sporinite (Sp) associated with liptodetrinite in vaguely fluorescing bituminite matrix; (c) Intense yellow fluorescing exsudatinite (Ex) associated with dull orange fluorescing bituminite (Bi); (d) Same view as (c) under reflected white light; field of view =0.25 mm.

GC-MS analysis was performed on a V 5975B MSD mass spectrometer with a gas chromatograph attached directly to the ion source (70eV ionization voltage, 100 milliamps filament emission current, 230°C interface temperature). For the analysis of biomarkers, the fragmentograms for triterpanes (m/z 191) and for steranes (m/z 217) were recorded as shown in Figures 7 and 8, respectively. Compounds were identified based on retention time and with reference to previously published work (e.g. Philp, 1985; Peters *et al*., 2005).

Table 1: Geochemical data for the studied samples.

Samples no.	Lithology	TOC Wt.%	Pyrolysis data						Extracts			
			S_1	S_2	S_3	HI	OI	T_{max}	CPI	Pr/Ph	Pr/C_{17}	Ph/C_{18}
Madbi Formation, Masila Basin												
MDB-1	Shale	3.78	1.68	22.95	0.79	607	21	433	------	------	------	------
MDB-2	Shale	2.40	2.22	13.90	0.96	588	41	433	0.99	2.29	1.37	0.68
MDB-3	Shale	3.60	1.64	15.22	1.34	450	37	430	1.06	2.04	1.00	0.54
MDB-4	Shale	3.89	1.24	10.67	0.79	586	43	430	1.00	1.96	1.65	0.65
MDB-5	Shale	6.96	5.90	52.32	1.17	752	17	438	0.98	1.94	1.52	0.82
MDB-6	Shale	3.23	2.60	17.08	0.74	529	23	436	1.01	1.87	1.58	0.83
Nyalau Formation, Sarawak												
NYA -1	Shale	2.41	0.17	1.71	0.09	71.0	4.0	434.7	1.00	2.40	3.90	1.00
NYA -2	Carbonaceous shale-mud	2.22	0.05	1.88	0.13	85.0	6.0	437.0	1.20	4.90	4.90	1.40
NYA -3	Carbonaceous shale	1.25	0.06	1.01	0.08	81.0	6.0	453.0	1.10	3.30	3.10	0.70
NYA -4	Coal- silt, mud	2.71	0.11	2.34	0.05	86.0	2.0	434.5	------	------	------	------
NYA -5	Carbonaceous sandstone	0.48	0.03	0.36	0.03	75.0	6.0	445.0	------	------	------	------
NYA -6	Coal	80.85	7.53	351.4	1.14	435	1.0	430.0	1.04	6.46	9.55	2.75
NYA -7	Coaly shale (Carbargilite)	10.23	1.37	30.19	0.15	295	1.0	435.5	1.70	3.30	-----	1.70
NYA -8	Sandstone laminated with shale	1.26	0.05	0.81	0.02	64.0	2.0	455.0	------	------	------	------
NYA -9	Coal	76.2	9.96	392.8	0.83	516	1.0	435.0	------	------	------	------
NYA -10	Coal	90.5	4.58	394.8	1.43	413	1.0	432.0	------	------	------	------

TOC: Total organic Carbon, wt. %

S1: Volatile hydrocarbon (HC) content, mg HC/ g rock

S2: Remaining HC generative potential, mg HC/ g rock

S3: carbon dioxide content, mg CO_2/ g rock

HI: Hydrogen Index = S2x 100 / TOC, mg HC/ g TOC

OI: Oxygen Index = S3x 100 / TOC, mg CO_2/ g TOC

Tmax: The maximum temperature of S2 peak

Pr: Pristane

Ph: Phytane

CPI: Carbon Preference Index $((2(C_{23}+C_{25}+C_{27}+C_{29})/[C_{22}+2(C_{24}+C_{26}+C_{28})+C_{30}])$.

Table 2: Summary of petrological data for studied samples from the Madbi Formation, Masila Basin and Nyalau Formation, Sarawak.

Sample no.	%Ro	Macerals analysis (%)				
		Vitrinite	Liptinite	Inertinite	Mineral Matter	Common liptinitic content
Madbi Formation, Masila Basin, Yemen						
MDB-1	0.80	4.00	60.00	Trace	36.00	Alginite Liptodetrinite, Cutinite, Sporinite
MDB-2	0.74	3.00	45.00	Trace	52.00	Alginite Liptodetrinite, Cutinite, Sporinite
MDB-3	------	5.00	55.00	Trace	40.00	Alginite Liptodetrinite, Cutinite, Resinite
MDB-4	0.79	5.00	60.00	Trace	35.00	Alginite Liptodetrinite, Cutinite
MDB-5	0.81	3.00	60.00	Trace	37.00	Alginite Liptodetrinite, Cutinite
MDB-6	0.88	3.00	60.00	Trace	37.00	Alginite Liptodetrinite, Cutinite, Sporinite
Nyalau Formation, Sarawak, Malaysia						
NYA-1	0.57	30.00	12.00	0.00	58.00	Sporinite, Exsudatinite, Liptodetrinite
NYA -2	0.63	45.00	12.00	1.00	42.00	Sporinite, Liptodetrinite, Bituminite, Resinite
NYA -4	0.57	73.50	10.60	3.80	12.10	Sporinite, Suberinite, Resinite, Liptodetrinite, Cutinite
NYA -5	0.62	35.00	11.00	1.00	53.00	Sporinite, Resinite, Liptodetrinite, Bituminite
NYA -6	0.60	55.00	30.00	3.00	12.00	Sporinite, Suberinite, Resinite, Liptodetrinite, Cutinite
NYA -7	0.58	25.00	30.00	1.00	44.00	Sporinite, Exsudatinite, Liptodetrinite
NYA -9	0.50	40.00	35.00	1.00	24.00	Sporinite, Suberinite, Resinite, Liptodetrinite, Cutinite, Exsudatinite
NYA -10	0.62	45.00	26.00	2.00	27.00	Sporinite, Bituminite, Exsudatinite, Resinite, Liptodetrinite, Cutinite

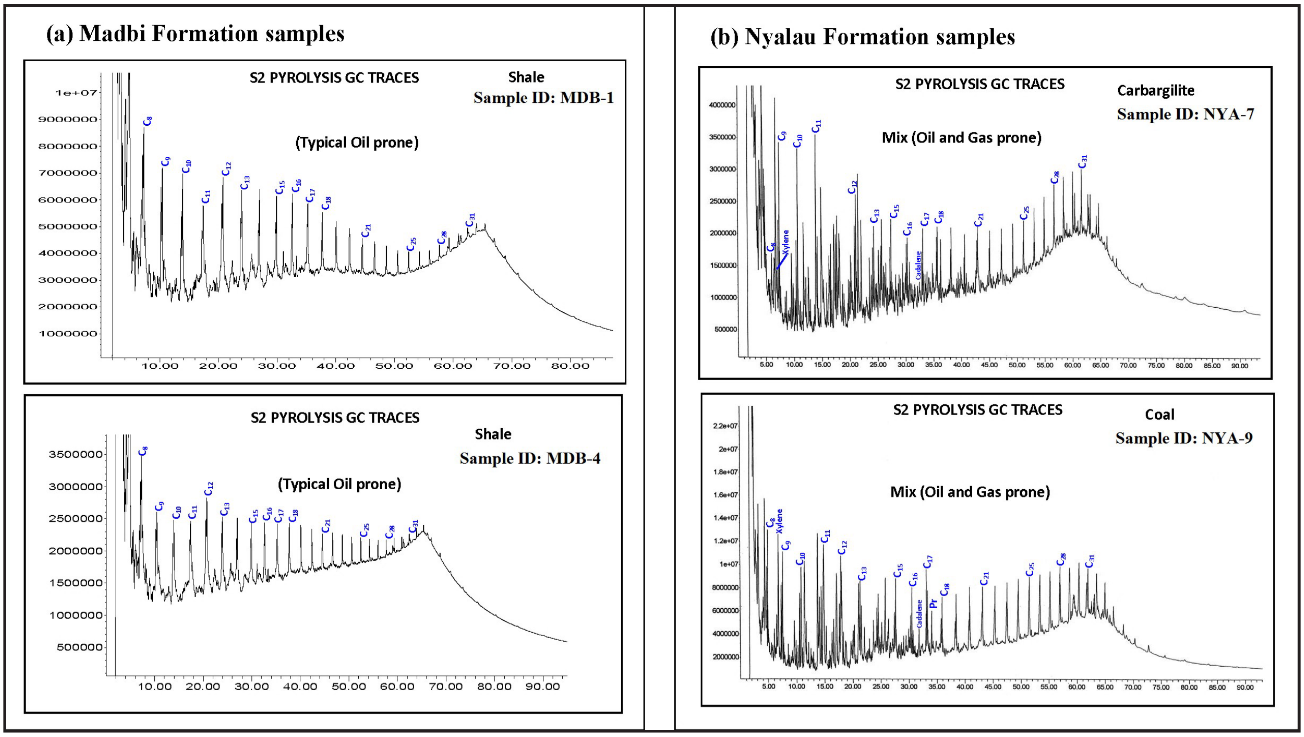

Figure 5: Py-GC pyrograms of coal and carbargillite of Nyalau Formation (right) and shale samples from Madbi Formation (left).

Pyrolysis-GC analysis was also performed (on extracted samples) at 600°C using a Frontier Lab Pyrolyser System coupled to an inert (quartz and Ultra Alloy-5, 30m, 0.25mm internal diameter) column fitted to an Agilent gas chromatograph (GC). The pyrolysis products passed through the GC column over a temperature range of 300°C to 600°C at 25°C/min. The pyrograms are as shown in Figure 5.

Rock-Eval pyrolysis and TOC analyses as described by Tissot & Welte (1984) were also performed. The results are shown in Table 1.

RESULTS AND DISCUSSION

Total organic carbon and Rock-Eval pyrolysis

The total organic carbon (TOC) and Rock-Eval pyrolysis analyses were performed on sixteen samples (six samples from the Madbi Formation and ten samples from Nyalau Formation). Total organic carbon (TOC wt. %) was carried out to determine the amount of organic matter. Most of the Madbi shale samples contain high total organic carbon (2.40 – 6.96 wt%, Table 1) having an average of approximately 4.0 wt%.

The average TOC values of the Nyalau coals, carbargilite and shales are 80, 10 and 2 wt%, respectively (Table 1). Coals and coaly sediments are not present in the Madbi Formation. Although this is obviously an important distinction between the two formations, the comparison here of the amount of the organic matter content will only be for the argillaceous samples. The TOC content of the Nyalau Formation shales and sandstones are distinctly lower compared to the Madbi Formation shale samples (average TOC 2 wt% versus 4 wt%).

Based on the Rock-Eval pyrolysis analysis, free hydrocarbons (S_1) in the rock and the amount of hydrocarbons generated from thermal breakdown of kerogen (S_2) and CO_2 (S_3) expelled from pyrolysis of kerogen are measured (see Table 1). In addition, the T_{max} value, which represents the temperature at the point where the S_2 peak is at its maximum i.e. where maximum hydrocarbon generation occurred (Espitalié, 1985) is also determined. Shale samples from Madbi Formation show Hydrogen Index values in the range of 450-752 mg HC/g TOC (average of 601 mg HC/g TOC) and are generally higher than those of Nyalau samples (71-516 mg HC/g TOC; average of 294 mg HC/g TOC). Therefore, the remaining potential of the Madbi and Nyalau sediments is generally good to excellent. Tmax values of the analysed samples from Madbi and Nyalau formations are in the range of 430-453°C, indicative of early to mid mature oil generation window.

Maceral/kerogen assemblage

Kerogen typing carried out on whole rock samples revealed that the Madbi shales consist predominantly of a mixture of Type I and II kerogen (Figure 3). In the Mabdi shales samples studied here, the Type I kerogen consists predominantly of bright yellow fluorescing alginite (Figure 3a and d) while the Type II kerogen mainly consists of liptinitic constituents, particularly cutinite, sporinite and liptodetrinite (Figure 3b-c). The maceral composition of the coals, carbargilite and organic rich sediments of the Nyalau Formation are displayed in Figure 4. Vitrinite, generally classified as Type III kerogen, is the most abundant maceral in the studied coal and coaly samples of the Nyalau

Formation (11.0%-73.5%; Table 2). All samples of the Nyalau Formation, however, contain significant amount of Type II liptinite macerals (10.6%-35.0%; Table 2). The most common liptinitic constituents are sporinite, suberinite, resinite, liptodetrinite, cutinite and exsudatinite (Figure 4). The microscopic features and origin of all these liptinite macerals have been previously described by Abdullah (1997) and Wan Hasiah (1999).

The dominance of oil-prone alginite in Madbi Formation samples and the high abundance of vitrinitic contituents with significance presence of oil-prone liptinitic contstituents in the Nyalau Formation samples as observed microscopically is supported by the pyrolysis gas chromatography analysis. This is discussed in the following section.

Pyrolysis gas chromatography (Py-GC)

Generally, the pyrolysis products of most Types I and II kerogen will extend out to the high molecular weight range compounds. Type I kerogen will produce abundant long-chain n-alkene/n-alkane doublets as shown by the prominent peaks in their pyrograms in the > C_{15} range, whereas Type II kerogen produces more naphthenic compounds represented by the large unresolved hump of material in the same (> C_{15}) molecular weight range (Dembicki, 2009). In contrast, with Type III kerogen the bulk of the pyrolysis product is confined to the low molecular weight (< C_{10}) range (Dembicki, 2009). These established observations of pyrolysis products support the general understanding of Type I/II kerogens being oil-prone and Type III being predominantly gas prone.

In this study, the Py-GC pyrograms of the coals, carbargilite and shale samples of the Nyalau Formation are dominated by *n*-alkene/alkane doublets that extend beyond nC_{30} (> C_{15}) indicative of the aliphatic-rich, and with significant aromatic compounds, suggesting a mixed oil and gas potential for these sediments (Figure 5b). This is therefore in agreement with the microscopical observation described above. Meanwhile, in the Madbi shale samples, the pyrograms are also dominated by *n*-alkene/alkane doublets that extend beyond nC_{30} (> C_{15}) (Figure 5a), hence in accordance with the high alginite content that was observed petrographically. However, contrary to the Nyalau pyrograms, the Madbi shale pyrograms are lacking in the aromatic compounds thus indicative of a predominantly oil-prone nature of the Madbi shales.

Biomarker distributions

n-Alkanes and isoprenoids

The gas chromatography (GC) analysis was performed on the saturated hydrocarbon fraction of eight samples, four from the Madbi Formation and four from Nyalau Formation. The gas chromatograms of three samples from each formations are as shown in Figure 6. The saturate gas chromatograms of the Madbi samples (Figure 6a) display a unimodal distribution of the *n*-alkanes with CPI values of close to 1.0 and show a high relative abundance of medium molecular weight n-alkanes in the nC_{15} - nC_{19} range suggesting that the organic matter was predominantly derived from marine algal material (e.g. Tissot *et al*., 1978; Brooks *et al*., 1992). On the other hand, the Nyalau samples, display a broader range of *n*-alkane maxima compared to the Mabdi Formation of n-C_{23} to n-C_{33} (high molecular weight) and possess relatively higher CPI values than the Madbi samples (Figure 6b). This is indicative of a significant terrestrial higher plant input as previously reported e.g. Powell & McKirdy (1973), Tissot *et al*. (1978), Tegelaar *et al*. (1989) and Abdullah (2003).

In summary, the two sets of GC distributions are therefore distinctly different, with the Madbi samples displaying a strongly unimodal distribution skewed to the medium molecular weight range, whereas the Nyalau samples display a less distinct unimodal distribution but skewed to the higher molecular weight range. As such the Nyalau Formation samples are distinctly more waxy than the Madbi samples.

Acyclic isoprenoids occur in significant amount in the Madbi Formation and in the Nyalau Formation samples. Pristane concentration is always higher than n-C_{17} in all of the analyzed samples. Phytane concentration is also higher than n-C_{18} in the Nyalau Formation samples, thus giving distinctively high pristane/ n-C_{17} and phytane/n-C_{18} ratios of 3.10-9.83 and 0.70-2.75, respectively. In the Madbi Formation samples, the phytane concentration is always lower than n-C_{18} thus giving a relatively lower phytane/n-C_{18} ratio (see Table 1). Comparatively lower values for the

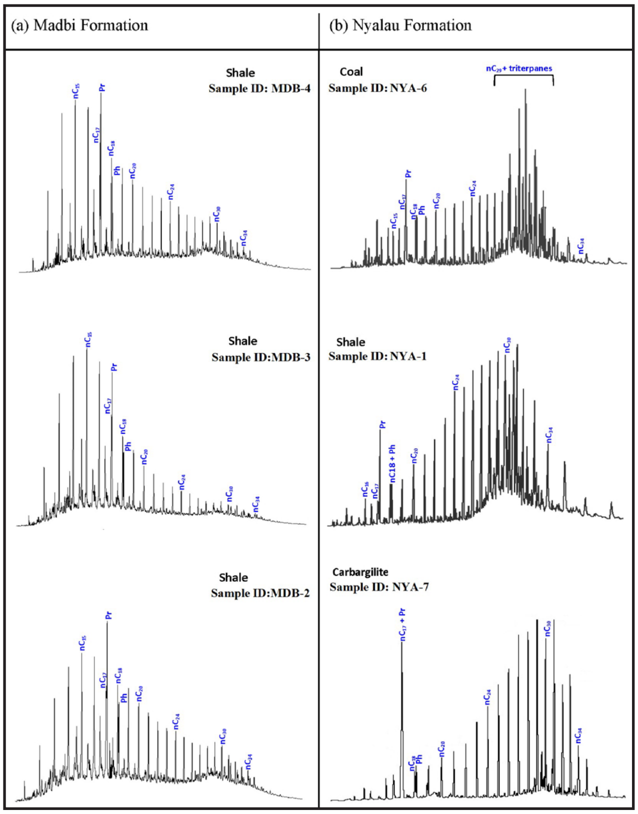

Figure 6: Gas chromatograms of saturated hydrocarbons of the Madbi Formation (left) and Nyalau Formation (right).

Pr/ n-C_{17} ratios (1.00-1.58) were displayed by the Madbi shale samples, which generally revealed relatively lower amounts of acyclic isoprenoids than the Nyalau Formation samples. The Pr/Ph ratios of the Nyalau sediments are in the range of 2.40-6.46 which suggest these sediments were deposited under a varying oxic to suboxic conditions (Powell & McKirdy, 1973; Didyk *et al*., 1978; Tissot & Welte, 1984; Large & Gize, 1996), while the Pr/Ph ratios of the Madbi shales range from 1.87 to 2.29 which suggest these samples were deposited under a consistent suboxic conditions (Powell, 1988; Amane & Hideki, 1997; Sarmiento & Rangel, 2004; Basant *et al*., 2005).

Triterpanes and steranes

The distributions of steranes and triterpanes are commonly studied using GC-MS by monitoring the ions m/z 217 and m/z 191 (Figures 7 and 8). There are also other important biomarker groups such as tricyclic terpanes (m/z 191), tetracyclic terpanes (m/z 191) and diasteranes (m/z 217) (Seifert & Moldowan, 1979; Peters *et al*., 2005). In this study, common biomarker parameters calculated based on m/z 191 and m/z 217 are as shown in Table 3. The assignment of the peaks of steranes and triterpanes labeled in Figures 7 and 8 are as listed in Table 4. Individual components were identified by comparison of their retention times and mass spectra with previously published literature (e.g. Philp, 1985; Peters & Moldowan, 1993; Peters *et al*., 2005).

The m/z 191 mass fragmentograms of the saturated hydrocarbon fractions (Figure 7) of all the studied samples analysed (Madbi and Nyalau) show high abundance of hopanes. The C_{29}/C_{30} hopane ratio is consistently low (< 0.5) in all of the Madbi samples. This is in contrast to the Nyalau Formation in which the C_{29}/C_{30} hopane ratio is higher and variable. A significant difference in the Madbi and Nyalau organofacies is seen in the C_{27}Tm/C_{27}Ts ratio. In all the Madbi samples the ratio is close to one whereas in the Nyalau samples the Tm abundance can be very high resulting in very high C_{27}Tm/C_{27}Ts ratios is the Nyalau samples (Table 3).

The relative abundance of oleanane is distinctly different between the two sample sets. The Nyalau samples display a variable but relatively high oleanane index (oleanane/ C_{30} hopane) in the range of 0.09-2.04. In marked contrast, oleanane is absent in all of the Madbi samples. Oleanane was widely reported to represent a higher plant input in rocks of Cretaceous or younger age (e.g. Ekweozor & Telnaes, 1990).

Tricyclic terpanes, are known to be associated with both marine and lacustrine source facies (e.g. Aquino *et al*., 1983; Philp, 1985), and can also be used to differentiate the two sets of samples in this study. In the Madbi shales the tricyclic terpanes, although not abundant, are clearly present whereas they are essentially absent in the Nyalau samples (Figure 7a). The C_{25} / C_{26} Tricyclic ratio is > 1.0 which is

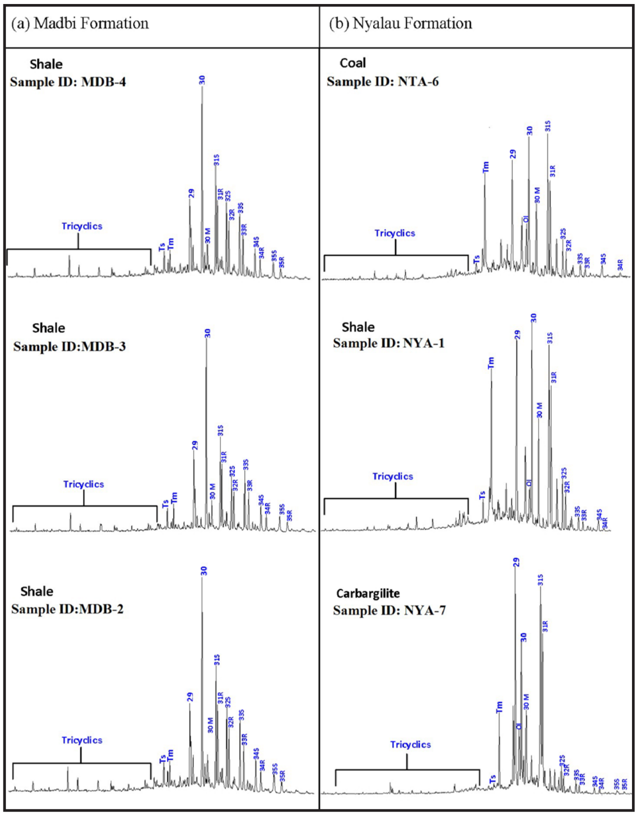

Figure 7: The m/z 191 mass fragmentograms of saturated hydrocarbon fractions of the Madbi shales (left) and Nyalau sediments (right).

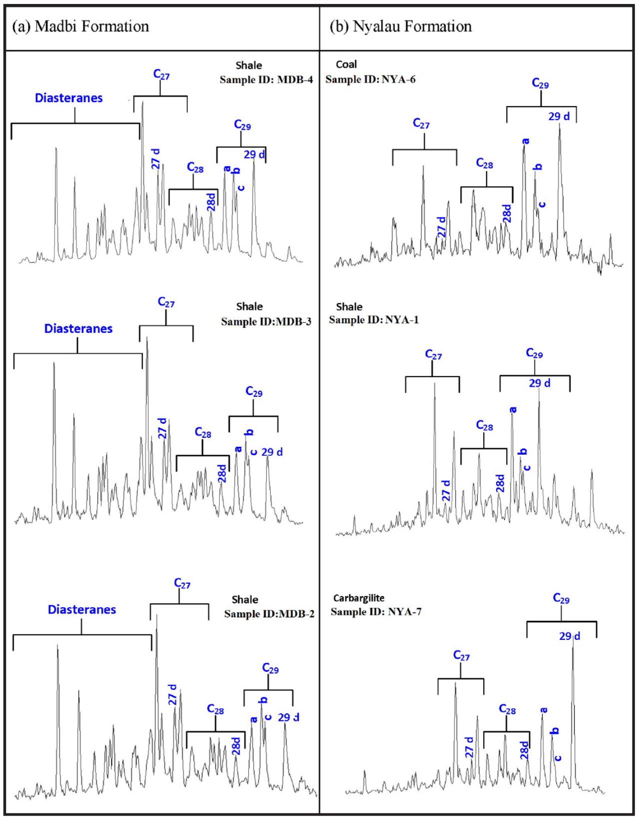

Figure 8: The m/z 217 mass fragmentograms of saturated hydrocarbon fractions of the Madbi shales (left) and Nyalau sediments (right).

Table 3: Biomarker parameters calculated from m/z 191 and m/z 217 mass fragmentograms.

Samples no.	Triterpanes (m/z 191)				Steranes (m/z 217)					
					C_{29} 20S/ (20S+20R)	C_{29} ββ/(ββ+αα)	Regular steranes			C_{29}/C_{27}
	C_{32} 22S/ (22S+22R)	Tm/Ts	OI/C_{30} H	C_{30}Mo/ C_{30}H			C_{27}	C_{28}	C_{29}	
Madbi Formation, Masila Basin										
MDB-3	0.60	0.94	OI absent	0.12	0.48	0.56	40.0	20.0	40.0	1.00
MDB-4	0.58	1.06	OI absent	0.15	0.47	0.53	42.0	20.0	38.0	0.91
MDB-5	0.60	1.31	OI absent	0.13	0.47	0.55	41.0	20.0	39.0	1.30
MDB-6	0.59	0.86	OI absent	0.13	0.48	0.56	40.0	18.0	42.0	1.10
Nyalau Formation, Sarawak										
NYA -1	0.60	9.00	0.53	0.26	0.46	0.44	14.4	35.9	49.7	3.50
NYA -2	0.60	26.00	2.04	0.16	0.35	0.36	16.2	12.4	71.4	4.40
NYA -7	0.62	8.00	0.13	0.14	0.36	0.37	22.9	8.30	68.8	1.80
NYA -9	0.57	4.50	0.09	0.22	0.43	0.40	32.0	8.0	60.0	1.60

(See Table 4 for Peak assignments)

Table 4: Peak assignments for alkane hydrocarbons in the gas chromatograms of aliphatic fractions in the m/z 191 and 217 mass fragmentograms.

	Compound	Abbreviation
(I) peak No.		
T_s	18α(H),22,29,30-trisnorneohopane	T_s
T_m	17α(H),22,29,30-trisnorhopane	T_m
29	17α,21β(H)-nor-hopane	C_{29} hop
OI	1817α(H)- oleanane	Oleanane Index
30	17α,21β(H)-hopane	hopane
3M	17 β,21α (H)-Moretane	C_{30}Mor
31S	17α,21β(H)-homohopane (22S)	C_{31}(22S)
31R	17α,21β(H)-homohopane (22R)	C_{31}(22R)
32S	17α,21β(H)-homohopane (22S)	C_{32}(22S)
32R	17α,21β(H)-homohopane (22R)	C_{32}(22R)
33S	17α,21β(H)-homohopane (22S)	C_{33}(22S)
33R	17α,21β(H)-homohopane (22R)	C_{33}(22R)
34S	17α,21β(H)-homohopane (22S)	C_{34}(22S)
34R	17α,21β(H)-homohopane (22R)	C_{34}(22R)
35S	17α,21β(H)-homohopane (22S)	C_{35}(22S)
35R	17α,21β(H)-homohopane (22R)	C_{35}(22R)
(II) peak No.		
a	5α,14α(H), 17α(H)-steranes 20S	ααα20S
b	5α,14β(H), 17β(H)-steranes 20R	αββ20R
c	5α,14β(H), 17β(H)-steranes 20S	αββ20S
d	5α,14α(H), 17α(H)-steranes 20R	ααα20R

in accordance with the Madbi shales being marine-derived supporting the facies interpretation of Beydoun *et al*. (1998). An absence of tricyclic terpanes in the coal-bearing Nyalau Formation is consistent with the predominantly terrigenous environment of deposition suggested by Liechti (1960).

The regular sterane and the rearranged sterane (diasterane) distributions of the two sample sets, as demonstrated by the m/z 217 mass fragmentograms, are fundamentally different. The Madbi Formation is characterized by a high relative abundance of diasteranes in contrast to the Nyalau Formation in which diasteranes are relatively low (Figure 8 and Table 3). Also noteworthy is that the abundance of C_{27} / C_{28} / C_{29} regular steranes for Madbi shale follow the order C_{27}>C_{29}>C_{28}, whilst the distributions of regular steranes in the Nyalau Formation is in the order C_{29}>C_{27}>C_{28}. This manifests itself in the C_{29}/ C_{27} sterane ratios (Table 3).

Biomarker ratios commonly applied as thermal maturity parameter (e.g. Peters & Moldowan, 1993; Peters *et al*., 2005) calculated in this study are the C_{29} 20S/(20S+20R) and the C_{29} αββ(αββ+ααα) sterane ratios (Table 3). The significance of these parameters will be discussed in Section 4.6.

Organic matter input and depositional environment setting

Biomarker distributions provide information about organic facies and depositional environment (Waples & Machihara, 1991; Hunt, 1996; Peters *et al*., 2005). In this study, organic facies and depositional environments were examined with the use of sterane and triterpane distributions and diagnostic ratios (Table 3). The organic facies parameters described in the previous section, such as Pr/Ph, Pristane/n-C_{17}, Phytane/n-C_{18}, Tm/Ts, oleanane/ C_{30} hopane ratios and abundance of regular steranes (C_{27}, C_{28} and C_{29}), were used to reflect variation in depositional conditions and/or source input for the Madbi and Nyalau formations, respectively. The Pristane/Phytane (Pr/Ph) ratio is one of the most commonly used geochemical parameters and has been widely invoked as an indicator of the redox conditions in the depositional environment and source of organic matter (Powell & McKirdy, 1973; Didyk *et al*., 1978; Tissot & Welte, 1984; Large & Gize, 1996). Organic matter originating predominantly from terrestrial plants are expected to have high Pr/Ph >3.0 (oxidizing conditions), whilst low values of Pr/Ph (<0.6) indicate anoxic conditions; values between about 1.0 and 3.0 suggest intermediate conditions (sub-oxic conditions) (Powell, 1988). The sub-oxic condition of deposition of the Madbi samples is suggested by the low Pr/Ph ratios of 1.87-2.29, while sub-oxic to oxic conditions of the Nyalau samples are as reflected by the relatively higher Pr/Ph ratios (2.40-6.46). Based on Pr/Ph, Pr/*n*-C_{17} and Ph/*n*-C_{18} ratios of the samples analysed, the Madbi sediments were considered to have been deposited in relatively more reducing conditions compared to the Nyalau sediments (Figure 9).

The relative distribution of C_{27}, C_{28} and C_{29} steranes is graphically represented in the form of a ternary diagram in Figure 10. Source input of organic matter undoubtedly has a strong influence on the sterane distribution of rock extracts (and oils) although there has been considerable debate as to the environmental significance of the distribution (e.g. Volkman, 1986; 1988). The original classification of Huang & Meinschein (1979) related C_{27} steranes to strong algal influence and C_{29} steranes to strong higher plant input (Figure 10). Based on this ternary diagram, the Madbi shales are considered to be deposited in an open marine environment whilst the Nyalau sediments were deposited in terrestrial environments (Figure 10). This biomarker-based environmental interpretation is fully supported by petrographic observation indicating that, for the Madbi samples at least, the Huang & Meinschein (1979) classification holds true. The microscopically observed presence of algae in the Madbi samples (Figure 3: a and d) correlating with abundant cholestane (C_{27}). In addition, tricyclic terpanes are present in the Madbi shales but are virtually absent in the Nyalau sediments. Tricyclic compounds are often associated with algal input (e.g. Simoneit, 1986; Aquino Neto *et al*., 1989; Volkman *et al*., 1989).

Tm and Ts are well known to be influenced by both maturation and type of organic matter (e.g. Seifert & Moldowan, 1978; Moldowan *et al*., 1986). All the analysed samples (Madbi and Nyalau) contain a mixture of land and marine-derived organic matter, albeit in different proportions, and subsequently the Tm/Ts ratio in the studied samples appears to be strongly influenced by source input rather than maturity (see Table 3). The terrestrial-derived Nyalau sediments have higher Tm/Ts ratios compared to the Madbi marine shale samples (Table 3) a characteristic typical for the Nyalau Formation coal as previously reported by Wan Hasiah (1999), thus also in agreement with Huang and Meinschein diagram (Figure 10) in relation to depositional environment and source input.

Thermal maturity of organic matter

For an organic-rich rock to become an effective source rock, it must reach a maturity level sufficient to generate hydrocarbons (Tissot & Welte, 1984). Several data and parameters have been used here to evaluate the level of organic maturity of the analysed Madbi and Nyalau samples; these include vitrinite reflectance data (%Ro), Carbon Preference Index (CPI) and pentacyclic triterpanes as well as sterane isomerisation ratios (Peters & Moldowan, 1993; Peters *et al.,* 2005). With increasing maturity, the proportion of 20S sterane increases as some of the 20R molecules changes configuration, and eventually an equilibrium between the two forms is attained at approximately equal proportion of 20R and 20S configurations (Mackenzie, 1984). The αββ steranes become predominant over ααα form, reaching a ratio of about 3:1 in mature samples, however this ratio is known to be affected by diagenetic conitions (e.g. ten Haven *et al*., 1986; Peakman & Maxwell, 1988).

The vitrinite reflectance values for the Madbi shales ranging from 0.74 to 0.88%, indicating that they are thermally mature and have entered the early mature to peak mature stage. The Nyalau sediments possess vitrinite reflectance values in the range 0.50 - 0.66% suggesting the samples are less mature than the Madbi Formation samples and are in the early mature stage for hydrocarbon generation (Table 2).

The thermal maturity was also evaluated using their biomarker distribution as shown in Table 3. As discussed above, the Madbi samples are considered higher maturity based on vitrinite reflectance. In general, the C_{32} 22S/22S+22R homohopane ratio increases from zero to about 0.6 at equilibrium (Seifert & Moldowan, 1986) during maturation. Values in the range of 0.50 to 0.54 have barely entered oil generation, whereas ratios from 0.57 up to 0.62

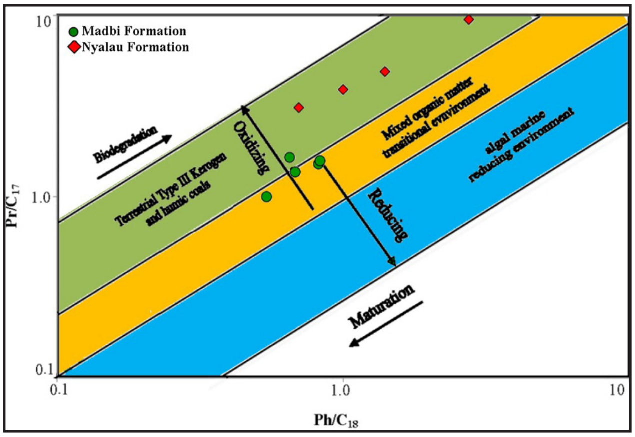

Figure 9: Log plot of the ratios of phytane to *n*-C_{18} alkane (Ph/*n*-C_{18}) versus pristane to *n*-C_{17} alkane (Pr/*n*-C_{17}) of the analysed samples (modified after Hunt, 1996; Shanmugam, 1985).

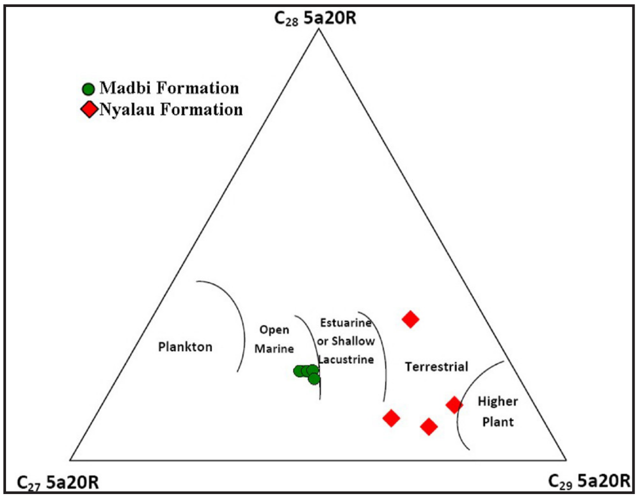

Figure 10: Relationship between sterane compositions, source input and depositional environment for the analysed Madbi and Nyalau formations samples (modified after Huang & Meinschein, 1979).

indicate that the oil window has been reached. In this study, most of the extracted samples of Madbi and Nyalau have values of C_{32} 22S/22S+22R from 0.57 to 0.62 which suggest that these samples are thermally mature and have entered the oil window. The 20S/(20S+20R) and ββ/(ββ+αα) C_{29} sterane ratios of the Madbi Shale samples also suggest a similar interpretation (Table 3), whereby most of the studied samples yielded higher values that are approaching the end point for 20S/(20S+20R) and ββ/(ββ+αα) C_{29} sterane parameters. In contrast, for the Nyalau samples, the 20S/(20S+20R) and ββ/(ββ+αα) C_{29} sterane ratios are relatively lower (0.35-0.46 and 0.36-0.44, respectively; Table 3), and these low values indicate that sterane transformations have not reached equilibrium and hence the samples of Nyalau Formation are not fully mature for hydrocarbon generation. Their offshore equivalents (Cycles I and II; Figure 2) on the other hand, are mature and have been identified as source rocks for the oil and gas within the Balingian Province of the Sarawak Basin (Mazlan & Abolins, 1999).

These maturity observations of the Nyalau and Madbi formations suggest that the sterane-based maturity parameters provide a better representation of thermal maturity compared to hopane-based parameters.

Hydrocarbon generation potential

Rock-Eval/SRA pyrolysis and total organic carbon analyses suggest very good petroleum generative potential for the marine Madbi shales and the terrestrial Nyalau coal/ carbargilite samples. These samples have high total organic carbon content (>2.0%) and pyrolysis S_2 yields are generally > 2 mg HC/g rock. Therefore, these samples (except for one sample) possess good to excellent potential for hydrocarbon generation (Figure 11). Samples that contain predominantly type III kerogen (by petrographic observation) and Hydrogen Index < 200 mg HC/g TOC (by pyrolysis) would be expected to generate gas and condensate, while samples visually dominated by type I and II kerogen and with Hydrogen Index >300 mg HC/g TOC would generate mainly oil (Peters & Cassa, 1994; Tissot & Welte, 1984). Observing these guidelines, the Madbi shales would be expected to generate oil and the Nyalau coals/carbargilite would be predominantly mixed oil and gas prone. On the other hand, the analysed clastic sediments (shales and sandstone) of the Nyalau Formation, do not possess oil-generating potential, although they may possess gas-generating potential.

The organic petrographic study of the Nyalau organic rich samples revealed the common occurrences of oil haze and an early phase of petroleum like material (exsudatinite) being generated in the Nyalau Formation coaly, Type III-dominated source rocks (Figure 4). The original precursors of this exuded material was identified to be predominately from the maceral suberinite and bituminite, which has been regarded as the most oil-prone organic constituents of the Nyalau Formation coals and carbargilites as previously described by Abdullah (1997; 2003) and Wan Hasiah (1999). The oil prone nature of these macerals is supported by the Py-GC (S_2) pyrograms as shown in Figure 5b, which are dominated by n-alkene/alkane doublets extending beyond n-C_{30}. These demonstrate a good case in which organic petrological and organic geochemical techniques being complement of each other.

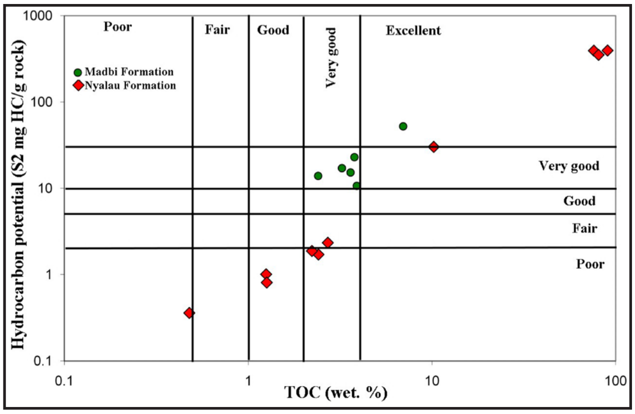

Figure 11: Pyrolysis S_2 versus total organic carbon (TOC) plot showing generative source rock potential for the analysed Madbi and Nyalau samples.

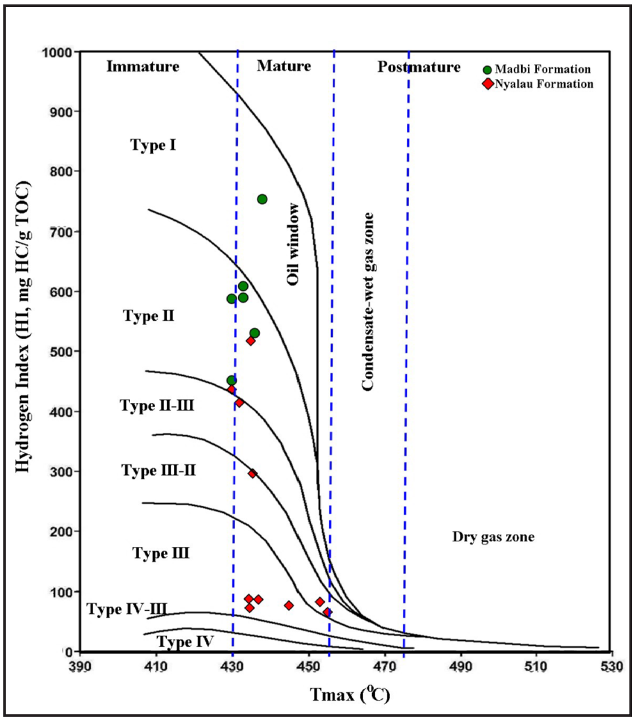

Figure 12: Plot of Hydrogen Index (HI) versus pyrolysis Tmax for the analysed samples showing kerogen quality and thermal maturity stages.

CONCLUSION

The organic geochemical and organic petrological approach performed in this study has clearly differentiated between sediments deposited in marine and coastal depositional setting. Utilizing the GC and GC-MS fingerprints it was possible to derive a clear interpretation of the depositional environment and organic matter source input. This has been achieved from the acyclic isoprenoid, triterpanes and sterane biomarker distributions. Biomarker parameters such as Tm/Ts, Pr/Ph, pristane/n-C$_{17}$, phytane/n-C$_{18}$, oleanane/C$_{30}$ hopane ratios and abundance of regular steranes reflect variation in depositional conditions and/or source input. Based on these parameters, the Madbi shale samples are considered to have been deposited in more reducing conditions (suboxic) than the Nyalau sediments (oxic to suboxic). These parameters are therefore in accordance with the Madbi shales been deposited in a marine environment dominated by algal-derived organic matter while the Nyalau sediments been deposited in a coastal depositional setting dominated by terrestrial-derived organic matter. In terms of oil generation potential, the Madbi shales and coaly sediments of the Nyalau formation have reasonably good liquid hydrocarbon generating potential (oil or condensate), owing to their liptinite-rich nature, HI > 300 mg HC/g TOC and high abundance of n-alkene/n-alkane doublets indicated by Py-GC pyrograms.

At early to peak oil window maturity, the Madbi shales are expected to be better source rocks for oil as suggested from their relatively higher abundance of Type I and II kerogen and high TOC compared to the organic rich sediments of the Nyalau Formation. Good oil-generating potential is also anticipated from the coals and carbargilites of the Nyalau Formation owing to their exinite-rich nature of Type II kerogen and the occurrence of exsudatinite indicative of the presence of early generated hydrocarbons. The oil-prone nature of the coaly sediments is supported by the dominance of n-alkene/alkane doublets in the Py-GC (S$_2$) traces of these terrestrially-derived sediments. The oil-prone nature of the marine Madbi shales is predominantly attributed to the dominance of Type I and II kerogens, as supported by a distinctively different S$_2$ pyrograms when compared to the coaly Type III-dominated Nyalau Formation sediments. This study also demonstrate the complementary nature between organic petrological and organic geochemical methods, and thus both these techniques ought to be applied in studies related to petroleum source rock evaluation and in depositional environment interpretation.

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