

An assessment of paleodepositional environment and maturity of organic matter in sediments of the Setap Shale and Belait formations in West Sabah, East Malaysia by organic geochemical methods

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Abstract The black shale samples collected from two Neogene formations in the Klias Peninsula area, West Sabah, have been assessed and characterized in details by gas chromatography, gas chromatography-mass spectrometry and a variety of organic geochemical parameters. The aims of this study are to describe the characteristics of organic matter of these sediments in terms of source/type of the organic matter, assess its thermal maturity and paleoenvironment of deposition, based primarily on biomarker distributions. The results of both formations do not reveal significant differences within the rock extracts. The gas chromatograms of the saturated hydrocarbon fractions of the Setap Shale and the Belait formations displayed monomodal n-alkane distributions and nearly identical regular sterane compositions with a predominance of C_{27} regular steranes. These are consistent with open marine depositional environments dominated by marine biological matter. Another related feature of these rock extracts is the presence of a high relative abundance of gammacerane, indicating anoxic marine hypersaline source depositional environment. The relatively high abundance of common land plant-derived biomarkers, such as bicadinanes and oleananes, is a clear indication of a major terrigenous input to the source of the extractable organic matter. The predominance of oleanane biomarkers in both formations is indicative of angiospermis input and Tertiary source rocks. The high C_{29}/C_{30} hopane ratios, moderate development of C_{33} - C_{35} hopanes, high abundance of tricyclic terpanes and a slight predominance of C_{27} regular sterane over C_{28} and C_{29} steranes are characteristic features tending to suggest a significant marine influence on these source rocks, thereby suggesting a mixed source input. The $22S/(22S+22R)C_{32}$ hopane ratio has reached equilibrium, and this is supported by the high maturity level as indicated by the $22S/22SC_{31-33}$ extended hopane ratios and $20S/(20S+20R)C_{29}$ regular steranes ratios.

Key words geochemical characterization; Sabah, Malaysia; Neogene; organic-rich sediments; angiospermis; gammacerane

1 Introduction

The sediments under investigation constitute part of the Setap Shale and Belait formations outcropping in the Klias area, onshore West Sabah (Fig. 1). The geochemical parameters discussed in this study are based on the average values of six outcrop samples collected from four different localities along the main road cuts from Kuala Penyu to Menumbuk comprising shales and mudstones representative of the Belait and Setap Shale formations, which are commonly carbonaceous and of Tertiary age. The geology of Sabah is complicated, reflecting the extreme kinematics com-

plexity of the South China Sea region (Tan and Lamy, 1990). Sabah is located at the intersection of two mega-tectonic trends: the NE-NW Borneo Trend and the NW-SE Sulu Trend.

The main geological elements of Sabah as demonstrated by Leong (1999), can be categorized into four phases: pre-Tertiary core, metamorphic and igneous complex; Cretaceous-Early Paleogene, ophiolites and indurated deep marine sediments; Paleogene Basins, mainly N-S elongated basins containing deep marine sediments to the east and west of the Cretaceous-Early Paleogene deep marine sediment; and Neogene Basins.

The pre-Tertiary core of onshore Sabah is repre-

sented by the crystalline basement of a metamorphic and igneous complex (amphibolites, hornblende schists and gneisses; granodiorites, tonalities, granites). The Cretaceous-Early paleogene sequence in general, is represented by the chert-splite Formation, the Madai-Baturong limestone, and the Sapulut, Trusmadi and East Crocker formations. The Chert-Splite Formation consists of extrusive igneous rocks associated with radiolarian cherts, limestones and clastics.

The radiolarian cherts are Early Cretaceous in age and the limestone is Late Cretaceous in age (Basir Jasin and Sanudin Tahir, 1988; Basir Jasin, 1992). The Madai-Baturong limestone consists of algal, oolitic and massive limestones of Cretaceous age (Adams and Kirk, 1962). The Sapulut, Trusmadi and East Crocker formations consist of thick turbiditic clastics, partly metamorphosed to slates and phyllites.

The Belait Formation consists of sandstones, shales, coals and conglomerates. Wilson (1964) interpreted the Belait Formation as fluvial to shallow marine sediments.

The Setap Shale and Belait formations were deposited during Miocene time (Mazlan et al., 1999), and are of particular importance as they are considered to contain fair hydrocarbon-generating potential. The Setap Shale Formation consists entirely of soft, dark gray shales and mudstones deposited in marine environments.

The aim of this paper is to discuss the use of biomarkers and organic geochemical parameters of these sediments in order to assess the sources/types of organic matter and to understand their thermal maturity and establish the paleoenvironment of deposition.

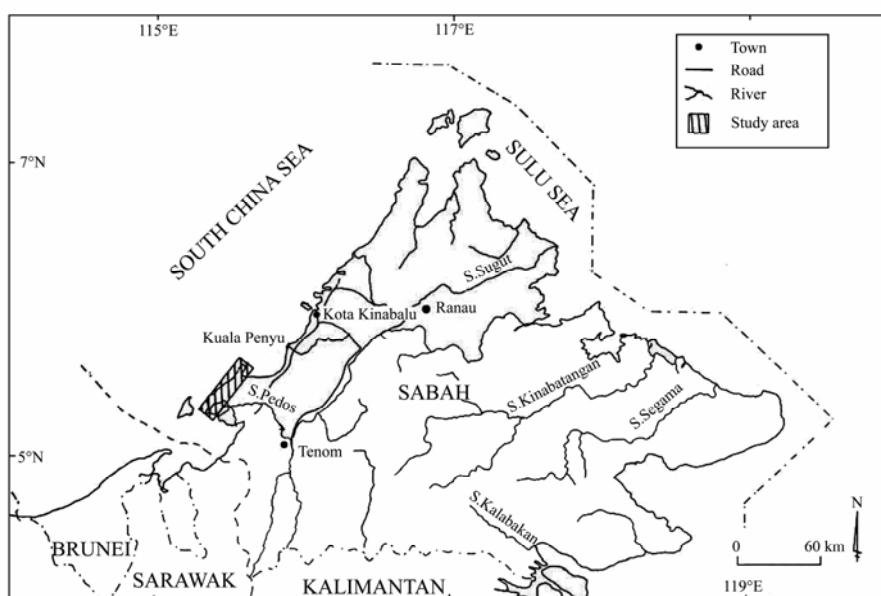


Fig. 1. The location of the study area.

2 Samples and experiments

In the Klias Peninsula area, six outcrop samples were collected from four different localities throughout the depositional area of Tertiary time, in West Sabah, East Malaysia, in order to obtain a broad representation of the regional organic geochemistry. The first group of samples denoted by Be₁, Be₂ and Be₈ refer to the black shale units of the Belait Formation were collected around the Kuala Penyu area. The second group of samples denoted by Se₁, Se₂ and Se₇ refer to the black shales of the Setap Shale Formation were collected from three scattered road cuts along the main road from Kuala Penyu to Menumbuk. Each sample was crushed to rock powder by a crushing machine to reduce the rock aggregate down to

monomineralic particles by vibrating a steel disk mill (Herzog, type: Hsm 100 A).

Approximately about 80–90 g of powdered black shale sample (<200 mesh) were extracted using a dichloromethane/methanol mixture (93:7) for 77 hours in a Soxhlet apparatus. Elemental sulphur was removed by activated copper during extraction. The extract was subjected to column chromatography on silica gel 60 (MERCK 0.063–0.200 mm), supported by a 2–3 cm layer of alumina. The saturated fraction was eluted by light petroleum (100 mL). The saturated hydrocarbon fractions were analyzed by gas chromatography (GC) and gas chromatography-mass spectrometry (GC-MS), equipped with a HP-5 capillary column (30 m×0.32 mm i.d., 0.25 μm film thickness) and helium was used as a carrier gas. The oven temperature was held at 40°C for 6 minutes and pro-

grammed from 40 to 320°C at 20 minutes. The mass spectrometer conditions were electron ionization at 70 eV with an ion source temperature at 250°C. Ions monitored were m/z 191 for triterpanes and m/z 217 for steranes. Identification of biomarkers was performed by comparing GC retention time data and mass spectra with the published data (Peters and Moldowan, 1993). Relative concentrations in gas chromatograms and mass fragmentations were determined based on the peak heights.

Table 1. Biomarker parameters for the rock extracts occurring in the study area

Parameter	Setap Shale Formation			Belait Forma		
	Sample No.			Sample No.		
	Se ₁	Se ₂	Se ₇	Be ₁	Be ₇	Be ₈
TOC	1.17	1.33	0.63	0.52	0.58	0.74
Pr/Ph	0.8	6.0	0.5	0.4	0.2	0.2
Pr/ <i>n</i> C ₁₇	0.5	11.7	0.9	0.8	0.7	0.5
Ph/ <i>n</i> C ₁₈	0.5	0.9	0.5	1.0	3.0	3.0
<i>n</i> -alkane max.	C ₂₅	C ₂₄	C ₂₅	C ₂₅	C ₂₅	C ₂₅
CPI	0.91	0.90	0.95	0.98	0.94	0.95
<i>n</i> C ₂₄ - <i>n</i> C ₃₅						
Tm/Ts	14	7.0	14	15	15	13
Ol/C ₃₀ hop.	0.45	1.26	0.30	0.11	0.13	0.14
C ₃₀ m/C ₃₀ hop.	0.29	0.36	0.13	0.18	0.17	0.13
C ₂₉ /C ₃₀ hopane	0.60	1.16	0.62	0.64	0.67	0.73
C ₂₄ tetra/C ₃₀ hop	0.16	0.19	0.53	1.00	0.88	0.39
C ₂₃ tri/C ₃₀ hop.	0.10	0.12	0.29	1.71	0.65	0.34
C ₂₄ tetra/C ₂₃ tri.	1.70	1.55	1.87	0.58	1.37	1.15
C ₃₂ hopane	0.53	0.57	0.57	0.57	0.55	0.64
22S/(22S+22R)						
C ₃₁ /22S/22R	1.26	1.25	1.29	1.25	1.29	1.42
C ₃₂ /22S/22R	1.27	1.33	1.33	1.33	1.29	1.50
C ₃₃ /22S/22R	1.50	1.33	1.50	1.33	1.30	1.60
C ₃₀ */C ₂₉ Ts	0.14	0.30	0.18	0.22	0.31	0.20
C ₃₅ /(22S+22R)/C ₃₄ (22S+22R)	0.9	0.8	0.7	0.9	n.d.	0.6
Gam./C ₃₀ hopane	0.09	0.04	0.03	0.09	0.08	0.06
C ₂₉ sterane						
20S/(20S+20R)	0.48	0.69	0.55	0.51	0.45	0.43
Sterane (%)						
C ₂₇	49	56	53	71	60	44
C ₂₈	18	21	14	12	12	14
C ₂₉	33	23	33	17	28	42

TOC. Total organic carbon; Pr/Ph. pristane/phytane; Pr/*n*C₁₇. pristane/normal-C₁₇; Ph/*n*C₁₈. phytane/normal-C₁₈; CPI₂₄₋₃₅. carbon preference index (after Bray and Evans, 1961); Tm. 17 α (H), 22, 29, 30-trisnorhopane; Ts. 18 α (H), 22, 29, 30-trisnorhopane; Ol/C₃₀. oleanane/C₃₀ hopane; C₃₀ m/C₃₀ hop. C₃₀ mortane/C₃₀ hopane; C₂₄ tetra/C₃₀ hop. C₂₄ tetracyclic terpanes/C₃₀ hopane; C₂₄ tetra/C₂₃ tri. C₂₄ tetracyclic terpanes/C₂₃ tricyclic terpanes; C₃₂ hopane 22S/(22S+22R). 17 α (H), 21 β (H)-bishomohopane (22S)/[17 α (H), 21 β (H)-bishomohopane (22S)+17 α (H), 21 β (H)-bishomohopane (22R)] of C₃₂ homohopane; C₂₃ tri./C₃₀ hop. C₂₃ tricyclic terpanes/C₃₀ hopane; 22S/22R of C₃₁₋₃₂₋₃₃. 17 α (H), 21 β (H)-bishomohopane (22S)/17 α (H), 21 β (H)-bishomohopane (22R) of C₃₁₋₃₂₋₃₃; C₃₀*/C₂₉ Ts. 17 α (H) diahopane/18 α (H)-norhopane; Gam./C₃₀ hopane. gammacerane/17 α (H), 21 β (H)-hopane; C₂₉ sterane 20S/(20S+20R). 5 α (H), 14 α (H), 17 α (H)-20S/[5 α (H), 14 α (H), 17 α (H)-20S+5 α (H), 14 α (H), 17 α (H)-20R] of C₂₉ sterane; C₂₉ sterane 20S/20R. 5 α (H), 14 α (H), 17 α (H)-20S/[5 α (H), 14 α (H), 17 α (H)-20R] sterane C₂₉; n.d. not determined.

3 Results and discussion

3.1 Total organic carbon (TOC)

The total organic carbon (TOC) contents (Table 1) exhibit a distinct variation of the abundance of organic matter present in these sediments. The Setap Shale samples possess a fair and rich total organic contents with TOC values ranging from 0.63 wt% to 1.33 wt% with an average of 1.04 wt%, whilst the Belait Formation samples have TOC values ranging from 0.52 wt% to 0.74 wt% with an average values of 0.60 wt%, which can be identified as poor and fair total organic contents (Peters 1986).

3.2 Biomarker distributions

Diagnostic biomarker ratios and indices are listed in Table 1. Representative gas chromatograms and mass fragmentograms (m/z 191) and (m/z 217) are in Figs. 2, 3 and 4 with peak identification in the m/z 191 mass fragmentograms listed in Table 2 and peaks in the m/z 217 fragmentograms identified in Table 3.

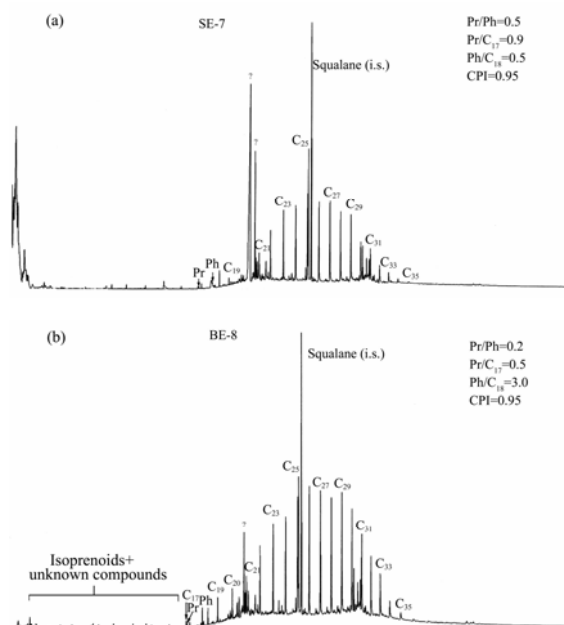


Fig. 2. Gas chromatograms of the saturated fraction from rock extracts of (a) Setap Shale Formation; (b) Belait Formation. Lower Pr/Ph ratio of <0.1 and CPI<0.1 suggest anoxicity depositional environment. i.s. Internal stander.

3.3 Normal alkane and acyclic isoprenoids

The distribution of saturated hydrocarbon fractions illustrated by the capillary gas chromatograms of six black shale samples from the Setap Shale and Belait formations is shown in Fig. 2. The GC traces of all the shales studied display a waxy appearance, being depleted in the *n*-C₁₀-*n*C₁₅ range and dominated by heavier members (*n*C₂₀₊). The chromatograms of these samples also clearly show a bimodal distribution of higher molecular weight alkanes.

The higher molecular weight *n*-alkanes reveal a

wide range of normal alkanes extending from nC_{10} to nC_{35} , and are maximized at peak nC_{25} . A number of samples show the presence of humps of unresolved complex hydrocarbon mixtures. The hump appears generally at around nC_{20} . The presence of hump is possibly indicative of biodegradation, thus accounting for the depletion of n-alkanes, in particular the lower-end member series.

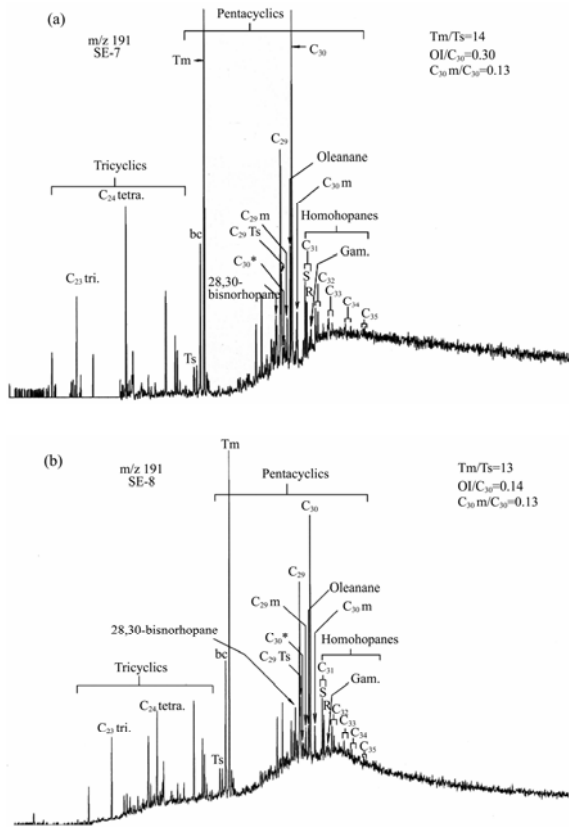


Fig. 3. Hopane (m/z 191) distributions from rock extracts of (a) Setap Shale; (b) Belait Formation.

The moderate molecular weight n -alkanes (nC_{10} - nC_{20}) and acyclic isoprenoids, such as pristane and phytane, occur in low concentrations in all the analyzed samples. The gas chromatograms display a smooth homologous series within the higher molecular weight n -alkane extending beyond nC_{30} , suggesting significant input of higher land plant organic matter into these sediments.

The fingerprints also show apparent evidence of slight biodegradation (i.e., partial removal of n -alkanes relative to entire alkane distribution) for most of the samples, being more affected around nC_{10} - nC_{20} . The presence of biodegradation could be related to the intrusion or mixing of oxygenated waters within these sediments due to prolonged surface exposures, which is a necessary condition if biodegradation had occurred.

Table 2. Peak assignments in the m/z 191 mass fragmentograms

C_{23} tri.	Tricyclic terpene
C_{24} tetra.	Tetracyclic terpene
C_{27} Tm	17 α (H), 22, 29, 30-trisnorhopane
C_{27} Ts	18 α (H), 22, 29, 30-trisnorhopane
bc	Trans-trans-trans-bicadinane
C_{27}	17 α (H), 21 β (H)-hopane
C_{29}	17 α (H), 21 β (H)-hopane
C_{29} Ts	18 α (H)-30-norhopane
C_{29} m	17 β (H), 21 α (H)-moretane
C_{30}	17 α (H)-diahopanes
OI	18 α (H)-oleanane
C_{30}	17 α (H), 21 β (H)-hopane
C_{30} m	17 β (H), 21 α (H)-moretane
C_{31} S	17 α (H), 21 β (H)-homohopane (22S)
C_{31} R	17 α (H), 21 β (H)-homohopane (22R)
Gam.	C_{30} gammacerane
C_{32} S	17 α (H), 21 β (H)-bishomohopane (22S)
C_{32} R	17 α (H), 21 β (H)-bishomohopane (22R)
C_{33} S	17 α (H), 21 β (H)-trishomohopane (22S)
C_{33} R	17 α (H), 21 β (H)-trishomohopane (22R)
C_{34} S	17 α (H), 21 β (H)-tetrakishomohopane (22S)
C_{34} R	17 α (H), 21 β (H)-tetrakishomohopane (22R)
C_{35} S	17 α (H), 21 β (H)-pentakishomohopane (22S)
C_{35} R	17 α (H), 21 β (H)-pentakishomohopane (22R)

Table 3. Peak assignments in the m/z 217 mass fragmentograms

Di (Diasterane)	C_{27} 13 β (H), 17 α (H)-diacholestane-20S
Di (Diasterane)	C_{27} 13 β (H), 17 α (H)-diacholestane-20R
C_{27} $\alpha\alpha\alpha$ S	5 α (H), 14 α (H), 17 α (H)-cholestane-20S
C_{27} $\alpha\alpha\alpha$ R	5 α (H), 14 β (H), 17 β (H)-cholestane-20S
C_{27} $\alpha\beta\beta$ R	5 α (H), 14 β (H), 17 β (H)-cholestane-20R
C_{27} $\alpha\beta\beta$ R	5 α (H), 14 α (H), 17 α (H)-cholestane-20R
C_{28} $\alpha\alpha\alpha$ S	5 α (H), 14 α (H), 17 α (H)-24-methylcholestane-20S
C_{28} $\alpha\beta\beta$ S	5 α (H), 14 β (H), 17 β (H)-24-methylcholestane-20S
C_{28} $\alpha\beta\beta$ R	5 α (H), 14 β (H), 17 β (H)-24-methylcholestane-20R
C_{28} $\alpha\alpha\alpha$ R	5 α (H), 14 α (H), 17 α (H)-24-methylcholestane-20R
C_{29} $\alpha\alpha\alpha$ S	5 α (H), 14 α (H), 17 α (H)-24-ethylcholestane-20S
C_{29} $\alpha\beta\beta$ S	5 α (H), 14 β (H), 17 β (H)-24-ethylcholestane-20S
C_{29} $\alpha\beta\beta$ R	5 α (H), 14 β (H), 17 β (H)-24-ethylcholestane-20R
C_{29} $\alpha\alpha\alpha$ R	5 α (H), 14 α (H), 17 α (H)-24-ethylcholestane-20R

3.4 Pr/Ph ratio

The isoprenoid alkanes pristane (Pr) and phytane (Ph) are present in all of the samples studied, usually as the major constituents of the isoprenoid alkane. Historically, the ratio of pristane to phytane has long been associated with the redox conditions of depositional environment (Brooks and Smith, 1969; Powell and Makriday, 1973). These early workers assumed that both pristane and phytane were derived from the phytol side chain of chlorophyll. More recently, it has been shown that pristane can be derived from α -tocopherol and phytane from the bis-phytyl ethers which occur in archeobacteria (Philip, 1994).

According to Cooper (1990), pristane and phytane are derived from the chlorophyll of higher plants, algae and photosynthetic bacteria and archaeobacteria. The existence of various precursors therefore indicates

that the Pr/Ph ratio should be used with care. In the samples studied here, pristane and phytane are present in low amounts, with Pr/Ph ratios less than 1.0, suggesting a reducing environment (anoxic condition).

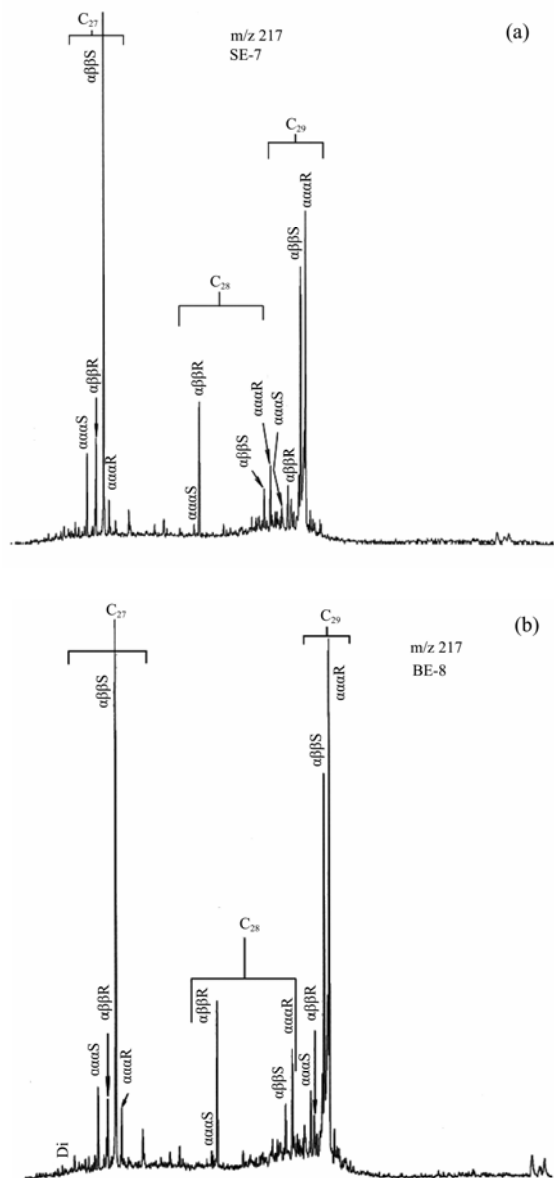


Fig. 4. Sterane (m/z 217) distributions from rock extracts of (a) Setap Shale; (b) Belait Formation, demonstrating similar source of organic matter and a predominance of C_{27} over C_{28} and C_{29} regular steranes, indicating major contribution from marine materials.

The samples Se_1 , Se_2 and Se_7 displayed Pr/Ph ratios of 0.8, 6.0 and 0.5, respectively, whilst the Be_1 , Be_7 and Be_8 illustrated Pr/Ph ratios of 0.4, 0.2 and 0.2, respectively. The relatively high Pr/Ph ratios in the samples of the Setap Shale Formation and the low Pr/Ph ratios in the samples of the Belait Formation are most likely to be associated with the source of organic matter, the effect of biodegradation and/or indicator of the extent of oxicity/anoxicity of the depositional

condition.

3.5 Carbon preference index (CPI)

The CPI value is influenced by the type of organic matter and by the maturity (Tissot and Welte, 1984). High CPI values represent an odd carbon number preference over even and occur in response to the chemistry in higher plants of surface waxes (an important precursor of waxy components in sedimentary organic matter). Hydrocarbons thought to be derived from coaly organic matter source contain carbon preference indices equal to or greater than one.

Moldowan et al. (1985) concluded that an odd carbon preference is characteristic of oils derived from source rocks deposited in non-marine depositional environments. In contrast, predominance of an even numbered n -alkane preference is commonly observed in bitumens and oils derived from carbonate or evaporite rocks (Palacas et al., 1984). If the total even and odd numbers of paraffins are equally abundant, the value of the (CPI) will be equal to one, as generally observed in highly mature samples (Tissot and Welte, 1984).

In these samples studied a slightly odd over even predominance of higher molecular weight n -alkanes (nC_{24} – nC_{35}) has been observed, producing CPI values <1.0 (Table 1). By the other meaning, the GC fingerprints revealed a noticeable odd over even predominance with CPI values of 0.91, 0.90 and 0.95 for samples Se_1 , Se_2 and Se_7 , respectively, whilst the samples Be_1 , Be_7 and Be_8 possess CPI values of 0.89, 0.94 and 0.95, respectively. The CPI values of <1.0 observed in most of the study samples is believed to be influenced by the type of organic matter and thermal maturity as all samples are known to possess a high maturity.

3.6 Tricyclic and tetracyclic terpanes (m/z 191)

Both tricyclic and tetracyclic terpanes are present in high abundance in all of the samples analyzed. The tricyclic terpanes are a useful series of biomarkers for different types of organic matter, maturity and depositional environments. In evaluating the tricyclic distributions in the black shales of the Setap Shale and Belait formations in West Sabah, the tricyclics apparently can be grouped into two end member types. One group member type is characterized by the pattern with a high C_{23} tricyclic terpane peak and the other with a high C_{24} tetracyclic terpane peak (Fig. 3).

The C_{24} is the major component in most of these samples with the exception of the sample (Be_1) only, which shows a dominance of C_{23} tricyclic terpane over C_{24} tetracyclic terpane. These features may indicate the presence of an algal kerogen component, in addition to higher plant input into these sediments.

3.7 Hopane

The m/z 191 chromatograms of the samples studied showed that the pattern of all samples belongs mostly to $17\alpha(H)$, $21\beta(H)$ hopanes series, with molecules ranging from C_{27} to C_{35} (Fig. 3). The C_{30} regular hopane is the most predominance member series followed by C_{29} norhopane and the ratios of C_{29}/C_{30} is in the range of 0.60 to 1.16 in the shales from the Setap Shale Formation, whilst the shales from the Belait Formation have a C_{29}/C_{30} ratios ranging from 0.64 to 0.73. The component $17\alpha(H)$ -trisorhopane (Tm) is dominant over its counterpart $18\alpha(H)$ -trisnorhopane (Ts) with Tm/Ts ratios ranging from 7.0 to 14 in the Setap Shale Formation, whilst in the case of the Belait Formation the Tm/Ts ratios varies from 5.0 to 7.0. The $17\beta(H)$, $21\alpha(H)$ series of molecules (moretanes) and the gammacerane are also present in varying abundance. The oleanane is present in all the study samples and identified as one peak representing $18\alpha(H)$ -oleanane and $18\beta(H)$ -oleanane. Biscadinanes (denoted, bc) are found in most of the black shales studied in different concentrations, all these features clearly suggest an input of organic matter derived from higher plants into these sediments. The extended hopanes which occur as stereoisomeric pairs, from C_{31} to C_{35} , commonly display a staircase distribution pattern (i.e., uniformly lower amount with increasing molecular weight). The C_{31} and higher homologues occur as 22S and 22R epimers.

3.8 Steranes and diasteranes m/z 217

Steranes commonly found in mature sediments and crude oils are derived via diagenesis from sterols which are widely dispersed in plants and microorganisms, with the C_{27} and C_{28} sterols being most abundant in marine organisms and the C_{29} sterols in higher plants (Gonzalez-vila, 1995).

The C_{27} , C_{28} and C_{29} steranes occur as 20S and 20R epimers. Steranes can be used as indicators of depositional environments. An abundance of C_{29} over C_{27} steranes would suggest a terrestrial source, whereas C_{27} steranes predominating over C_{29} steranes would indicate mainly a marine or aquatic source (Palacas et al., 1984). In this study the steranes are present at higher concentrations as tricyclic, tetracyclic and pentacyclic terpanes.

In the m/z 217 fragmentograms (Fig. 4) the C_{27} sterane is the most predominant member series followed by the C_{29} and C_{28} steranes which are lowest. The distribution of $C_{27}:C_{28}:C_{29}$ steranes (Table 1) occurs in relatively abundant quantities in nearly all the examined shales.

The rearranged steranes (diasteranes) are either absent or are present in minor or trace amounts in all

the analyzed samples. It has been proposed that the relative concentrations of the diasteranes reflect the presence of clay minerals and their ability to catalyze sterane rearrangement reactions. However, it has become clear that the oxicity/anoxicity of the depositional environment provides another explanation to the fate of the sterols. Highly anoxic environments will rapidly reduce the steranes, hence reducing the amount of steranes available for the rearrangement reactions, therefore, more oxic environments will leave more steranes unaffected and hence available for rearrangement (Philip, 1994).

It can be said that the absence or presence of the rearranged steranes in minor or trace amounts in shales of the Setap Shale and Belait formations possibly related to the limited occurrence of acid-catalyzed rearrangement of regular steranes and/or more closely reflects the oxicity/anoxicity environment rather than presence or absence of clay minerals (Moldowan et al., 1986).

3.9 Type/source of organic matter

The distribution of n -alkanes in crude oils can be used to indicate the source of organic matter (Han and Calvin, 1969; Volkman et al., 1981; Duan, 2000; Duan and Ma, 2001). The gas chromatograms of the saturated hydrocarbons display minor dissimilarities in features in the black shales from the Setap Shale and Belait formations.

The n -alkane chromatograms exhibit smooth homologous series of n -alkanes extending beyond n - C_{35} with the maxima at n - C_{25} , slight odd-even predominance and high relative concentrations of long chain n -alkanes. The waxy appearance of these samples suggests a significant contribution of organic matter input of land plant origin. The distribution of n -alkanes suggests that the study sediments have organic matter source from aquatic organisms and land plant organisms with the latter being slightly dominant.

The main feature that distinguishes the rock extracts in this study is the relative abundance of oleanane, a biological marker diagnostic of higher plants. It has been suggested as a marker for angiosperms (flowering plants) (Ekweozor et al., 1979; Moldowan et al., 1994).

Oleanane generally does not occur in rocks or oils prior to angiosperm diversification on land, which occurred during the Late Cretaceous. Oleanane/hopane ratios over 20% are characteristic of Tertiary source rocks and oils. On the other hand, oleanane can be absent from source rocks deposited far from angiosperm input. Applying this parameter to assess the angiosperm contribution to the source rocks of the Setap Shale and the Belait rock extracts shows that the

rock extracts for both formations with measurable oleanane concentrations were almost certainly generated from Tertiary sources (Moldowan et al., 1994), and therefore confirmed and supports the Tertiary age determination setting of these formations proposed by earlier workers. The high abundance of bicadinanes and oleanane in these rock extracts also suggest considerable terrigenous input.

The distribution of regular steranes has been used to determine the organic matter source of crude oils (Moldowan et al., 1985; Philip et al., 1991). The relative abundances of C_{27} , C_{28} and C_{29} regular steranes in the Setap Shale Formation are in the range of 49%–55%, 14%–21% and 22%–33%, respectively, whilst in the Belait Formation the relative abundances of C_{27} , C_{28} and C_{29} regular steranes are within the range of 44%–71%, 12%–14% and 17%–42%, respectively (Table 1). The values of the Belait Formation vary over a narrow range. These characteristics of the regular steranes further reflect that the organic materials in these samples originated from the similar organic source with slightly higher marine organic matter.

The distribution of regular steranes, shown in the ternary diagram (Fig. 5), reveals a preference of C_{27} steranes over C_{28} and C_{29} , a feature commonly seen in organic matter of marine origin. The high C_{29}/C_{30} hopane ratios and moderate development of C_{33} – C_{35} hopanes would also be in line with such a suggestion being characteristic of marine-source organic matter.

Aquino Neto et al. (1983) suggested a microbial origin for both tricyclic and tetracyclic terpanes series. Philp and Gilbert (1986), on the other hand, associated the high abundance of C_{24} tetracyclic terpanes with high input of terrestrially derived organic matter. The high abundance of C_{24} tetracyclic terpanes in the study samples could therefore be associated with higher land-plants, algae, or microbial sources.

The rock extracts of these shales generally display high concentrations of C_{23} tricyclic terpanes and therefore have relatively high C_{23} tricyclic terpanes/ C_{30} hopane ratios. The presence of tricyclic terpanes in sediment extracts and oils has been associated with the presence of marine influence by workers such as Aquino Neto et al. (1983) and Mello et al. (1988), and therefore supports the marine depositional setting of these formations as proposed by workers such as Wilson (1964) and Lee (1977).

The Pr/Ph ratios in most of the samples studied (except for sample Se₂) are within the range of 0.2–0.8 (Table 1), which seems to be diagnostic a highly reducing (anoxic) marine depositional environment (Alexander et al., 1997). The lower Pr/Ph ratios in the Setap Shale and Belait samples can be taken as evidence for the origin of the organic matter *in-situ*.

Pr/ n - C_{17} and Ph/ n - C_{18} ratios have been widely

used as indicators of source rock types, depositional environments and organic matter maturity (Connan and Cassou, 1980; Peters et al., 1999; Duan et al., 2006)

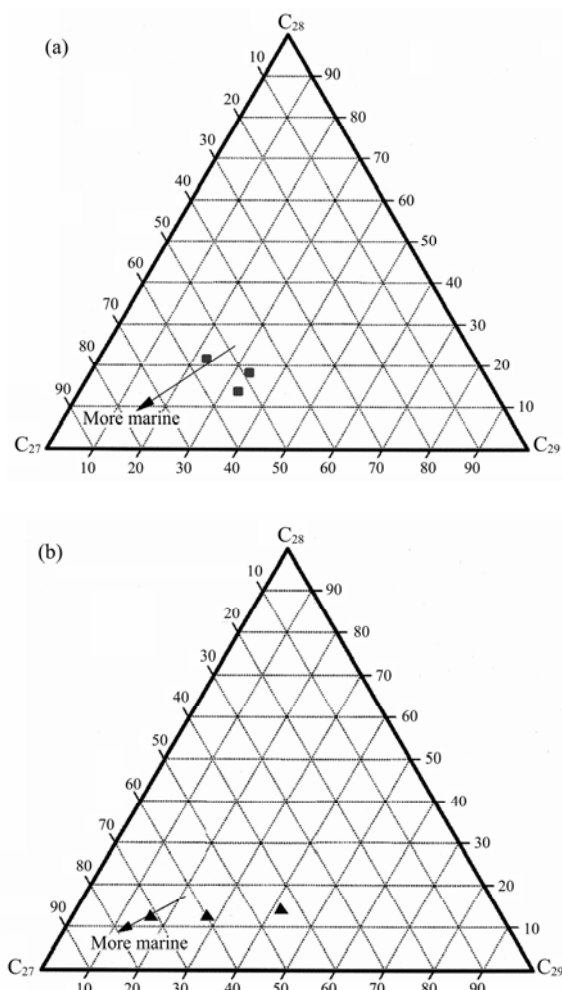


Fig. 5. The ternary diagram illustrating the relative distribution of C_{27} , C_{28} , and C_{29} regular steranes. It exhibits the predominance of C_{27} over C_{28} and C_{29} regular steranes, indicating major contribution from marine materials. (a) Setap Shale Formation; (b) Belait Formation.

The lower Pr/ n - C_{17} and Ph/ n - C_{18} ratios in most of the samples studied are probably caused by their maturity in addition to the source of organic matter. The difference in Pr/ n - C_{17} and Ph/ n - C_{18} ratios is also present among the samples studied. Generally, the Pr/ n - C_{17} and Ph/ n - C_{18} ratios decrease with increasing thermal maturity (Peters et al., 1999)

Based on the above discussed parameters, although differences between the lithologies within a particular formation could not be made, distinction can be made between the dominant types of organic matter that is present in the shales. It can be said that the Setap Shale and Belait formations seem to have received substantial amounts of land-source organic matter that had been transported into a marine deposi-

tional setting and was deposited in a photosynthetic organism-dominating environment (thereby suggesting a mixed source input).

3.10 Thermal maturity

Maturation of organic matter in sediments reflects a chemical change in which temperature acts during burial of sediments, causing many changes in the original organic matter. Many parameters have been suggested and used as possible indicators of organic maturity such as mean vitrinite reflectance R_o (%), the amount of extractable soluble organic matter, n -alkane preference index (CPI) and pentacyclic triterpane isomer ratios (Douglas and Williams, 1981). The geochemical maturity parameters for the samples studied are presented in Table 1.

In general, the ratios of Tm/Ts [$17\alpha(H)$, 22, 29, 30-trisnorhopane/ $18\alpha(H)$, 22, 29, 30-trisnorhopane] in samples derived from similar sources decrease with increasing maturity, so that the ratios can be used as a maturity indicator for samples from similar source (Seifert and Moldowan, 1978). However, for those two particular formations, Tm is predominant with very low concentrations of Ts isomer in all the rock extracts.

No distinct variation has been observed in Tm/Ts ratio. Among the samples studied here, this ratio may not be indicative of thermal maturity, but, more likely, is strongly influenced by source differences (Palacas et al., 1984).

It is well established that the carbon preference index (CPI) values are influenced by the type of organic matter and by the degree of maturity. Higher CPI values >1.5 always refer to relatively immature samples, low CPI values do not necessarily mean higher maturity, they can also mean a lack of higher normal alkanes originating from terrestrial input (Tissot and Welte, 1984). Bray and Evans (1961) quantified the odd/even preference in n -alkane distributions by defining a carbon preference index (CPI) and they found this value is higher in immature sediments, but near unity, or just below, in crude oils.

The slight odd-even predominance of n -alkanes in these samples (CPI_{24–35}: -0.89–0.95, Table 1) is consistent with their thermal maturity range. A decrease in CPI values may indicate an increase in thermal maturity. Extracts from samples of the Setap Shale and Belait formations have a predominance of Tm over Ts. In addition, the abundance of C₂₉Ts is high and the abundance of C₃₀m is relatively low with the C₃₁–C₃₅ extended hopanes. The lower moretane concentrations and elevated concentrations of the Ts series reflect the higher maturity of these sediments. The stereoisomeric ratios, 22S/22R of the extended hopanes (C_{31–35}) also have been used as maturity in-

dicators (Mackenzie et al., 1980; Seifert and Moldowan, 1978). In general, 22S/22R ratios <1.2 are considered to be immature, whereas ratios of 1.3 or greater indicate that the rocks are near or within the oil generation level (Palacas et al., 1984). Using these values as the maturity guide line in these rock extracts, all the samples studied show 22S/22R ratios >1.25 for the C₃₁, C₃₂ and C₃₃ hopanes (Table 1), indicating that these sediments have reach a high level of thermal maturity (probably in the late oil window range).

The 20S/20S+20R for the C₂₉ sterane ratios and the 22S/22S+22R for the C₃₁ and C₃₂ extended hopane ratios have been widely used as maturity indicators. The calculated values for these ratios for the Setap Shale and Belait samples are within a narrow range to each other with little variation and do not unambiguously distinguish the small maturity differences between the two formations. The C₃₂ homohopane maturity indicators 22S/22S+SSR are close to equilibrium values (0.57–0.62; Peters and Moldowan, 1993).

The 22S/2S+22R ratio for C₃₂ hopane in all the samples studied is about 0.60 (i.e., in the range of 0.53–0.57 & 0.55–0.64 for the extracts of the Setap Shale and Belait formations respectively, Table 1). The 22S/22S+22R ratio has an equilibrium value of 0.6 which corresponds to the onset of hydrocarbon generation (Mackenzie, 1984).

The isomerization ratio of the C₂₉ steranes, as shown by the epimer ratios of 20S/20S+20R, varying from 0.48 to 0.69 for the extracts of the Setap Shale Formation, whereas, the ratios range from 0.43 to 0.51 for the extracts of the Belait Formation, typical of equilibrium values, thus supporting the 22S/22S+22R for the C₃₂ extended hopane ratios and the stereoisomeric ratios, 22S/22R of the extended hopanes (C_{31–33}).

This high maturity suggests that the sediments have been buried at considerable depth, prior to being uplifted to their present position. Although of high maturity the samples of the Setap Shale are still within the oil window range (toward the end of it), suggesting these samples have not been too severely affected by thermal metamorphism or active tectonic activities of the Sabah.

3.11 Depositional environment

High Pr/Ph ratios have been generally inferred to indicate that the organic matter has undergone some degree of oxidation and decarboxylation of the phytol side-chain of chlorophyll which leads to the formation of pristane. Under reducing conditions, phytane is more likely to be produced through hydrogenation and dehydration of phytol (Brooks et al., 1969). Organic matter derived predominantly from terrestrial plants would therefore be expected to show a high Pr/Ph ratio and organic matter derived predominantly from

aquatic photosynthetic organisms, such as algae, could also have a high Pr/Ph ratio if the water column was well oxygenated (Clayton, 1993).

In peat swamp environments, with low aerobic bacterial activity, most of the phytol is converted to pristane with only small amounts of phytane. Thus, oils from source rocks deposited under peat swamp conditions will have high Pr/Ph (>3.0) and Pr/nC₁₇ (>1.0) ratios (Didyk et al., 1978; Lijmbach, 1975), whereas in marine or open water sediments with high bacterial activity yields only relatively small amounts of pristane and phytane as a result give low Pr/Ph (<2.0) and Pr/nC₁₇ (<1.0) ratios. This is observed for all of the Setap Shale and Belait samples analyzed. Although the Pr/Ph ratio values for the samples studied are most likely to be influenced by source type, the low Pr/Ph (<1) ratios suggesting an aquatic depositional environment under reducing bottom conditions. This is in agreement with the depositional environments postulated by workers such as Wilson (1964) who postulated the Belait Formation as fluvial to shallow marine sediments and the Setap Shale is a formation consisting of soft, dark gray shales and mudstones deposited in marine environments.

The hopane distribution patterns of all samples are characterized by a regular stair-step progression of C₃₁ to C₃₅ homohopanes. This and the very small homohopane indices (C₃₅/C₃₄-C₃₅) suggest anoxic conditions. The plot of Pr/Ph ratios vs. C₃₅/C₃₄ homohopanes also suggests less oxic depositional environments for these extracts (Fig. 6).

According to Peters and Moldowan (1993), the oxygen concentrations in the depositional environments can be evaluated using the 17 α (H)-diahopane/18 α (H)-30-norneohopane (C₃₀*/C₂₉ Ts) ratio. This ratio increases with increasing oxygen concentrations. That is, low ratios indicate anoxic and high ratios oxic conditions. Because 17 α (H)-diahopanes are more stable than 18 α (H)-30-norneohopanes, increasing maturity should also lead to the increase of ratios (Peters and Moldowan, 1993). In the samples studied the assumed depleted oxygen is in agreement with a relatively low (C₃₀*/C₂₉ Ts) ratio (Table 1).

The relatively enhanced concentrations of the C₃₁-C₃₅ compared to the C₃₀ hopane and C₂₉ norhopane have been proposed to be typically associated with carbonate sedimentary environments (Palacas et al., 1984). However, relatively high concentrations of extended hopanes and C₂₉-norhopanes were found in the rock extracts of the Setap Shale and the Belait formations, although the extracted samples were actually black shales and mudstones, not carbonates. Enhanced concentrations of extended hopanes in all the samples studied have been proposed to reflect the more anoxic nature of a depositional environment (Moldowan, 1988).

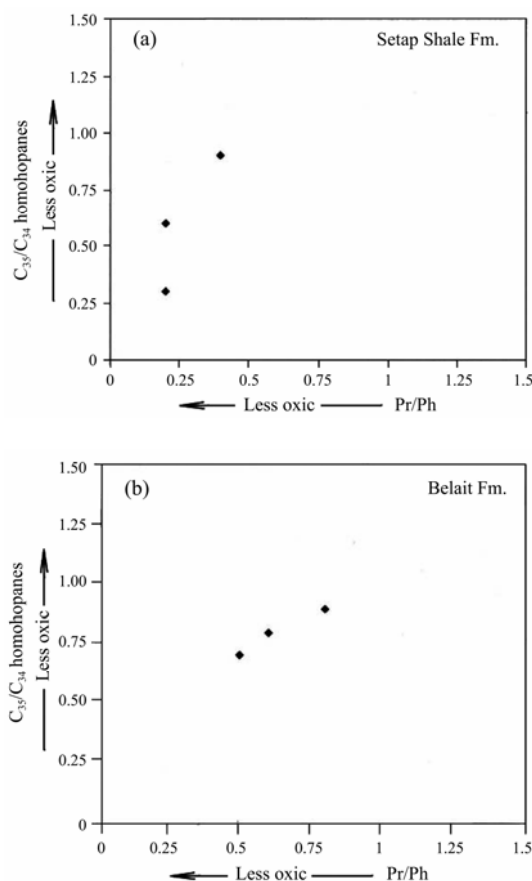


Fig. 6. Pr/Ph ratios vs. C₃₅/C₃₄ homohopanes as indicators of redox depositional environments for rock extracts of (a) the Setap shale Formation and (b) the Belait Formation.

The relatively high abundance of gammacerane in all the samples studied suggests a hypersaline source rock deposited under stratified anoxic marine conditions (Peters and Moldowan, 1991; Sinninghe et al., 1995), as supported by high gammacerane indices (gammacerane/hopane) and Pr/Ph ratios (<1.0).

4 Conclusions

The results from this investigation of organic-matter sediments in the Klias Peninsula area, West Sabah using organic geochemistry data show that the CPI values and measurement of the stereoisometric ratios (22S/22S+22R for C₃₂ hopanes and 20S/20S+20R for C₂₉ regular steranes) of the extended hopane and sterane series indicate that the sediments studied have reached a relatively high level of thermal maturity.

Feature include lower Pr/Ph ratios (<0.1), strong predominance of high molecular weight *n*-alkane, slight predominance of odd-numbered alkane (CPI₂₄₋₃₅<1), relatively high contents of 28, 30-bisnorhopane, low value of C₃₅/C₃₄ homohopanes (<1), high C₂₄ tetra/C₂₃ tricyclic ratio, presence of bi-

cadinanes and predominance of oleananes may be taken to indicate the presence of marine microbial materials, in addition to terrigenous input to the source of the extractable organic matter, but most probably reflect the more reducing conditions of the shallow marine environments. The presence of a high relative abundance of gammacerane also indicates anoxic marine hypersaline source depositional environment

The predominance of oleanane biomarkers in both formations is indicative of angiospermis input and Tertiary source rocks. The presence of significant marine influence in all the samples studied is suggested by the high C_{29}/C_{30} hopane ratios, moderate development of C_{33} - C_{35} hopanes, a predominance of C_{27} regular sterane over C_{28} and C_{29} steranes and the high abundance of tricyclic terpanes, thereby suggesting a mixed source input.

The distribution of tetracyclic, tricyclic and pentacyclic terpanes shows little variation between the study samples, suggesting that all the biomarker extracts were derived from the same organic mater types that were subsequently reworked/alterd by bacterial activity within the marine influence, suggested by the high abundance of tricyclic terpanes in all the samples studied.

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