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Biomarkers as Palaeoenvironment and Thermal Maturity Indicators of the Sandakan Formation (Late Miocene) East Sabah, Malaysia

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ABSTRACT This paper discusses biomarker distributions and their application as thermal maturity and palaeoenvironmental indicators for the Late Miocene Sandakan Formation. Parameters such as CPI (carbon preference index) and transformation of C_{31} and/or C_{32} 22S/ (22S+22R) hopane ratios, suggest that the samples analysed are thermally immature for oil generation. Very low ratios of C_{31} and/or C_{32} 22S/ (22S+22R) hopanes and very high CPI values are typical of low maturity. Vitrinite reflectance (%R_o) supports the low thermal maturity and an absence of features indicative of hydrocarbon generation. The dominance of n-C_{31} over n-C_{17} alkanes, C_{29} over C_{30} hopane and oleanane over C_{30} hopane suggest a high input of terrestrially-derived organic matter. The wide spread in parameters such as pristane/n-C_{17}, phytane/n-C_{18}, and Pristane/ Phytane ratios may, however, reflect variation in depositional conditions and/or source input within the Sandakan Formation. These samples appear to have been deposited in varying depositional conditions of oxic to sub-oxic to reducing conditions of deposition; of peat-swamp environments to alternating brackish swamps to more open-water conditions. A low relative abundance of tricyclic terpanes suggests only slight marine influence in the depositional setting, with perhaps slightly saline conditions based on minor occurrence of C_{24} tetracyclics and C_{23} tricyclics.

ABSTRAK Kertaskerja ini membincangkan taburan penanda biologi dan aplikasinya sebagai penunjuk kematangan terma dan palaeopersekitaran bagi Formasi Sandakan yang berumur Miosen Lewat. Parameter seperti CPI ("Carbon Preference Index") dan transformasi C_{31} dan/atau nisbah hopana C_{32} 22R/(22S+22R) mencadangkan yang sampel yang dianalisa adalah tidak matang secara terma untuk menjana minyak. Nisbah hopana C_{31} dan/atau C_{32} 22R/(22S+22R) yang rendah dan nilai CPI yang tinggi adalah kebiasaan bagi kematangan rendah. Pantulan vitrinit (%Ro) menyokong kematangan terma yang rendah dan ketidakhadiran ciri-ciri penunjuk bagi penjanaan hidrokarbon. Dominasi n-C_{31} dibanding n-C_{17} alkana, C_{29} dibanding C_{30} hopana dan oleanan dibanding C_{30} hopana mencadangkan kedapatan bahan organik asalan terrestrial yang tinggi. Taburan yang luas bagi parameter seperti pristan/n-C_{17}, phytan/n-C_{18}, dan nisbah Pristan/Phytan, mencadangkan variasi dalam keadaan persekitaran dan/atau punca asalan bahan organik di dalam Formasi Sandakan. Sampel-sampel ini berkemungkinan telah dienapkan dalam keadaan persekitaran yang berbagai dari oksik ke sub-oksik ke keadaan penurunan; dari persekitaran paya gambut berselang ke paya air payau ke keadaan air terbuka. Kedapatan tricyclik terpana yang rendah mencadangkan cuma sedikit sahaja pengaruh marin dalam persekitaran pengenapan, dengan kemungkinan sedikit keadaan payau berdasarkan kehadiran C_{24} tetracyclik dan C_{23} tricyclik yang sedikit.

(Biomarkers, Sandakan Formation, depositional conditions, thermal maturity, vitrinite reflectance (%Ro))

INTRODUCTION

The study area constitutes part of the Northeast (NE) Sabah basin. The NE Sabah basin, which is located along the east coast of Sabah (Figure 1), can be divided into two deep sedimentation areas and a platform area. The deep sedimentation areas comprise (i) the Central Sabah Sub-basin which occupies the Sandakan Rift of Tjia et al. [1] and (ii) the Sandakan Sub-basin [2]. The sedimentary succession within the basin has been dated to be of Miocene to Pliocene in age, and can be found exposed in the Sandakan Peninsula and the Dent Peninsula, which lie between Labuk Bay and Darvel Bay. The NE Sabah Basin formed following the late Eocene uplift of the ophiolite-deepwater sediments complex (Chert-spillite, Sapulut, Trusmadi and East Crocker formations). Deposition begins in a deepwater setting during the Oligocene, and continued until Pliocene with tectonics, to a large extent, controlling the basin depth and the sediment type. The variety of sediments range from deepwater clastics, limestones, olistostrome ('slump breccias' and 'melange'); fluviodeltaic and shallow marine clastics and also some volcaniclastics [3].

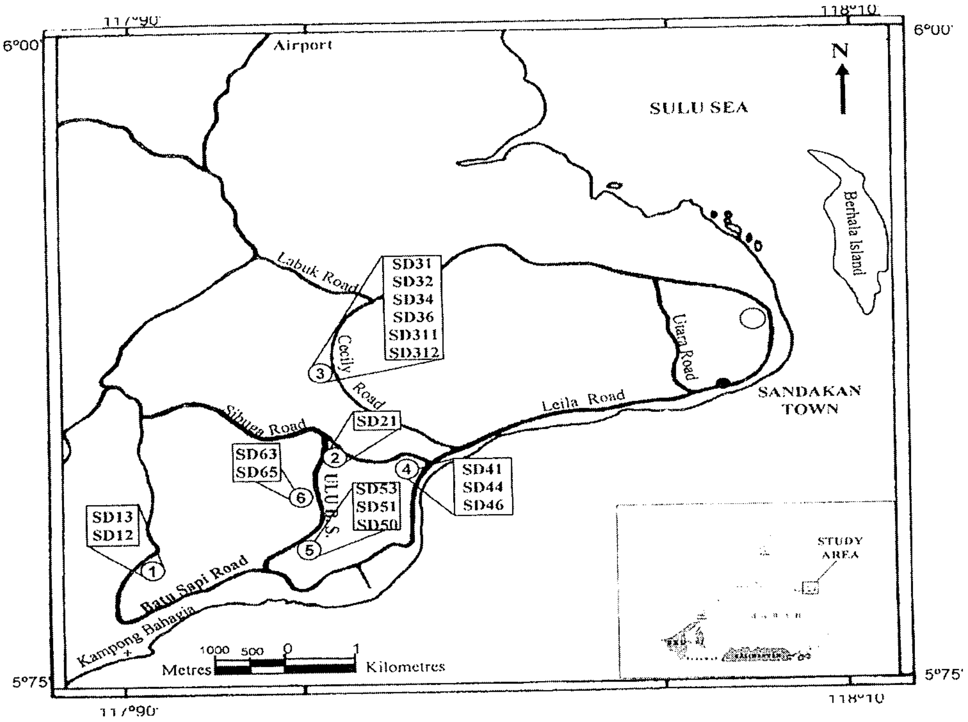

Figure 1. Map Showing the Sample Localities

1. IOI Outcrop
2. Ulu Batu Sapi – A Outcrop
3. Cecily Outcrop
4. Taman Samundera Outcrop
5. Ulu Batu – C Outcrop
6. Ulu Batu Sapi – B Outcrop

EXPERIMENTAL PROCEDURES

A number of coal and shale samples from the Upper Miocene Sandakan Formation were crushed weighed, and Soxhlet extracted for 72 hours using an azeotropic mixture (93:7) of dichloromethane and methanol. The extractable organic matter was subsequently fractionated using liquid chromatography. The chromatography column (30 cm× 0.75 cm i. d.), was packed with approximately 20 cm of silica gel (BDH 60-120 mesh) supporting a 2-3 cm layer of alumina. The silica was slurry-packed using light petroleum (bp 40-60°C), whilst the alumina was gravity packed. The column was carefully tapped during packing to prevent building up of air pockets. A known quantity (approximately 50 -100 mg) of the extract was dissolved in dichloromethane and absorbed onto alumina. The solvent was then removed from the extract by using a stream of Nitrogen. The adsorbed extractable organic matter was added to the top of the column. The column was then developed with solvents of increasing polarity i.e.

100 ml light petroleum, 100 ml dichloromethane, and 50 ml methanol, consecutively. The eluates were collected in separate 250 ml round-bottom flasks. The solvent was reduced by rotary evaporation and the weight of the eluates was recorded. The saturated hydrocarbon fraction of these sediments was analysed using gas chromatography mass spectrometry (GC-MS) on a Hewlett-Packard 5890 GC (split/splitless injection) interfaced to a Hewlett-Packard 5970 MSD. Separation was performed on a 25m × 0.2mm i. d. fused silica column coated with 0.11 um methyl silicon (HP-Ultra 1).

Microscopic examination of polished coal and shale blocks was performed on a Leitz (DMRXP) microscope MPV reflected light (Lambda 546 nm) using ×50/0.85 oil immersion objective under 'white' light and blue light excitation. Vitrinite reflectance measurements were performed in plane-polarized reflected 'white' light using a MPV SP photometer head.

RESULTS

The TIC (total ion current) trace of the aliphatic fractions of selected samples analysed is shown in Figure 2, and the derived data is summarized in Table 2. Most of TIC traces show bimodal profiles of n-alkanes. A selection of geochemical parameters was calculated from the acquired GCMS data and is discussed below.

Pristane/ phytane ratios

Pristane (Pr) and phytane (Ph) are present in all of the samples studied, usually as the most major constituents of the isoprenoid alkanes. Most of samples studied here display relatively low Pr/Ph ratios (Figure 2, Table 1) and show variation values between 0.5 and 1.6, the highest ratio occurs in the sample SD63 which has a value of 2.6. The samples SD41, SD32, SD51, SD31, SD311 are characterised by Pr/Ph ratios less than 1.00, whilst the rest samples have values greater than 1.0.

CPI (carbon preference index)

The measurement of CPI in this study is after Bray and Evans [4]. High CPI values represent an odd carbon number preference over even and occur in response to the chemistry of surface waxes in higher plants. Usually CPI values of greater than 1.0 (odd preference), indicate the extract is thermally immature. Values of CPI in this study are high (>1.7) (Table 1). Some samples exhibit n-alkane distributions with a strong odd predominance (6.84 for shale sample SD36, Table 1). Coal samples SD32 and SD34 show values of 2.67 and 2.75, respectively.

Tm/ Ts ratios

In this present study, most of the studied samples show high Tm/ Ts ratios. In general, the average of Tm/ Ts ratio of the samples is about 2.9. The coal samples (SD34, SD32) have values of 2.50 and 4.00, respectively. Shale samples have Tm/ Ts ratios ranging between 1.50 and 6.00 (Table 2). Thus, no distinct differentiation can be made between the coals and the shales based on Tm/ Ts alone.

Terpanes

The m/z 191 mass chromatograms of the aliphatic hydrocarbon fractions (Figure 3) of all the samples analysed show high abundances of pentacyclic triterpanes with relatively lower amounts of tricyclic and tetracyclic terpanes. Peak identification of all these compounds are based on their retention times and comparison of mass spectra with those published in previous literature [5]. The relative abundance of C_{29} hopane is generally high in all samples studied (Figure 3), while the C_{30} hopane is less abundant. For shale samples, the C_{29}/C_{30} ratio displays very high values between 2.68 and 20.00, whilst, the coal samples (SD32 and SD34) show values 3.30 and 4.47 respectively (Table 2).

A C_{24}–tetracyclic terpane is identified in most of samples. The values for C_{24} tetracyclic to C_{30} hopane ratio (C_{24} tetra/ C_{30} hopane) are displayed to be in the range of 0.13-0.42 (Table 2). The C_{23} tricyclic/ C_{30} hopanes ratios are generally low. Samples SD63, SD41, SD36, SD34 and SD32 are shown to have very low values of C_{23} tri/C_{30} hopane: 0.04, 0.06, 0.05, 0.07 and 0.03, respectively. While the samples SD21, SD65, and SD46 show slightly higher values of 0.21, 0.19, and 0.10, respectively. In the remaining samples, C_{23} tricyclic are absent. Values of C_{23} tricyclic over norhopane (C_{23} tri/C_{29} norhopane) ratios are also very low in all samples. They range from 0.01 to 0.06 (Table 2).

Homohopane isomerisation; 22S/ (22S+22R)

In this study, homohopanes are observed to be moderately low in most samples. The values of C_{31} and/or C_{32} 22S/ (22S+22R) homohopane range from 0.12 to 0.44. The ratio could not be calculated for some samples, such as the coal sample (SD34), and shale samples of (SD311 and SD31), due to low concentration (Figure 3; Table 2).

Oleanane index (Oleanane/C_{30} hopane)

There is a correlation between the level of higher-plant input and the oleanane index [6]. 18α (H)-oleanane is present in moderate abundance in most of the samples studied (Figure 3). The oleanane index is also known to be maturity-dependent [7]. However, for all of the samples analysed here, no significant variation is observed in the thermal maturity. In the present study, the oleanane index ranges from 0.12 to 0.57 except for coal samples and shales associated with coals which possess slightly higher values of up to 2.57 (See Table 2).

Petrographic Data

A selection of samples was studied under the microscope. The results of this petrographic study are discussed below.

Vitrinite reflectance (%Ro)

Mean vitrinite reflectance data of nine investigated samples in this study are shown in Table 3. Samples show a range of values of vitrinite reflectance from 0.35 to 0.45 %Ro. The shale samples display values of vitrinite reflectance between 0.36 and 0.42, whilst the coal samples show a range of values from 0.35 to 0.45.

Petrographic description

The coals of the Sandakan Formation consist predominantly of vitrinite. However, the examination of the coal samples under both white light and blue light excitation show macerals of the liptinite group are also present. Suberinite and resinite have been observed commonly in most of the samples studied here (Figures 4 and 5). Under white light, suberinite has low reflectance and occurs as dark grey to dark brown thin bands with toothed edges of variable thickness associated with phlobaphanitic cells.

Resinite is the most common liptinite maceral, and under white light it is homogenous and dark brown in colour. It commonly occurs as elliptical lipid resin bodies in structureless vitrinite matrix (Figure 4). However, the shale samples rarely show the presence of these macerals. Under "blue light" excitation, resin generally shows a yellow to yellow-orange fluorescence colour and apears to be partially structured (see Plate 4.3 of Tarek [8])

No oil generative features as described by Wan Hasiah [9] were observed in the Sandakan samples suggesting that no generation of liquid hydrocarbons has taken place within these coals and coaly sediments.

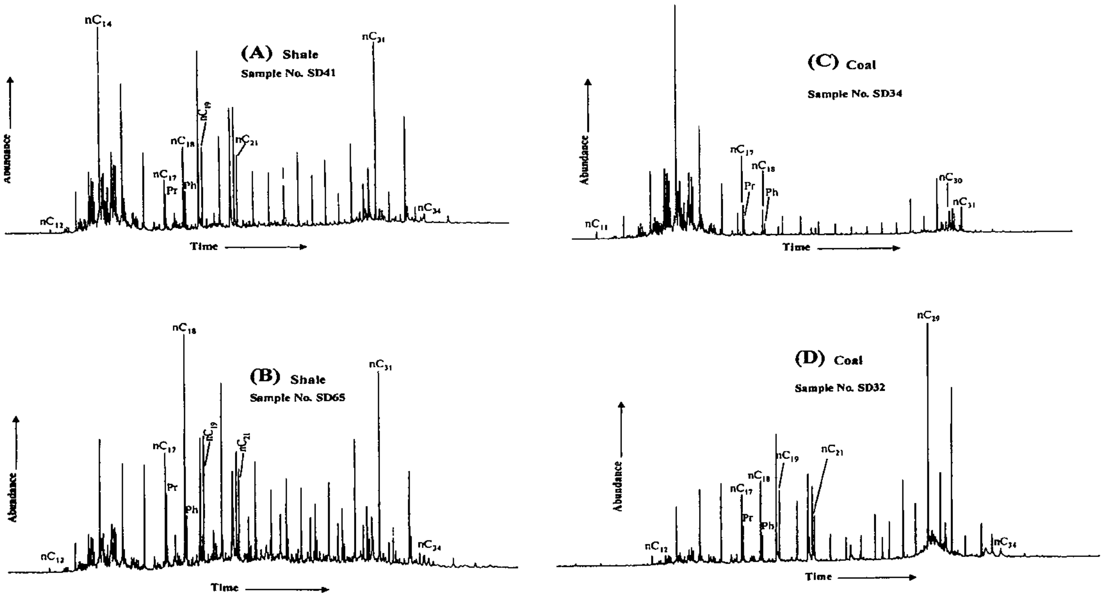

Figure 2. Gas chromatograms of saturated hydrocarbons from Sandakan Formation

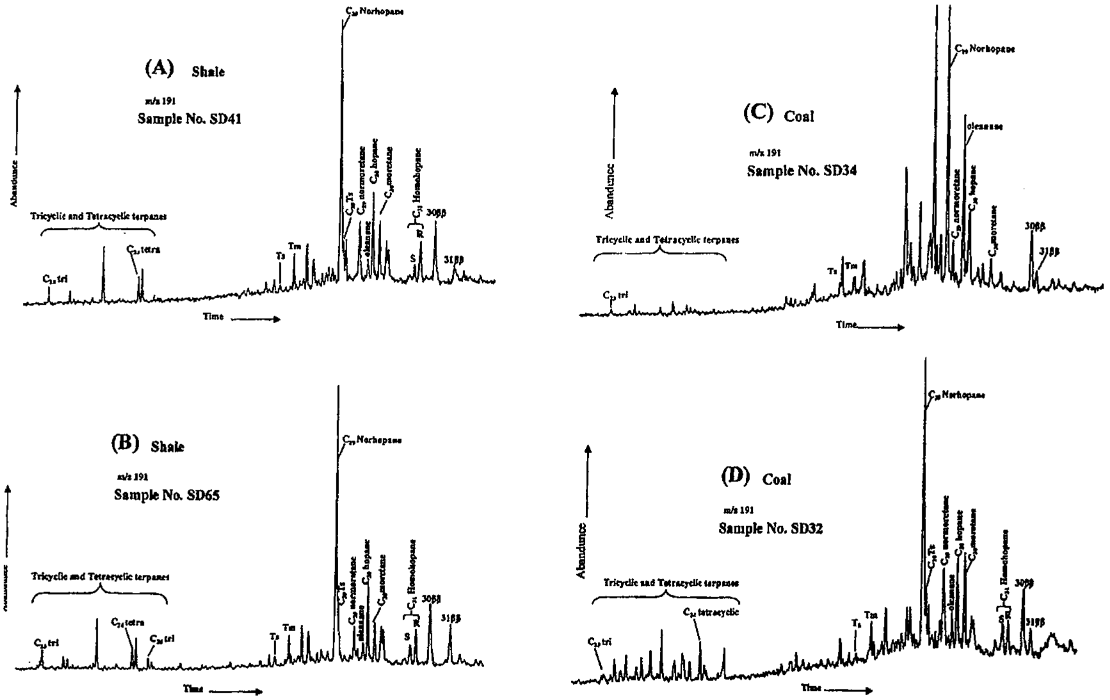

Figure 3. m/z 191 Triterpane mass fragmentograms of selected samples (A, B, C and D)

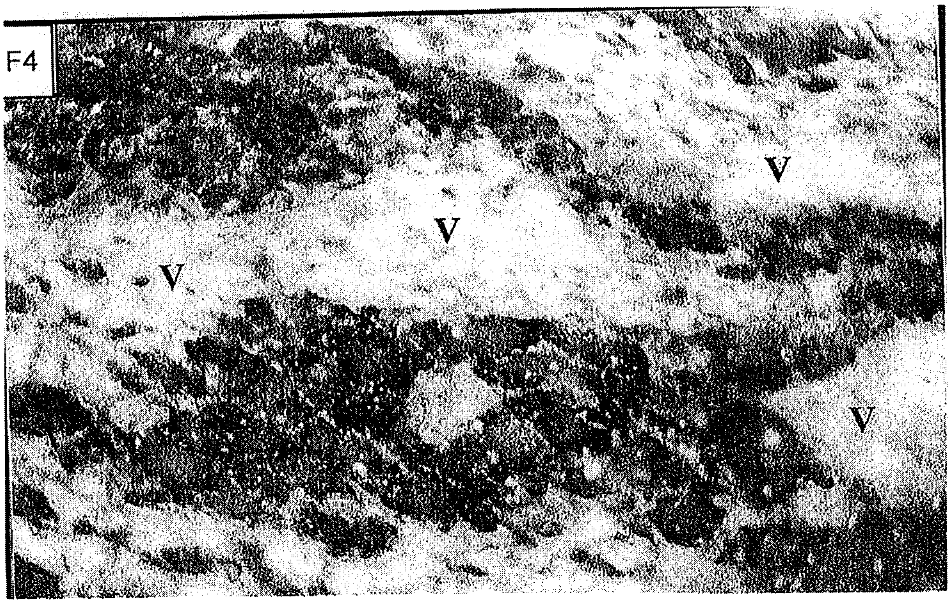

Figure 4. Resinite associated with vitrinite (v) in sample SD33 (coal); reflected light, field width = 0.25 mm

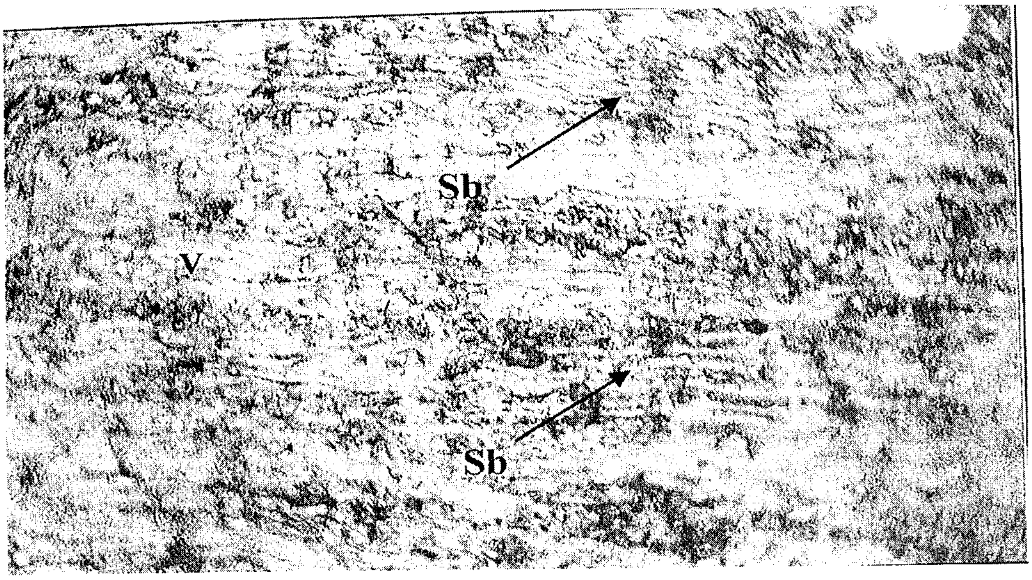

Figure 5. Suberinite (sb) association with vitrinite (v) is commonly observed in coal samples of Sandakan Formation; reflected light, field width = 0.25 mm

Table 1. TIC data of n-alkane and acyclic isoprenoids

Sample No.	Lithology	Range	n-alkane Maximum	nC_{31}/nC_{17}	nC_{25}/nC_{15}	CPI	Pr/Ph	Pr/nC_{17}	Ph/nC_{18}
SD63	Shale	nC_{13}-nC_{33}	nC_{18}	1.72	1.69	1.79	2.06	0.71	0.14
SD21	Shale	nC_{12}-nC_{33}	nC_{14}	0.95	0.41	2.10	1.41	0.63	0.33
SD65	Shale	nC_{12}-nC_{34}	nC_{18}	1.67	0.73	2.24	1.63	0.63	0.19
SD41	Shale	nC_{12}-nC_{34}	nC_{14}	3.70	0.52	2.35	0.94	0.68	0.43
SD46	Shale	nC_{13}-nC_{33}	nC_{14}	1.58	0.54	2.41	1.60	1.06	0.47
SD36	Shale	nC_{14}-nC_{33}	nC_{31}	15.73	2.18	6.84	1.47	2.00	0.86
SD13	Shale	nC_{13}-nC_{33}	nC_{14}	1.41	0.17	3.27	1.57	0.69	0.40
SD51	Shale	nC_{13}-nC_{34}	nC_{31}	6.70	0.68	3.49	0.80	0.94	0.50
SD34	Coal	nC_{11}-nC_{31}	nC_{14}	0.82	0.11	2.75	1.36	0.53	0.45
SD311	Shale	nC_{13}-nC_{33}	nC_{31}	23.00	0.85	3.31	0.90	2.50	1.57
SD32	Coal	nC_{12}-nC_{34}	nC_{29}	2.50	0.65	2.67	0.51	0.33	0.96
SD31	Shale	nC_{13}-nC_{33}	nC_{31}	3.04	0.12	3.28	0.72	1.04	1.13

Pr: Pristane; Ph: Phytane; CPI: Carbon preference index, CPI values are calculated using the formula of Bray and Evans [4]

$$\mathrm{CPI} = 1/2 \left[\frac{C_{25}+C_{27}+C_{29}+C_{31}+C_{33}}{C_{26}+C_{28}+C_{30}+C_{32}+C_{34}} + \frac{C_{25}+C_{27}+C_{29}+C_{31}+C_{33}}{C_{24}+C_{26}+C_{28}+C_{30}+C_{32}} \right]$$

Table 2. Biomarker ratios of selected samples

Sample No.	Lithology	C_{29} hopane/ C_{30} hopane	Tm/ Ts	Oleanane/ C_{30} hopane	C_{31} 22S/(22S+22R)	C_{24} tetra/ C_{30} hopane	C_{23} tri/ C_{30} hopane	C_{23} tri/ C_{29} norhopane
SD63	Shale	2.68	2.50	0.12	0.21	0.13	0.04	0.01
SD21	Shale	3.27	2.40	0.16	0.30	0.25	0.21	0.06
SD65	Shale	3.57	3.00	0.19	0.33	0.23	0.19	0.05
SD41	Shale	3.12	6.00	0.19	0.28	0.31	0.06	0.02
SD46	Shale	3.28	1.75	0.16	0.28	0.16	0.10	0.03
SD36	Shale	3.64	2.00	0.14	0.18	0.15	0.05	0.01
SD13	Shale	11.87	1.50	0.25	0.33	0.25	-	-
SD51	Shale	6.85	1.75	0.21	0.12	0.21	-	-
SD34	Coal	4.47	2.50	2.57	-	-	0.07	0.01
SD311	Shale	20.00	4.75	0.25	-	-	-	-
SD32	Coal	3.30	4.00	0.40	0.44	0.42	0.03	0.01
SD31	Shale	13.00	3.40	0.57	-	-	-	-

Tm: 17α (H),-22,29,30- trisnorhopane; Ts: 18α (H),-22,29,30-trinorneohopane; 22S/(22S+22R) of C_{31}17α (H), 21β (H) homohopane ratio; C_{23} tri: C_{23} tricyclic terpanes; C_{24} tetra: C_{24} tetracyclic terpanes

Table 3. Vitrinite reflectance data (%Ro)

Sample No.	Lithology	R_{min}	R_{max}	$R_{mean\text{-}value}$ (%Ro)	Standard Dev.	Total No.
SD65	Shale	0.30	0.49	0.36	0.06	25
SD44	Shale	0.30	0.52	0.40	0.05	25
SD46	Shale	0.31	0.45	0.38	0.03	25
SD12	Coal	0.41	0.48	0.43	0.02	30
SD34	Coal	0.41	0.46	0.43	0.01	30
SD50	Coal	0.32	0.41	0.35	0.02	30
SD53	Coal	0.40	0.46	0.45	0.01	30
SD312	Coaly Shale	0.27	0.48	0.37	0.05	30
SD31	Shale	0.35	0.52	0.42	0.05	25

DISCUSSION

Origin of Organic Matter and Depositional Conditions

Most of the TIC traces are shown to have bimodal distribution of n-alkanes which may indicate a mixture of terrestrial organic matter and algal organic matter. The samples have a maximum of n-alkanes at n-C_{14}, n-C_{18}, n-C_{29}, or n-C_{31} (Figure 2; Table 1).

However, the dominance of nC_{31} over nC_{17}, C_{29} over C_{30} hopane and oleanane over C_{30} hopane (Tables 1 and 2) in most of the samples analysed suggest a high input of terrestrially-derived organic matter. This is supported further by the high values of Tm/Ts ratios in most of the analysed samples. High Tm/Ts ratios are indicative of terrestrial-derived organic matter deposited under oxic environment [10].

Pristane/Phytane (Pr/Ph) ratios have been used to asses the redox potential of a depositional environment [11]. Pr/Ph ratios less than unity have been reported to indicate an anoxic depositional environment and the oxic environment is indicated by ratios greater than unity [12]. Volkman and Maxwell [13] pointed out that for source rocks within the oil generative window, high Pr/Ph ratios (> 3.0) indicate terrestrial organic matter input under oxic conditions, and low values (< 0.6) indicate anoxic, commonly hypersaline environments. It can therefore be deduced that the samples in this investigation, which have a strong variation of Pr/Ph ratios between 0.51 and 2.06, were deposited in mixed anoxic to sub-oxic conditions. Nevertheless, Peters and Moldowan [12] warned that for samples showing Pr/Ph ratios in the range 0.8 to 2.5, it is not recommended to make paleoenvironment interpretations without corroborating data. Furthermore, Pristane/Phytane ratios are known to be affected by maturation [14] and by differences in the precursors of acyclic isoprenoids (i.e. bacterial origin; e.g. [15, 16]).

There is little variation for the Pr/nC_{17} and Ph/nC_{18} ratios among the analysed samples. However, it is interesting to note that these ratios are slightly higher in shales compared to coals of similar sub-environments Table 1). This suggests a stronger influence of depositional conditions compared to source input for these two ratios.

Connan et al. [17] reported the occurrence of C_{24} tetracyclic in a hypersaline environment. It was also reported in carbonates and evaporite depositional environments by Connan and Dessort, [18]. On the other hand, Philp and Gilbert [5] regarded the occurrence of C_{24} tetracyclic as a terrestrial marker.

Although, the C_{24} tetracyclic is low in abundance in samples studied here, this occurrence may indicate that the environments might have been slightly affected by saline depositional conditions albeit with considerable terrestrial organic matter input.

The C_{23} tricyclic terpanes/C_{30} hopanes and C_{23} tricyclic terpanes/C_{29} norhopane ratios in most of samples studied are very low. This is due to the low maturity of all the studied samples however, the occurrence of tricyclic terpanes is indicative of a marine influenced depositional condition [19].

The hopanes are the predominant compounds (the most abundant is C_{29}) as shown in the m/z 191 mass chromatograms of the Sandakan Formation samples (Figure 3). The higher values of C_{29} hopane/C_{30} hopane ratios (Table 2) suggest a stronger terrestrial source input compared to samples possessing lower values of this ratio [20].

Source rock thermal maturity

Thermal maturity describes the extent of heat driven reactions which convert sedimentary organic matter into petroleum [12]. The maturation parameters are summarized in Tables 1, 2 and 3. In this study several parameters have been investigated and used as possible indicators of maturity of organic matter including vitrinite reflectance (%R_o), n-alkane preference index (CPI) and pentacyclic triterpanes isomers ratios.

Vitrinite reflectance data is widely used to determine the thermal maturity of kerogen in sedimentary rocks [21, 22]. It is more related to the thermal stress experienced by vitrinite than to petroleum generation. The thermal maturity of the coal and the shale samples in this study, based on vitrinite reflectance measurements range from 0.35 % Ro to 0.45 % Ro (Table 3), and thus the Sandakan Formation is not sufficiently mature to generate oil or gas.

The relative abundance of odd to even carbon numbered n-paraffins has been used to estimate thermal maturities of source rock extracts and oil. The measurement of CPI ratios in this study display very high CPI values (> 2), thus supporting the immature nature of the studied samples as based on vitrinite reflectance (Table 3). CPI values above 1.0 (odd preference) indicate the extract is thermally immature [12]. Low (CPI) values do not necessary mean higher maturity; they can also mean a lack of higher n-alkane stemming from terrestrial input [23].

The C_{31} isomerisation ratio ranges between 0.12 to 0.44 (Table 2). Seifert and Moldowan [23] reported that the isomerisation ratios in the range of 0.50 to 0.54 indicated that a source rock has barely entered oil generation window. Ratios in the range between 0.57 and 0.62 indicate that a main phase of oil generation has been reached or surpassed. All the samples analysed here display very low isomerisation (less than 0.5) of C_{31} and/or C_{32} 22S/ (22S+22R) hopane ratio (Table 2), thus these samples are thermally immature.

CONCLUSION

Biomarkers and petrographic characteristics suggest that the samples analysed are thermally immature for oil generation. Petrographic examination showed no features indicative of hydrocarbon generation.

The dominance of n-C_{31} over n-C_{17} alkanes, high C_{29}/C_{30} hopane and moderate oleanane/C_{30} hopane ratios suggest a high input of terrestrially-derived organic matter. The low occurrence of tricyclic terpanes suggests only a possible slight marine influence. The occurrence of C_{24} tetracyclic as well as C_{23} tricyclic may suggest slight saline depositional conditions.

The differences in parameters such as pristane/n-C_{17}, phytane/n-C_{18}, and Pristane/ Phytane ratios may, however, reflect variation in depositional conditions and/or source input. In general the biomarker analysis performed, based on isoprenoid and normal alkane distributions, suggest that these samples were deposited in varying depositional conditions of oxic to sub-oxic to reducing conditions of deposition i.e. variations from peat-swamp environments to alternating brackish swamps and more open-water conditions.

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