Source rock characteristics and hydrocarbon generation potential in Brunei-Muara district, Brunei Darussalam: a comparative case study from selected Miocene-Quaternary formations

Liyana Nadiah Osli1 · Mohamed Ragab Shalaby1,2 · Md. Aminul Islam1

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Abstract
A comparative analysis on source rock properties has been carried out on the Miocene-Pliocene formations as well as the Quaternary terrace deposits using Rock–Eval pyrolysis results and organic petrography as well as some biomarkers results. Samples were obtained from outcrops of the Quaternary terrace deposits, Pliocene-aged Liang Formation together with the Miocene Miri and Setap Shale formations in Brunei-Muara district, with sample lithologies ranging from coal, coaly shale, shale and lignitic sand. High total organic carbon (TOC) and $S_2$ values ranging from 41.8 to 62.4% and 7.40 mg HC/g rock to 122 mg HC/g rock, respectively, are identified in coals of the terrace deposit, Liang and Miri formations, making these as the best potential source rock due to the “good to excellent” generating potential. Meanwhile, a “fair to poor” potential is exhibited for the coaly shale, shale and lignitic sand samples as a result of their low TOC, HI and $S_2$ values. The organic matter is composed of kerogen type III (gas prone) and type II-III (mixed oil and gas prone). Organic matter in all studied formations originate from a terrestrial-source, as proven by the abundance of huminite. Organic petrographical and biomarkers studies suggest that the coals and lignitic sand samples were deposited in a mangrove-type mire in a lower delta setting, under oxic and limnic to limnotelmatic conditions, except sample DD2-1, which is deposited in a less water-saturated environment. The samples display the presence of bi-modal and normal distribution of n-alkanes. For all of the samples, the dominating plant types in the palaeomire are of soft, herbaceous plants and this is supported by the low vegetation index and moderate $P_{aq}$ values. All the studied samples are thermally immature to early mature, as exhibited by the $T_{max}$ values that range from 300 to 437 °C and vitrinite reflectance readings of 0.22% to 0.46%.

Keywords Miri formation · Setap Shale formation · Liang formation · Quaternary terrace deposits · Source rock characterization · Organic petrography · Coal rank

Introduction
In Brunei Darussalam, good-quality source rocks exist only as centimeter-thick layers and lenses, without having a mappable thickness and lateral distribution (Scherer and Hitam 1992). These organic-rich layers are present in the Neogene sediments of Brunei, which are regularly studied on varying subject matters; most are associated with petroleum geology (James 1984; Levell 1987; Sandal 1996; Schreurs 1997; Petronas 1999; Lambie and Cullen 2013), regional tectonics (Tingay et al. 2003, 2009), sedimentology (Lambie et al. 2002; Siddiqui et al. 2013; Back et al. 2008; Lambie and Cullen 2013; Rahman et al. 2014) and palaeodepositional environment studies (Back et al. 2001; Torres et al. 2011).

The Belait Formation has continuously become the primary formation of focus for source rock characterization studies in Brunei Darussalam, such as those carried out by Abdullah (1999; 2003) and Curiale et al. (2000). The Belait Formation of the Champion Delta complex accommodates organic-rich sediments in the form of either in-situ coal seams or marine sediments with dispersed organic matter (Schreurs 1997). Similar coal-bearing sediments are also identified in other formations other than the Belait

* Liyana Nadiah Osli
anadiah.osli@gmail.com
1 Department of Geosciences, Universiti Brunei Darussalam, Jalan Tungku Link, Bandar Seri Begawan BE 1410, Negara Brunei Darussalam
2 Geology Department, Faculty of Science, Tanta University, Tanta 31527, Egypt
Formation in Brunei Darussalam. In the Brunei-Muara district, coals are identified in the Miri and Liang formations, as well as in terrace deposits. The Miri Formation is lithologically similar with the Belait Formation and both of them are synchronous with each other, together with the Seria and Setap Shale formations (Figs. 1 and 2; Wilford 1961).

Younger deposits occur as terraces, which are deposited within the last million years, covering an area of approximately 2400 sq km in Brunei (Tate 1971). Wood particles are present within the terrace deposits, which can somehow be comparable with the lignites present in the other formations. The MR1 outcrop occurring along the Muara-Tutong highway is an exposure of Quaternary terrace deposits separated from the underlying layers of sand and shale belonging to the Belait Formation by an angular unconformity (Fig. 3a and b). A closer examination of the terrace deposit indicates the presence of brown-coloured carbonaceous (Fig. 3c) sand with high-angle cross-bedding (Fig. 3d) and occasional pebble layers as well as lignified wood.

To date, there has been no published source rock studies concerning the Miri, Liang and Setap Shale formations of Brunei Darussalam. Most of the available studies that focus on one or some of the aforementioned formations primarily concern the formations’ potential as a petroleum reservoir. Studies on the geology of terrace deposits in Brunei are also limited but there are multiple studies carried out on these formations in neighboring regions. The organic geochemistry and depositional environment of the Liang Formation in Sarawak, Malaysia has been analyzed by Hakimi et al. (2013) and Murtaza et al. (2018), respectively. The Miri Formation in Sarawak has been widely assessed for its reservoir potential (Abieda et al. 2005; Jia and Rahman 2009; Siddiqui et al. 2014) and depositional facies (Rahman and Tahir 2018). Organic matter maturity and palaeodepositional environment of the Setap Shale Formation of Sabah, Malaysia was investigated by Burgan and Ali (2010) and were comparatively examined with the Belait Formation of the same region. Similar exposures of terrace deposits are also observed in Miri, Sarawak, and these units have been examined by Kessler and Jong (2014). Although Quaternary deposits in Brunei are not typically examined for their petroleum properties, it is still worth examining the lignites and lignitic sands of the terrace, especially relating to their potential as a possible source rock.

The central idea of this research is to evaluate the source rock potential of the terrace deposits, Liang, Miri and Setap Shale formations using outcrop samples in the Brunei-Muara district of Brunei Darussalam (Fig. 1a–c). To do so, the geochemical properties and hydrocarbon generation potential of the three formations are investigated. Our results are based on the integration of data from Rock–Eval pyrolysis, organic petrography and biological markers in order to characterize the potential source rocks.

Geochemical properties examined include organic matter type, coal rank and thermal maturity, whereas palaeoenvironment and the conditions of depositions were mostly identified through organic petrography. Furthermore, the intention of this study is also to provide more information on the Liang, Miri and Setap Shale formations to enhance our understanding about the petroleum system as well as the general stratigraphy of Brunei.

### Geological settings

The geology of Brunei is mainly controlled by delta systems that evolved since Miocene (James 1984; Sandal 1996; Schreurs 1997). The oldest of the three delta complexes is the Meligan delta that was generated in the Palaeogene. Middle to late Miocene deposits belonging to the Champion delta are separated from the Meligan Delta by a significant regional unconformity (Hutchinson 2005). Baram Delta is the youngest of the three deltas and occurred from the early Pliocene to recent (Sandal 1996). Formation of these delta systems can be traced back to the geological history of Borneo that was active since the Mesozoic. In the Cenozoic, the clockwise rotation of the Philippine Sea plate led to the plate boundaries changes that are essential in developing the current tectonic pattern observed in Southeast Asia (Hall 1997). Borneo began its anti-clockwise rotation in Neogene, during which there was also a change in sedimentation in basins that surrounded the island. A counter-clockwise rotation of the island of no more than 25° began approximately 20 Ma (Hall 1996; Moss et al. 1997; Baillie et al. 2004). This is attributed to the northward motion of Australia during that time (Hall 1996) and concurrently happening to this is the anti-clockwise rotation of west Sulawesi and adjacent Sundaland blocks (Hall 1996). Borneo was uplifted as a result of compressional forces in the Neogene (Baillie et al. 2004), causing rapid delta progradation that results in an elevated sediment production. Erosion of central Borneo mountains led to more sediment input into surrounding basins including the Baram basin (Hutchison 1996; Hall and Nichols 2002; Baillie et al. 2004).

Hydrocarbons in Brunei Darussalam are attributed to sediments of the Champion delta, which is represented by the Setap, Belait, Lambir, Miri and Seria formations (Fig. 2; James 1984; Koopman 1996; Sandal 1996; Curiale et al. 2000). The argillaceous Setap Shale Formation is overlain by the Belait, Miri, Seria and Lambir Formations (Tate
The Belait Formation is synchronous with the Setap Shale Formation (early to Middle Miocene) and the Lam
bir, Miri and Seria formations (middle to Late Miocene). Environment of deposition differs between the formations,
as observed from outcrops across the study area. The Belait Formation is composed of primarily sandstone with shales
and coal interbeds. The Miri Formation is lithologically similar with the Belait Formation, comprising of mainly arenaceous sediments with subordinate clays and shales (Wilford 1961), although it is relatively more marine as it was deposited in a shallow marine environment. The Setap Shale was
deposited in a deep marine environment that occurred in the Oligocene, comprising of clay and shale with thin silty
and sandy beds (Wilford 1961; Back et al. 2001). The Liang Formation consists of two members; the Berakas Member
is located within the Berakas Syncline whereas the Lumut Member is located in the Lumut Hills (Wilford 1961). The
main lithologies of this formation are sand, clay and gravel with lignite beds.

![Chronostratigraphy of onshore Brunei Darussalam (modified after Sandal 1996)](image)

**Fig. 2** Chronostratigraphy of onshore Brunei Darussalam (modified after Sandal 1996)
Methodology

Field work and sampling

A total of 16 samples derived from the Quaternary terrace, Miri, Setap Shale and the Berakas Member of Liang Formations were collected from 8 different locations across the Brunei-Muara district are assessed in this study (Fig. 1). Lithology of samples collected range from coals, coaly shale, lignitic sand and shale. In Table 2, sample ID and corresponding lithology are summarized. All coal samples of this study are in-situ except MR1-1, LU1-1 and TL1-1.
samples. Outcrops were selected based on information derived from literature, reference from previous field trips, minimal disturbance and outcrop accessibility. Prior to sampling, sedimentological structures are recorded.

**Analytical procedures**

All 16 samples collected from the terrace deposits, Liang, Miri and Setup Shale formations were analyzed to obtain Rock–Eval data in Hydrocarbons Chemistry and Technology Lab, Technical University of Crete. Following this, representative samples from each formation were selected for subsequent analyses in this study. For organic petrography and vitrinite reflectance, a total of 8 samples were examined in the Department of Geology, University of Patras, Greece. Biomarkers analyses were carried out in Hydrocarbons Chemistry and Technology Lab, Technical University of Crete, in which 5 samples were chosen for rock extraction and fractionation, whereas 3 samples were selected for gas chromatography (Table 1).

In Rock–Eval pyrolysis, samples are initially crushed, sieved and dried overnight at 40 °C. Rock samples were then purged with nitrogen gas and heated at 300 °C for 6 min. The samples were subsequently pyrolyzed to 650 °C and further burnt in the oxidation oven in air. Principal pyrolysis parameters such as $S_1$ (a measure of free hydrocarbons or bitumen available in the source rock before pyrolysis; mg HC/gm rock), $S_2$ (a measure of the potential hydrocarbon volume formed during thermal pyrolysis; mgHC/gm rock), $S_3$ (a measure of inorganic-originated CO$_2$; mg CO2/gm rock), $T_{\text{max}}$ (temperature with the maximum amount of hydrocarbon generated during pyrolysis) and TOC (total organic content) were obtained from the Rocksix V4.08 software.

Primary parameters obtained from Rock–Eval pyrolysis can be integrated in different ways to analyze several properties of a potential source rock. PI is defined as the amount of free hydrocarbon generated to the total amount of hydrocarbon that the organic matter is capable of generating (Tissot and Welte 1984; Peters and Cassa 1994). This parameter is calculated through the formula ($\text{PI} = \frac{S_1}{\text{PY}}$), whereas Production Yield (PY) is generated from the sum of S1 and S2 ($\text{PY} = S_1 + S_2$). Two parameters that are useful in determining the kerogen types present in a source rock are Hydrogen Index (HI) and Oxygen Index (OI), in which both parameters are calculated using the formulae ($\text{HI} = \frac{S_2}{\text{TOC}} \times 100$) and ($\text{OI} = \frac{S_3}{\text{TOC}} \times 100$) respectively.

Selected samples that were initially crushed to appropriate sizes (< 1 mm) or block samples were polished and prepared according to ISO 7404–2 (2009) for maceral analysis. Examination of samples were carried out using a LEICA DMRX microscope at the Department of Geology, University of Patras. Maceral identification was performed in oil immersion under both white incident light and blue-light excitation (ISO 7404–3 2009; ASTM D7708 2014), following the nomenclature of the Stopes-Heerlen System as modified by ICCP System 1994 (ICCP 1998; 2001; Sýkorová et al. 2005; Pickel et al. 2017). Relative abundance of macerals was determined using point-count analyses (500 points).

Mineral matter input in rocks is represented as the ash content of the samples (Diesel 1992). Ash yields were identified

| Formation name | Sample ID | Geochemical analysis | Maceral abundance | Vitrinite reflectance | Ash content | Rock extraction and fractionation | GC-MS Chromatography |
|----------------|-----------|----------------------|-------------------|----------------------|-------------|----------------------------------|-----------------------|
| Terrace deposit | MR1-1     |                      |                   |                      |             |                                  |                       |
|                 | MR1-2     |                      |                   |                      |             |                                  |                       |
|                 | MR1-3     |                      |                   |                      |             |                                  |                       |
| Liang formation | BR1-1     |                      |                   |                      |             |                                  |                       |
|                 | BR2-1     |                      |                   |                      |             |                                  |                       |
|                 | BR2-2     |                      |                   |                      |             |                                  |                       |
|                 | BR2-3     |                      |                   |                      |             |                                  |                       |
|                 | BR2-5     |                      |                   |                      |             |                                  |                       |
|                 | BR2-6     |                      |                   |                      |             |                                  |                       |
| Miri formation  | LU1-1     |                      |                   |                      |             |                                  |                       |
|                 | TL1-1     |                      |                   |                      |             |                                  |                       |
|                 | TN1-1     |                      |                   |                      |             |                                  |                       |
|                 | DD2-1     |                      |                   |                      |             |                                  |                       |
| Setup shale formation | JD1-1   |                      |                   |                      |             |                                  |                       |
|                 | JD1-2     |                      |                   |                      |             |                                  |                       |
|                 | JD1-3     |                      |                   |                      |             |                                  |                       |
from proximate analysis that was undertaken according to ASTM D3174 (2004). In identifying ash yields, samples were initially crushed to sizes of less than 200 μm and combusted at 750 °C using a Selecta Muffle furnace. Percentage of ash is determined by weighing the ash left behind post-combustion with respect to the weight of sample [(weight of ash post-combustion/total sample weight before combustion) × 100].

In biomarkers analysis, approximately 30 g of rock from selected samples were initially crushed and sieved using 60 mesh before undergoing solvent extraction using the Soxhlet technique, which utilizes a dichloromethane-methanol mixture (90:10 volume). Copper tips were used during extraction to eliminate presence of elemental sulphur. Excess n-pentane were then utilized for de-asphalting of rock extracts and subsequently filtered using Teflon syringe filters (0.45 μm). Open-column chromatography was applied to separate the isolated maltene portion into saturates, aromatics and polar (NSO) compound groups. These compounds were then eluted using different solutions; 7 ml of n-pentane for saturates, 6.5 ml of toluene for aromatics and a 60:40 v/v mixture of toluene-methanol for NSO compounds.

In GC–MS analysis, n-hexane (0.5 ml) were used to dissolve the saturated and aromatic fractions, with 15 μl of internal standard added. This standard is composed of a Chiron mixture S-4121-ASS-IO containing n-C12-D26, 1-Fluoronaphthalene, 3-Fluorophenanthrene, n-C16-D34, 2-Fluorochrysene, and 5β(H)Cholane. GC–MS analysis was carried out on an Agilent 5975E Mass Spectrometer and an automatic liquid sampler (ALS). The electron impact (EI)-mass spectra were acquired at 70 eV in full scan mode (scan range from m/z = 50–850) as well as in Single Ion Monitoring (SIM) on selected ions of interest. Identification of compounds were done using their relative retention times as well as with comparison with mass spectra recorded in literature. Concentration of n-alkanes were calculated using the Relative Response Factors (RRF) that was determined by a 5-point calibration, using a quantitative standard mixture that contains n-alkane and isoprenoid.

Different biomarker parameters obtained from the analyses and their derivations which include the pristane/phytane ratio, isoprenoid/n-alkanes ratio and the Carbon Preference Index (CPI) can be correlated together for palaeodepositional environment interpretation. The Carbon Preference Index (CPI) is a derivative from biomarkers results that can be referred to in indicating the thermal maturity of samples (Peters et al., 2007b). A thermally immature organic matter is indicated by CPI values that are significantly high or containing values that are less than 1.0 (Peters et al., 2005). CPI is calculated through the formula:

$$CPI = 0.5 \times \left( \frac{C_{25} + C_{27} + C_{29} + C_{31} + C_{33}}{C_{24} + C_{26} + C_{28} + C_{30} + C_{32}} \right. \left. + \frac{C_{25} + C_{27} + C_{29} + C_{31} + C_{33}}{C_{26} + C_{28} + C_{30} + C_{32} + C_{34}} \right)$$

**Results**

**Lithological features**

Sedimentary structures, field-based lithology and other prominent features as observed on outcrops were recorded before channel sampling was carried out. Sediments with organogenic and organic-rich characteristics were selected for sampling; these vary from coals to carbonaceous rocks and shales. The lithological columns of selected outcrops are constructed to show the sample collection site, lithological variations and sedimentary structures recorded (Figs. 3, 4, and 5).

Preliminary classification of the samples based on lithology can be further improved with the use of ash content of the samples, which is a representative of mineral matter input of the samples (Diessel 1992). The results show that MR1-1 and MR1-3 samples from terrace deposits have ash content of 11.3% and 90.8% respectively. In the Liang Formation, coal samples have ash content ranging from 4.74 to 12.2%, with the highest ash content identified in sample BR2-2. A very low ash content of 0.64% is identified in LU1-1 sample of the Miri Formation. In the Setap Shale Formation, ash content of 92.3% is detected.

Ash values detected in the samples can be plotted on the classification of coal introduced by ECE-UN (1998) as displayed in Fig. 6. According to the classification, most of the coals from terrace deposits, Liang and Miri formations are characterized as high-grade coals due to the low ash content. On the contrary, the high ash value of more than 90% has led to samples JD1-3, MR1-3 and DD2-1 being characterized as non-carbonaceous rocks (Fig. 6).

**Rock–Eval pyrolysis and geochemical parameters**

The TOC value for the MR1-1 coal sample is 54.8%, while the lignitic sand samples have TOC readings that range from 3.2 to 4.13%. The TOC values for samples of the Liang Formation ranges from 1 to 48.9%, with the highest TOC value recorded in BR2-1 coal sample. All coal samples in the formation display high TOC values that averages to more than 40%, while the coaly shale, lignitic
sand and shale samples contain lower TOC readings that range from 1 to 2.65%. The average TOC value for coal samples of the Miri Formation is 59.3%, while the shale samples record readings that range from 0.61 to 1.41%. The Setap Shale Formation contains very low TOC and $S_1$ values ranges from 0.03 to 0.07 mg HC/g and $S_2$ values ranging between 0.61 and 0.75 mg HC/g rock.

The $S_1$ and $S_2$ values for terrace deposits range from 0.04 to 5.81 mg HC/g rock and 0.55 to 105.9 mg HC/g rock respectively, with the highest readings for both parameters identified in MR1-1 sample. In the Liang Formation, the $S_1$ and $S_2$ values are ranging from 0.03 mg HC/g to 33.5 mg HC/g and 0.24 mg HC/g rock to 45.3 mg HC/g rock respectively. Low $S_1$ readings are recorded for the coal and shale samples of the Miri Formation, amounting up to 1.42 mg HC/g rock. Shale samples display low $S_2$ readings that are less than 1 mg HC/g rock, while the $S_2$ values for coal ranges from 36.3 to 121.5 mg HC/g rock.

$T_{\text{max}}$ is the temperature with the maximum generation of hydrocarbon. In the terrace deposits, $T_{\text{max}}$ values range from 355 to 409 °C, while the $T_{\text{max}}$ values for the Liang Formation are ranging from 300 to 424 °C for all studied lithology. The $T_{\text{max}}$ readings recorded for the Miri Formation ranges between 378 and 433 °C. In the Setap Shale Formation, relatively high $T_{\text{max}}$ readings are recorded—with readings ranging from 432 to 437 °C.

The PI values for the terrace deposits are low, with values that range from 0.05 to 0.16. The PI values of the Liang
Formation are recorded to range from 0.07 to 0.43, with the highest reading identified in BR2-5 sample. All samples of the Miri Formation contain low PI values that range from 0.01 to 0.07. PI values in the Setap Shale averages to an amount of 0.79% and 0.08 respectively.

In the terrace deposit, coal sample MR1-1 has a high HI reading of 193 mg HC/g TOC but a low OI of 29 mg CO2/g TOC. Alternatively, the lignitic sand samples record high OI values that range from 209—252 CO2/g TOC and low HI readings that range from 14 to 17 mg HC/g TOC. The HI values vary from 18 to 95 mg HC/g TOC, while the OI values for the Liang Formation are between 68 mg CO2/g TOC and 229 mg CO2/g TOC. A wide range of HI and OI values are recorded within the studied formations, with the coals of terrace deposit and Miri Formation containing high values of more than 100 mg HC/g TOC. Values for $T_{\text{max}}$ in coal samples from Liang and Miri formations as well as terrace deposits range from 300 to 404 °C in general, with the Setap Shale Formation having high $T_{\text{max}}$ readings. The geochemical results are summarized in Table 2.

**Organic petrography**

Selected samples belonging to the terrace deposits, Liang, Miri and Setap Shale formations are analysed for the assemblage and abundance of macerals, as summarized in Table 3. For the terrace deposits, Liang and Miri formations, huminite is the predominant maceral group, averaging to 89.3%, 95.5% and 72.5% respectively. In contrast, mineral matter component constitutes the highest constituent in Setap Shale
Formation, with an average value of 73%, followed by huminite which makes up an average of 46%.

Eu-ulminite B is the most abundant maceral in MR1-1 coal sample, comprising a value of 29.5% (Figs. 7a, b, c and e). This maceral is composed of closed cell lumens and cell walls that are created during humification (Diessel 1992). This maceral is also abundant in the Liang Formation, amounting to 73% as detected in BR2-1 sample (Figs. 9a, b, d and e). Eu-ulminite B is also present in the Miri Formation with values ranging from 9 to 80% (Fig. 10a).

Densinite is the second most abundant maceral in MR1-1 coal sample, having an average value of 18.5%. (Figs. 7a, b and d; 8a and c). The maceral densinite is composed of gelified small particles cemented by amorphous humic matter, with no fluorescence. However, this maceral is absent in the Liang Formation but are present in the Miri Formation, with values ranging from 2.5 to 36.5% (Figs. 10a, c and d).

Similarly, attrinite is also absent in the Liang Formation but recorded in terrace deposits and Miri Formation. Attrinite comprises of a mixture of fine huminitic particles and ungelified amorphous huminitic substances (Sýkorová et al. 2005). In the MR1-1 and MR1-3 samples, attrinite is recorded with an average value of 11.5% and 7.5% respectively (Figs. 7d and e). In the Miri Formation, this maceral is present with values that range from 1 to 38.6%.

Corpohuminite, which is made up of structureless features with filled with humic materials, is grey in colour and does not fluoresce (Sýkorová et al. 2005). This maceral averages to 7.5% in the coal sample of terrace deposits (Fig. 7e) and 23% in the Liang Formation, being the most abundant in samples BR2-1 and BR2-3 (Figs. 9a, c and d). In the Miri Formation, corpohuminite is present at 11% to 19% in composition.

Gelinite is a maceral formed from the reprecipitation of dissolved organic matter in a gel form. It is most abundant in MR1-3 sample of terrace deposit (81.5%) and averages to 2% in the Liang Formation (Fig. 9b). In the Miri Formation, gelinite is also present (2.8%).

The second most abundant maceral group present in the coals of terrace deposits, Liang, Miri formations is liptinite, constituting an average of 14%, 7.67% and 13.5% respectively. The dominating liptinite maceral in the terrace deposit is sporinite as recorded in MR1-1 sample with a value of 4% (Figs. 8b and d). This maceral is present with an abundance of 1% and 2.5% in the Liang and Miri Formations respectively. Alginite is identified in terrace coal deposit with a value of 3% (Fig. 8b) and is absent in the Liang Formation. This maceral is present with an abundance of 6% in the Miri Formation (Figs. 10a, b and d). Petrographic results indicate the predominance of flattened discs of telaginite of dull yellow fluorescence in the samples.

Cutinite is a maceral that consists of fossil cuticles from leaves and stems (Pickel et al. 2017). This maceral is identified in the terrace coal sample, Liang and Miri Formations,
Table 2  Lithological features, Rock–Eval pyrolysis results, ash yield and vitrinite reflectance of the studied formations

| Fm. name     | Sample No | Lith          | Sample ID | TOC (wt%) | Rock–Eval Pyrolysis | Derivative parameters | VR% | Ash (wt.%) |
|--------------|-----------|---------------|-----------|-----------|----------------------|-----------------------|-----|-----------|
|              |           |               |           |           | S1 (mg HC/g rock)   | S2 (mg CO2/g rock)   | Tmax (°C) | PY | PI | HI (mg HC/g TOC) | OI (mg CO2/g TOC) |
| Terrace deposit 1 | Coal     | MR1-1         | 54.8      | 5.81      | 106       | 16.0               | 382   | 112 | 0.05 | 193 | 29 | 0.40 | 11.3 |
|               | Lignitic Sand | MR1-2        | 3.20      | 0.04      | 0.55      | 6.68               | 409   | 0.59 | 0.07 | 17  | 209 | –   | –   |
|               | MR1-3     |               | 4.13      | 0.11      | 0.57      | 10.4               | 355   | 0.68 | 0.16 | 14  | 252 | 0.30 | 90.8 |
| Liang 2       | Shale     | BR1-1         | 1.00      | 0.03      | 0.24      | 0.68               | 424   | 0.27 | 0.11 | 24  | 68  | –   | –   |
|               | Coal      | BR2-1         | 48.9      | 1.14      | 15.5      | 37.5               | 363   | 16.6 | 0.07 | 32  | 77  | 0.22 | 4.74 |
|               | BR2-2     |               | 41.8      | 1.31      | 7.49      | 95.8               | 347   | 8.80 | 0.15 | 18  | 229 | –   | –   |
|               | BR2-3     |               | 47.9      | 33.5      | 45.3      | 55.4               | 300   | 78.7 | 0.43 | 95  | 116 | 0.32 | 5.02 |
|               | Coaly shale | BR2-5    | 1.28      | 0.11      | 0.69      | 1.79               | 414   | 0.80 | 0.14 | 54  | 140 | –   | –   |
|               | BR2-6     |               | 2.65      | 0.18      | 2.18      | 2.50               | 408   | 2.36 | 0.08 | 82  | 94  | –   | –   |
| Mihi 4        | Coal      | LU1-1         | 62.4      | 1.41      | 122       | 9.38               | 404   | 123  | 0.01 | 195 | 15  | 0.46 | 0.64 |
|               | TL1-1     |               | 56.3      | 1.42      | 36.3      | 33.8               | 378   | 37.7 | 0.04 | 64  | 60  | 0.32 | 7.85 |
|               | TN1-1     |               | 0.61      | 0.01      | 0.29      | 0.48               | 433   | 0.30 | 0.02 | 48  | 79  | –   | –   |
|               | DD1-1     |               | 1.41      | 0.04      | 0.55      | 0.52               | 418   | 0.59 | 0.07 | 39  | 37  | –   | 92.5 |
| Setap Shale 8 | Shale     | JD1-1         | 0.78      | 0.03      | 0.65      | 0.19               | 435   | 0.68 | 0.05 | 83  | 24  | –   | –   |
|               | JD1-2     |               | 0.79      | 0.07      | 0.61      | 0.28               | 437   | 0.68 | 0.10 | 77  | 35  | –   | –   |
|               | JD1-3     |               | 0.81      | 0.08      | 0.75      | 0.32               | 432   | 0.83 | 0.10 | 93  | 40  | –   | 92.3 |
with abundances amounting up to 5.3% (Figs. 7e and f; Figs. 8c, e and f; Fig. 9c and e; Figs. 10a, d, e and f).

Suberinite is a maceral that originates from suberin of cork tissues (Pickel et al. 2017). This maceral makes up 1.5% of the terrace deposit coal sample. In the Liang Formation, suberinite is recorded to have an abundance of 2.5%. Maceral analysis indicates that the Miri Formation is made up of 6% of suberinite. Other liptinite macerals present are resinite (Figs. 10c and d), bituminite and liptodetrinite.

Inertinite macerals are present in all studied deposits, except for the samples in the Liang Formation. Inertodetrinite is composed of small inertinite fragments that are remnants produced from wildfires (ICCP 2001). This maceral is amounting to 2%, 16% and 20% in the terrace deposits, Miri and Setup Shale Formations respectively.

The most abundant inertinite maceral in the terrace deposit is funginite, a maceral that consists of fungal remains, including fungal spores, hyphae, and mycelia (ICCP 2001). Funginite is most abundant in MR1-1 sample, amounting up to 3.5% (Figs. 7a, b and d). Funginite makes up 6% of the Miri Formation (Fig. 9f; Fig. 10c). Semifusinite is made of woody tissues that are partially heated and constitutes an abundance of 2% in both terrace deposits and Miri Formations (Fig. 9f).

Carbonates are detected in terrace deposits, Miri and Setup Shale Formations, with the highest volume recorded in the MR1-3 sample of the terrace deposit. Pyrite is identified with an average value of 3%, 1%, 3.13% and 2% in the terrace deposits, Liang, Miri and Setup Shale formations respectively.

| Table 3 | Organic petrographic results showing the maceral constitution of the studied samples |
| Formation name | Terrace deposits | Liang | Miri | Setup Shale |
| Sample ID/Maceral type | MR1-1 | MR1-3 | BR2-1 | v | LU11 | TL1-1 | DD2-1 | JD1-3 |
| Huminite | Telohuminite | 1.0 | – | – | – | – | – | – | – |
| | Texto-ulminite | 4.0 | – | – | – | – | – | – | 2.0 |
| | Eu-ulminite A | – | – | – | – | – | – | 2.0 | – |
| | Eu-ulminite B | 29.5 | 5.5 | 73.0 | 70.0 | 10.5 | 80.0 | 9.0 | – |
| Total | 34.5 | 5.5 | 73.0 | 70.0 | 10.5 | 80.0 | 13.0 | 0.0 |
| Detrohuminite | Attrinite | 11.5 | 7.5 | – | – | 1.0 | – | 38.5 | 46.0 |
| | Densinite | 18.5 | 2.0 | – | – | 36.5 | – | 2.5 | – |
| Total | 30.0 | 9.5 | – | – | 37.5 | – | 41.0 | 46.0 |
| Gelohuminite | Gelinite | 2.5 | 81.5 | 2.0 | – | 3.0 | – | 2.5 | – |
| | Corphuminite | 11.5 | 3.5 | 23.0 | 23.0 | 11.0 | 19.0 | – | – |
| Total | 14.0 | 85.0 | 25.0 | 23.0 | 14.0 | 19.0 | 2.5 | – |
| Total | 78.5 | 100.0 | 98.0 | 93.0 | 62.0 | 99.0 | 56.5 | 46.0 |
| Inertinite | Fusinite | – | – | – | – | – | 9.0 | – |
| | Semifusinite | 2.0 | – | – | – | 2.0 | – | – | – |
| | Funginite | 3.5 | – | – | – | 6.0 | – | – | – |
| | Secretinite | 0.5 | – | – | – | 0.5 | – | – | – |
| | Inertodetrinite | 1.5 | – | – | – | 3.5 | – | 16.0 | 20.0 |
| Total | 7.5 | – | – | – | 12.0 | – | 25.0 | 20.0 |
| Total | 14.0 | 0.0 | 2.0 | 7.0 | 26.0 | 1.0 | 18.5 | 34.0 |
| Liptinite | Sporinite | 4.0 | – | – | 1.0 | 2.0 | – | 2.5 | – |
| | Cutinite | 2.0 | – | – | 1.0 | 8.0 | – | 2.5 | – |
| | Alginite | 3.0 | – | – | – | 6.0 | – | – | 8.0 |
| | Suberinite | 1.5 | – | 2.0 | 5.0 | 6.0 | – | – | – |
| | Resinite | 1.5 | – | – | – | 1.0 | – | – | – |
| | Bituminite | – | – | – | – | – | 4.5 | 3.0 | – |
| | Liptodetrinite | 2.0 | – | – | – | 3.0 | 1.0 | 9.0 | 23.0 |
| Total | 14.0 | 0.0 | 1.0 | 7.0 | 26.0 | 1.0 | 18.5 | 34.0 |
| Mineral matter | Carbonates | 0.5 | 15.0 | – | – | – | – | 6.9 | 1.0 |
| | Pyrite | 3.0 | – | 1.0 | 1.0 | 3.0 | 3.0 | 3.4 | 2.0 |
| | Clays | – | – | – | – | 2.0 | – | 68.0 | 70.0 |
| | Other silicates | – | 50.0 | – | – | – | – | – | – |
| Total | 3.6 | 65.0 | 1.0 | 1.0 | 5.0 | 3.0 | 78.3 | 73.0 |
respectively. For Miri and Setap Shale Formations, clay constitutes more than 50% in the samples.

As a deep marine formation, very few macerals are detected in the samples of the Setap Shale Formation. Huminite and inertinite macerals are identified as fragments within the mainly clayey matrix of the sample. The sample records 46% of attrinite, followed by 23% of liptodetrinite and 20% of inertodetrinite. The sample is dominated by clay (70%), with pyrite and carbonates occurring in low amounts of 2% and 1% respectively. The clay matrix is observed to be fluorescing, indicating the possibility of bituminite within the sample.

In this study, the mean random vitrinite reflectance for the terrace deposits, Liang and Miri formations were measured in eu-ulminite and recorded to range from 0.22 to 0.46% (Table 3). Ash yields of selected samples show that very high ash values of more than 90% are recorded for MR1-3, DD2-1 and JD1-3. Other samples contain ash yields that range from 0.64 to 12.2%, with the lowest value belonging to LU1-1 sample.

**Biomarker distribution**

To further validate results obtained from Rock–Eval pyrolysis and organic petrography to, a total of five samples were selected for biomarkers analysis. One sample derived from terrace deposit (MR1-1), two samples obtained from the Liang Formation (BR2-1 and BR2-3), one sample derived from the Miri Formation (LU1-1) and one sample taken from the Setap Shale Formation (JD1-3) were chosen for biomarkers analysis.

Biological markers, or best known as biomarkers, are composed of complex molecular fossils generated from once-living organisms and can be found in source rock extracts (Peters et al. 2007a). Biomarker data are one of the
important means to assess a source rock’s palaeodepositional environment and thermal maturity.

**Rock extraction and fractionation**

The coal sample from terrace deposit records a high extract yield of 74.5 mg HC/g rock. Among the two coal samples from the Liang Formation, sample BR2-3 has the highest extract yield (bitumen) with a value of 89.3 mg HC/g rock. In the Miri Formation, sample LU1-1 also displays a high extract yield value of 57.1 mg HC/g rock. Sample JD1-3 displays a very low extract yield of less than 1 mg HC/g rock. For all samples except JD1-3, there is a higher percentage of asphaltene over maltene (Table 4).

Among the analyzed samples, the highest aromatic content was identified in sample MR1-1, with a value of 45% while sample BR2-1 is composed of almost entirely of asphaltene (99%). Polar compounds are generally high for samples MR1-1, BR2-3 and LU1-1, with the highest value identified with BR2-3 of 82% (Table 4). For the Miri Formation, sample LU1-1 displays high asphaltene (95%) and polar compounds (61%).

**n-alkanes and isoprenoids**

Gas chromatogram fingerprints of $n$-alkanes is used to identify source of organic matter (Peters et al. 2007a), by examining the mode and skewness of $n$-alkanes identified in a sample. Organic matter associated with a terrestrial source display a bimodal distribution of $n$-alkanes and skew toward $n$-C$_{23}$–$n$-C$_{31}$ (Eglinton and Hamilton 1967). Lacustrine and marine oils from shaly source rocks are reflected by the odd-predominance of $n$-alkanes (Peters et al. 2007a).
Distribution of \( n \)-alkanes for selected samples from terrace deposits, Liang and Miri formations are illustrated in Fig. 11. The GC–MS chromatograms of sample MR1-1 show an evident bi-modal distribution with high concentrations of \( n \)-\text{C}_{27}, \( n \)-\text{C}_{29}, \( n \)-\text{C}_{31} and \( n \)-\text{C}_{33} (Fig. 12a). Meanwhile, samples BR2-3 of the Liang Formation and LU1-1 of the Miri Formation display a normal distribution of \( n \)-alkanes (Figs. 11b and c). Among the studied samples, sample LU1-1 has the highest pristane (Pr) and phytane (Ph) values of 15.7 and 1.4 respectively, leading to high Pr/Ph value of 11.2 (Table 5).

**Discussion**

**Type of organic matter–kerogen type**

Sediments of the terrace deposit, Liang, Miri and Setap Shale formations are classified based on the ECE-UN (1998) classification scheme which takes into account the sediments’ ash content and vitrinite reflectance values (Fig. 6). Ash content of the studied Formations identify the coals as high grade (BR2-1, BR2-3, LU1-1 and TL1-1) to medium grade (MR1-1). Meanwhile, the shale samples of Miri and Setap Shale formations are characterized as carbonaceous rocks due to the high ash yields of the samples exceeding 90% (Fig. 6).

Proportions of main maceral groups present in the studied deposits gave an impression regarding possible kerogen types present in the studied formations. A ternary diagram involving the main maceral groups in Fig. 12 shows that the
coal and lignitic sand samples of the terrace deposits contain kerogen type III (gas prone) due to the predominance of huminite, together with BR2-1 and BR2-2 of the Liang Formation. The coals of the Miri Formation contain kerogen type II-III (LU1-1) and type III (TL1-1), as a result of liptinite abundance.

Several parameters obtained from Rock–Eval pyrolysis, such as TOC, S₂, HI and OI are utilized to further evaluate the kerogen types present in the studied formations. In a cross-plot of S₂ versus TOC (Fig. 13), the kerogen types present in samples of different lithologies from the studied formations vary from dry gas prone, type II-III (mixed oil and gas) and type III (gas prone). The MR1-1 coal sample from the terrace deposit contain kerogen type II-III (mixed oil gas prone), while the lignitic sand samples are dry gas prone. In the Liang Formation, kerogen type III is present

### Table 4

| Formation   | Sample | Extract yield (mg/g) | Maltenes (wt.%) | Asphaltenes | Saturates (wt.%) | Aromatics | NSO |
|-------------|--------|----------------------|-----------------|-------------|-----------------|-----------|-----|
| Terrace deposits | MR1-1  | 74.46                | 35.8            | 64.2        | 12              | 45        | 43  |
| Liang       | BR2-1  | 23.11                | 0.6             | 99.4        | –               | –         | –   |
|             | BR2-3  | 89.34                | 15.8            | 84.2        | 3               | 14        | 82  |
| Miri        | LU1-1  | 57.12                | 4.5             | 95.5        | 21              | 18        | 61  |
| Setup Shale | JD1-3  | 0.60                 | –               | –           | –               | –         | –   |
Fig. 11  a, b and c Gas chromatograms of saturate fractions of samples from terrace (a), Liang (b) and Miri (c) Formations

Fig. 12  A ternary diagram illustrating the type of kerogen available in the terrace deposit, Liang and Miri formations using the distribution of maceral composition (modified after Tissot and Welte 1984)

| Table 5  Calculated n-alkane and isoprenoid ratios for the studied samples |
|-------------------------------------------------|-------------|-------------|-------------|-------------|-------------|-------------|-------------|-------------|
| Formation            | Sample | Pr/Ph | Pr/nC<sub>17</sub> | Ph/nC<sub>18</sub> | R<sub>22</sub> | P<sub>47</sub> | nC<sub>19</sub>/nC<sub>31</sub> | CPI         |
| Terrace deposits     | MR1-1   | 6.81   | 7              | 1.14           | 1.05         | 0.15         | 0.034        | 3.77        |
| Liang                | BR2-3   | –      | 0              | 0              | 1.86         | 0.39         | 0            | 2.11        |
| Miri                 | LU1-1   | 11.2   | 28.5           | 2.46           | 1.36         | 0.16         | 0.018        | 2.64        |
in samples BR2-3, BR2-5 and BR2-6, whereas dry gas is present in BR2-1, BR2-2 and BR1-1 samples. The LU1-1 and TL1-1 coal samples from the Miri Formation contain kerogen types II-III (mixed oil and gas prone) and type III (gas prone). However, the shale samples of this formation contain dry gas. All Setap Shale Formation samples are gas prone.

A more precise method of identifying types of organic matter present in sample is accomplished by using the modified Van Krevelen diagram, which utilizes the parameters HI and OI. The cross-plot between both parameters in Fig. 14 agrees with the results in Fig. 13, in which the LU1-1 and MR1-1 samples of the Miri Formation and terrace deposit respectively contain kerogen type II-III (mixed oil and gas) and type III (gas prone). Kerogen type III (gas prone) is present for TL1-1 (Miri Formation), BR2-3 (Liang Formation), BR2-6 (Liang Formation) and all Setap Shale samples. For the rest of the samples, they fall under the region of kerogen type IV, which is equivalent to dry gas.

**Palaeodepositional environment**

The types, abundance and distribution of macerals present in coals will reflect the palaeofacies and conditions in which the peat was previously deposited (Shalaby et al. 2019). In this study, two facies models are constructed according to Calder et al. (1991) and Diessel (1992), modified by Kalaitzidis et al. (2004; 2010). Four parameters are examined in these coal facies diagrams; tissue preservation index (TPI), gelification index (GI), Groundwater index (GWI) and vegetation index (VI).

The state of plant tissue preservation is demonstrated in TPI, whereas GI reflects the degree of wet or dry conditions in the peat-forming site (Diessel 1992). Wet environments in peat formation is indicated by high GI values, which can be affected by factors such as basin subsidence rate and groundwater table changes (Diessel 1992). In this study, the TPI and GI values are calculated based on Kalaitzidis et al. (2004; 2010) to reflect low rank coals. The coal samples in this study contain TPI values that range from 0 to 1.4, except
samples BR2-1, BR2-3 and TL1-1, in which very high values are identified. A range of GI values are recorded, reflecting different degrees of gelification, with samples BR2-1, BR2-3 and TL1-1 containing the highest values of more than 80. All calculated TPI and GI values from the terrace deposit, Liang and Miri formations coal samples are plotted together in a cross-plot between GI versus TPI in Fig. 15 (a), to illustrate the depositional facies of the samples. An overall assessment indicates that the coals are generally deposited in a lower to upper delta plain setting under limnic to limnotelmatm conditions. Contrary to the sample lithology, DD2-1 is shown to be deposited in an environment that is less water-saturated.

The hydrological state of a palaeomire is a useful tool to assess the depositional facies of coals, as changes in water input can cause alteration in the maceral composition. Therefore, a cross-plot of the groundwater index and vegetation index is constructed to illustrate the hydrological regime of a palaeomire (Fig. 15b). In this study, the GWI values for terrace deposit are 0.784 and 11.7, while an average value of 0.35 is recorded for the Liang Formation. The Miri Formation records GWI ranging from 0.28 to 4.85.

The high GI, TPI and VI values identified in BR2-1, BR2-3 and TL1-1 are attributed to the preservation of root systems which are predominant in these samples. For the rest of the samples for the studied formations, the main plant input is of herbaceous plants which is reflected by the low VI values. The site of deposition for most of the coal sample has a high water table with possible marine influence as reflected by the presence of pyrite in most samples. This is fitting with the lower to upper delta plain setting as identified in the cross-plots.

Several ratios based on gas chromatographic results can be used for studying the palaeodepositional conditions. Ratio
of pristane to phytane (Pr/Ph) is a useful tool to detect the depositional facies of coals, in which a Pr/Ph value of less than 1 indicates a reducing and anoxic environment (Powell and McKirdy 1973; Didyk et al. 1978). An oxic to suboxic depositional condition is identified from a Pr/Ph value of more than 3 (Peters et al. 2007b), whereas a value of more than 10 is indicative of a peat swamp environment. In this study, samples MR1-1 and LU1-1 have Pr/Ph values of 6.81 and 11.2 respectively, indicating terrigenous organic matter deposited in an oxic condition.

The relationship of pristane and phytane with other n-alkanes can indicate the thermal maturity of the studied samples. A cross-plot of Ph/n-C18 versus Pr/n-C17 in Fig. 16 indicates that the coal samples from the terrace deposit and Miri formations are derived from terrigenous organic matter and these samples are thermally immature. The R22 index indicates the salinity of a depositional setting and it is defined through the formula $2 \times \frac{C_{22}}{(C_{21} + C_{23})}$ (ten Haven et al. 1988). All the analyzed samples display low R22 index values of less than 2, with the highest value of 1.86 identified in sample BR2-3 of the Liang Formation.
Use of $P_{aq}$ ratio can also contribute to palaeodepositional environment analysis as this index illustrates the hydrological regime of depositional site of the studied sample (Ficken et al. 2000). The $P_{aq}$ ratio is defined as $(nC_{23} + nC_{25})/(nC_{23} + nC_{25} + nC_{29} + nC_{31})$ and acts as a proxy for submerged/floating aquatic macrophyte versus terrestrial plant input to lake sediments that is retrieved from n-alkane information (Ficken et al. 2000). Terrestrial land input is indicated by low $P_{aq}$ values of less than 0.1, whereas values ranging from 0.1 to 0.4 suggest emergent macrophytes. In this study, the $P_{aq}$ values for the samples contain values ranging from 0.14 to 0.39, suggesting the presence of terrestrial, emergent plant input in the depositional site. This is parallel with primarily low vegetation index values (with the exception of BR2-1, BR2-3 and TL1-1 samples) calculated from maceral abundances as displayed in Fig. 15(b).

The Miocene-Quaternary formations as source rocks

Hydrocarbon generation potential

The generating potential of all selected samples from the studied formations are assessed through the relationship between $S_2$ and TOC (Fig. 17). The cross-plot exhibits the “excellent” generating potential for terrace coals, whereas the lignitic sand samples have “poor” potential of hydrocarbon generation. In the Liang Formation, the coal samples have a “good to excellent” generating potential and “poor” generation potential for the coaly shale as well as shale samples. An “excellent” generating potential is also observed in the Miri Formation coal samples and a “poor” generating potential is identified for the shale samples of the same formation. The Setap Shale Formation records no potential in hydrocarbon generating potential due to the low $S_2$ and TOC values.

Thermal maturity

Vitrinite reflectance is a parameter that can be utilized to indicate the evaluation of source rock thermal maturity as a result of its sensitivity to temperature changes (Jumat et al. 2018; Osli et al. 2019). In this study, the ECE-UN (1998) classification utilizes vitrinite reflectance values of selected coal and coaly shale samples from studied formations in order to rank the samples. The classification in Fig. 6 shows that the samples have vitrinite reflectance readings that range from 0.30 to 0.46%, giving a sub-bituminous to medium rank D for the samples.

Other maturity-related parameters to be applied in thermal maturity evaluation include $T_{max}$ and PI. The distribution of thermal maturity for studied samples are illustrated in a cross-plot of HI versus $T_{max}$ (Fig. 18), which shows that majority of the samples lies within the “immature” window. All samples belonging to the Setap Shale Formation,
Fig. 18  A cross-plot of HI versus $T_{\text{max}}$ to indicate the thermal maturity state of the studied formations

Fig. 19  A cross-plot of $T_{\text{max}}$ versus $\%R_o$ to show the thermal maturity of the studied formations
together with TN1-1 and KL1-1 of the Miri Formation, are within the thermally mature zone. Additionally, the low thermal maturity state of the terrace deposit, Liang and Miri coal samples are similarly identified in a cross-plot of \( R^\text{max} \) versus vitrinite reflectance in Fig. 19. The low thermal maturity state of the selected samples from the studied formations are also confirmed by the CPI values that are ranging from 2.11 to 3.77.

**Conclusion**

This study investigated the geochemical characteristics and organic petrographical properties of the terrace deposits, Liang, Miri and Setap Shale formations. Abundance of macerals and Rock–Eval pyrolysis results indicate that the dominating kerogen types identified in the samples of these formations is kerogen type III (gas-prone) due to abundance of huminite. Minor kerogen type II–III (oil and gas prone) is also identified as a result of liptinite being the second most abundant maceral in the studied formations. Maceral analysis and geochemical characterization results conclude that the coal samples of the studied Formations are identified to be deposited in a mangrove setting that is exposed to marine influence. This is further supported by the bi-modal and normal distribution of \( n \)-alkanes in the studied samples. Although these outcrops samples are thermally immature, the best source rock generation potential among the four deposits belong to coals, whereas any non-coal layers are of fair to poor potential.

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