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### Research paper

# Higher plant biomarker signatures of Early Eocene sediments of North Eastern India

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#### ABSTRACT

The biomarkers, molecular fossils of paleobiota, have been studied in detail from a coal-bearing sequence of Early Eocene Lakadong sandstone member of Sylhet Formation of Upper Assam Basin, NE India, using gas chromatography-mass spectrometry. Corresponding hydrocarbon source potential of the same unit has been studied using Rock-Eval pyrolysis. The Rock-Eval data suggest dominant terrestrial land plant contribution, i.e., Type III kerogen and the sediments of the section had entered the early stage of catagenesis. The alkane and isoprenoid distributions are characterized by odd over even preference (OEP) (1.1–1.2) of *n*-alkanes and high pristane/phytane ratio (2.9–7.6) which corroborate the Rock-Eval data. The distribution of aliphatic biomarkers is characterized by dominance of  $C_{24}$  tetracyclics over  $C_{23}$  tricyclics, high concentration of des-E hopane, moderate to high  $C_{29}/C_{30}$  hopane ratio, extremely low oleanane index and low  $C_{35}$  homohopane index. The extremely low oleanane index is correlated with a high degree of aromatization of oleanane skeleton, favoured by acidic swampy depositional environment. The presence of diterpanes at the base of the Early Eocene section indicates contribution from gymnosperms (Coniferales order of land plants) whereas angiospermous biomarkers with oleanoid skeleton are also preserved. This pattern subsequently evolved to angiosperm dominated towards the top of the section with no gymnosperm signature indicating possible climatic variation during the Early Eocene. Towards the top of the section presence of bicadinanes indicates contribution of Dipterocarpaceae family of trees and advent of the evergreen tropical rain forest element in eastern India in Early Eocene, similar to the pattern found in western India, e.g., Cambay and Kutch basins.

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#### 1. Introduction

Paleogene coal-bearing shales have long been identified as a proven source facies for the south-east Asian fluvio-deltaic oils (Curiale et al., 1994 and references therein; Murray et al., 1994 and references therein). The Paleogene basins in this region are recognized as complete petroleum systems and among different types of Paleogene source rock facies, fluvio-deltaic ones are of most importance (Sudarmono and Eza, 1997). Geologically as well as geographically adjacent to these basins is the Assam Basin of North East India, a well explored petroleum province in Indian for more than a century. With a polyphase tectonic history, Tertiary sediments in the shelf zone of the basin are thick, ranging from 3.6 km to over 7 km, and include fluviatile to shallow marine Paleogene

and continental fluviatile Neogene sediments overlying the granitic basement (Handique and Mallick, 1990). During a major part of the Late Cretaceous and early Paleogene, the shelf zone of the Assam Basin was a passive continental margin with a predominantly nearshore to shallow marine depositional environment (Jauhri et al., 2006). Changes in the depositional setting of the shelf zone to a deltaic-estuarine one happened during Eocene and Oligocene, followed by a fluviatile phase in Miocene and younger times (Mathur et al., 2001). A regional stratigraphic chart of Upper Assam Basin has been provided in Figure 1.There are two active petroleum systems in this basin, Paleocene-Eocene Lakadong-Langpar system (Raju and Mathur, 1995) and Eocene-Oligocene Barail-Kopili system (Goswami et al., 2005). The early Paleogene shelf and marginal marine sediments, i.e., members of Sylhet Formation (Late Paleocene to Mid Eocene) are preserved as outcrops along the southern fringes of the Shillong plateau bounded by Dauki fault at south (Sahni and Kumar 1974; Garg and Jain, 1995; Prasad et al., 2006). The early Paleogene outcrop section is equivalent to the







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**Figure 1.** Generalized stratigraphic chart of Upper Assam Basin (after Raju and Mathur, 1995; Mathur et al., 2001).

subcrop stratigraphy of Upper Assam Basin, which has contributed to the source potential of the Lakadong–Langpar petroleum system (Singh et al., 1986). To the best of our knowledge, the present study represents the first detailed organic geochemical study of the outcrop of the Lakadong sandstone member. The present study is focussed on a detailed organic geochemical characterization of the coal and dark carbonaceous shales intercalated in the Early Eocene Lakadong Sandstone member of Sylhet Formation by means of Rock-Eval pyrolysis and GC–MS, to evaluate hydrocarbon source potential of the coal-bearing sediments as well as to reveal the paleobotanical precursors of the organic matter of the Early Eocene prevailed in the studied area.

#### 2. Samples and methods

#### 2.1. Samples

The coal and dark shale samples were collected from the Lakadong sandstone member of the Lakadong Formation (Fig. 1) exposed along the hilly terrain, at the Mawmluh limestone quarry (25°15′23.8″ N: 91°42′50.1″ E) about 4 km south of Cherrapunji in the East Khasi Hill district of Meghalaya, NE India. The Lakadong sandstone member is a marginal marine lithounit at the bottom which gradually changes to fluvial with shallow channel sands towards top and with several intercalations of thin coal and dark shale beds, thickness ranging between 0.5 and 1 m. The overall thickness of Lakadong sandstone member in Mawmluh quarry section is 45 m (Fig. 2). The Lakadong sandstone overlies the Lakadong limestone member (Tewari et al., 2010), which is rich in microfossils with an age range from Late Paleocene to Early Eocene (Jauhri et al., 2006).

The samples were collected from the Mawmluh Limestone quarry section outcrop. The section is relatively fresh as removal of overburden has been carried out by mine authority very recently. No visual weathered profile was observed while studying the section. A suite of ten representative samples collected from the Mawmluh section were used for Rock-Eval pyrolysis analysis and biomarker analyses.

#### 2.2. Rock-Eval pyrolysis

Pyrolysis experiments were conducted using a "Standard" Rock-Eval 6 pyrolyser. Briefly, samples were first pyrolysed under an inert N<sub>2</sub> atmosphere and the residual carbon was subsequently burnt in an oxidation oven. The amount of hydrocarbons (HC) released during pyrolysis was detected with a flame ionization detector (FID), while online infrared detector measures continuously the released CO and CO<sub>2</sub>. The samples were first pyrolysed from 300 °C to 650 °C at a rate of 25 °C/min. The oxidation phase starts with an isothermal stage at 300 °C, followed by an increase to 750 °C at a rate of 25 °C/min to incinerate all the residual carbon.

#### 2.3. Sample preparation by column chromatography

The selected rock samples were powdered in sterilized mortar pestle. Prior to use, the glasswares were fired at 300 °C for 4 h and then sterilized in methanol followed by dichloromethane (DCM). A solution of dichloromethane:methanol (9:1) was then added to each powdered sample followed by ultrasonication for 30 min. The extracted solution was decanted after allowing precipitation of the powdered sample. This process was repeated twice to pull the extractable organic matter from the rock to a feasible extent. The precipitation of asphaltenes was performed using *n*-hexane and subsequent freezing and the extracts decanted, were separated into saturate and aromatic fractions by liquid column chromatography using glass pipette ~ 13 cm column packed with activated (120° for >8 h) silica gel (100–200 mesh), aliphatic fraction was eluted with *n*-hexane (3/8 of column volume) and aromatic fraction with a solution of *n*-hexane:DCM (4:1; 4 column volume).

#### 2.4. Gas chromatography–mass spectrometry (GC–MS)

The saturated and aromatic hydrocarbon fractions were analysed by gas chromatography—mass spectrometry (GC—MS) in full scan mode. The analyses were conducted on Agilent 5975 MSD attached with an Agilent 7890 gas chromatograph. The GC was fitted with DB-1 fused silica capillary column (30 m  $\times$  0.25 mm i.d., 0.25 µm film thickness) and alternatively with HP-5MS fused silica capillary column (30 m  $\times$  0.25 mm i.d., 0.25 µm film thickness) and

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