# Assessment of Thermal Maturity of the Mesozoic Organic-Rich Rocks of Southern England.

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## ABSTRACT

This paper assesses the thermal maturity of the Mesozoic organic-rich rocks of Dorset area in the Wessex Basin. The objectives of this study are to sample the organic-rich rock outcrops from the Dorset area of southern England and to evaluate the level of thermal maturity of these organic-rich rocks by organic geochemical analyses.

A total of eight outcrop samples of organic-rich rocks collected from six Mesozoic stratigraphic intervals along the coastline of Dorset were analyzed using Rock-Eval pyrolysis and Gas Chromatography-Mass Spectrometry techniques. The Rock-Eval results suggested that the analysed samples have Tmax values typically less than 435°C, and thus are thermally immature. Biomarker-based thermal maturity ratios such as hopane $C_{31}$-22S/22S+22R, sterane $C_{29}$-20S/20S+20R and trisnorhopane, Ts/Ts+Tm ratios, however, indicated that the Purbeck Black Shale and the Gault Clay samples are thermally mature. The correlation between the pyrolysis Tmax and biomarker parameters (e.g. hopane $C_{31}$-22S/22S+22R ratio) confirmed that the Gault Clay sample has only experienced low thermal stress and cannot be considered thermally mature.

It is concluded that the anomalous maturity based on hopane and sterane isomerization ratios is a result of reworked organic matter incorporated into the sediments of the Gault Clay. It is recommended that besides the Kimmeridge Clay, the Blue Lias, and the Oxford Clay Formations which are considered mature elsewhere in the basin, the Purbeck Black Shale should also be considered for hydrocarbon exploration.

(Keywords: mesozoic, thermal maturity, rock-eval pyrolysis, gas chromatography-mass spectrometry, biomarker)

## INTRODUCTION

The thermal maturity of potential source rocks or oils can be evaluated by a number of geochemical methods such as kerogen analyses (e.g., Vitrinite reflectance measurement, Pyrolysis Tmax, Thermal Alteration Index, Optical activity) and Gas Chromatography-Mass Spectrometry (GC-MS) technique through biomarker studies.

Biomarkers are individual compounds which are precursors of petroleum, and whose chemical structures provide information on the origin of the organic matter, depositional environments or settings, burial history, and the thermal stress the rocks had undergone (Brocks and Summons, 2004). Thus, biomarkers can be used as indicators of the total thermal history of the organic matter, and therefore as indicators of maturity (Wapples and Michihara, 1991).

The GC-MS technique remains an invaluable and reliable method that gives a robust interpretation. Consequently, it is routinely used by oil and gas industries during hydrocarbon exploration to reduce exploration risks. Like any other geochemical methods used in thermal maturity evaluation of source rocks or oils, the Rock-Eval pyrolysis and GC-MS techniques also have their drawbacks especially those related to the origin and nature of organic matter in the former and our confidence in whether the bitumen is indigenous or not in the latter. However, fortunately, the GC-MS technique resolves some issues related to pyrolysis interpretations while various geochemical tests (Extract/TOC ratio, Production Index from pyrolysis and extract composition) are also available to help determine if a given rock is stained or contaminated (Peters, 1986).

Though some of the above-listed geochemical methods have been employed to study thermal maturity of parts of the Mesozoic organic-rich rocks considered in this paper, the Purbeck Black Shale, the Nothe Clay and the Gault Clay Formations have been incredibly neglected for decades, and therefore have scanty data and discourse in the literature.

## LITERATURE REVIEW

Oil exploration in the Wessex Basin starts from the early twentieth century. The successes recorded in the second half of the century especially with the discovery of the Wytch Farm oilfield in the eastern Dorset in 1973 significantly encouraged hydrocarbon exploration throughout the basin (Buchanan, 1998). Subsequently, the potential source rocks in the basin remain the focus of petroleum geochemists as well as oil industries. Thus, they have employed various geochemical methods and otherwise to assess the generation potential and thermal maturity of these rocks. Of these potential source rocks, the Kimmeridge Clay Formation was mostly intensively studied (Gallois, 2004).

Ebukanson and Kinghorn (1985), through their TOC and vitrinite reflectance measurements, showed that the Liassic shales and the Kimmeridge Clay are marginally immature at outcrop, though they contain high organic matter contents. Ebukanson and Kinghorn (1986a) investigated three Jurassic potential source rocks in the Wessex basin namely the Kimmeridge Clay, Oxford Clay and Liassic mudstones, and their maturity studies and burial history modeling indicated that only the Liassic mudstones are sufficiently mature to have generated substantial amounts of oil and gas only in the deeper buried areas. They also added that the Oxford Clay is marginally mature and lie at the top of the oil generation window and that the Kimmeridge Clay is everywhere immature.

Stoneley (1992) reported that in the greater part of the southern Wessex Basin, the potential source rocks are either immature or just at the threshold of oil generation. Smith *et al*., (2010) claimed that the Liassic shales are mature for gas generation. Small gas accumulations (dubbed ‘enigmatic’) occur on the margins of the Weald Basin and that they are associated with oil at Wytch Farm in the Wessex Basin (Butler and Pullan, 1990).

## MATERIALS AND METHODS

This study involved outcrops sampling of the organic-rich rocks along the coastline of Dorset, southern England and total of eight samples were collected as shown in Figure 1 while some of the photos of the outcrops are shown in Figure 2. The samples names and location details are summarized in Table 1. The outcrop samples were taken to the Organic Geochemistry and Petrophysics Laboratory, Imperial College London, where they were oven-dried overnight at 110$^0$C, and pulverized with the aid of pestle and mortar. The crushed rock samples were analyzed geochemically by Rock-Eval pyrolysis and GC-MS techniques.

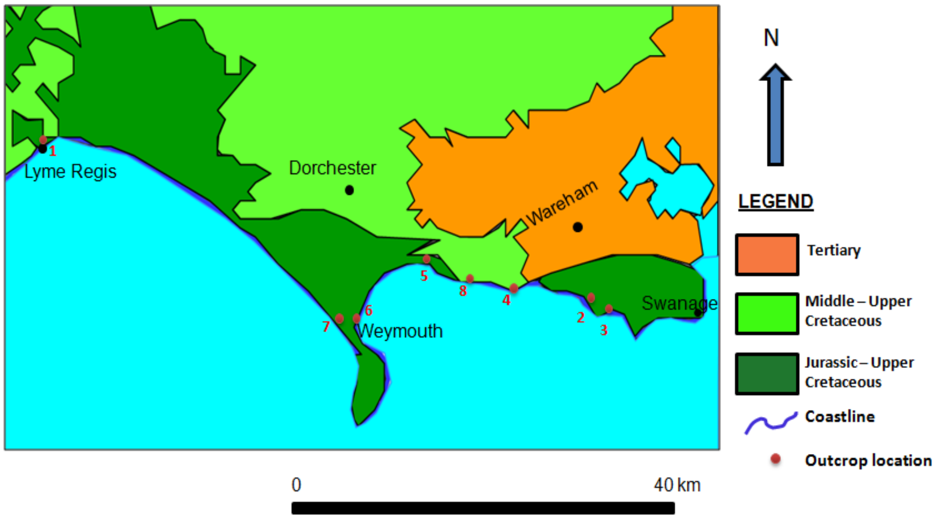
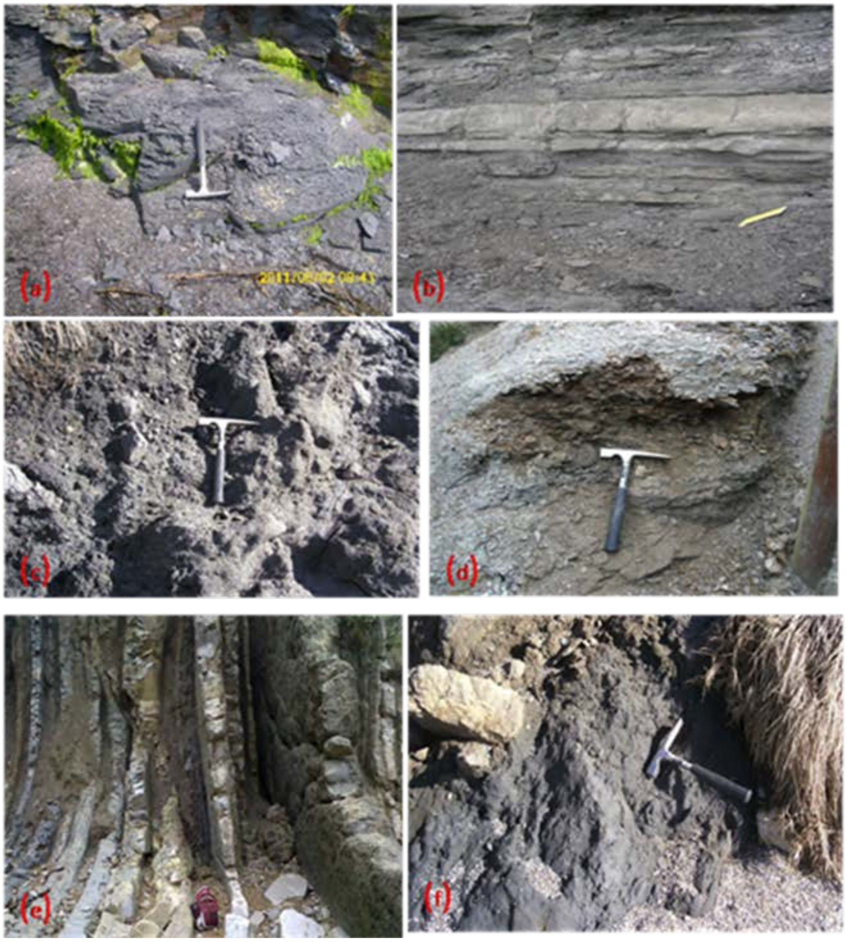


**Figure 1:** The Map of the Study Area with Outcrop Locations (Stoneley and Selley, 1986).



(a). Kimmeridge Clay outcrop at Kimmeridge Bay

(b). The Blue Lias outcrop at Lyme Regis

(c). Nothe Clay outcrop at Osmington Mills, east of Weymouth

(d). Oxford Clay outcrop at Chickerell, Weymouth

(e). Purbeck Black Shale outcrop at Durdle Door

(f). Gault Clay outcrop, west of Lulworth Cove.

**Figure 2**: Some Photos of the Outcrops.

**Table 1**: Table Showing the Samples Details.

| Outcrop / Formation | Location |
|---|---|
| Blue Lias (1) | Lyme Regis |
| Kimmeridge Clay (2) | Kimmeridge Bay |
| Kimmeridge Clay (3) | Clavell's Hard |
| Gault Clay (4) | Lulworth Cove beach |
| Nothe Clay (5) | Osmington Mills |
| Oxford Clay (6) | Chickerell, Weymouth |
| Oxford Clay (7) | East Fleet Beach, Weymouth |
| Purbeck Black Shale (8) | Durdle Door beach |

## Rock-Eval Pyrolysis Analysis

The Rock-Eval pyrolysis analysis was carried out on the pulverized rock samples at the GH Geochemical Laboratory, University of Liverpool, following standard pyrolysis experimentation procedure (Espitalié *et al.*, 1977; Peters, 1986) on Rock-Eval II machine. Pyrolysis of c. 100 – 400 mg samples at 300°C for 3mins was followed by programmed pyrolysis at a temperature rise of 25°C/min under an inert atmosphere.

## Gas Chromatography – Mass Spectrometry (GC-MS) Analysis

A portion of pulverized rock sample was subjected to dichloromethane - methanol (93:7

v/v) ultrasonic extraction at Organic Geochemistry and Petrophysics Laboratory, Imperial College London. The extractable organic matter (EOM) obtained for each sample was fractionated using column chromatographic separation technique via elution with 3 ml each of hexane, dichloromethane and methanol in this order, to separate aliphatic, aromatic and polar fractions, respectively.

After addition of the prepared internal standards consisting of 46 µg of adamantane and 40 µg of squalane in hexane for saturate fraction, and 38.8 µg of hexamethylbenzene and 39.2 µg of p-terphenyl in hexane for aromatic fraction, the gas chromatography-mass spectrometry analyses of these hydrocarbon fractions were carried out on the Agilent 6890N gas chromatograph linked with a 5973 Mass Selective Detector (MSD).

The samples were analyzed in full scan mode and later in Selected Ion Mode (SIM) for the same length of time. The sample (1 µl of solution) was injected into an Agilent HP-5MS column on an Agilent 6890N gas chromatograph, before passing on to the 5973 MSD using helium as a carrier gas. The separation was performed on a fused silica capillary column. The GC oven was initially held at 50 °C for 1 minute, and then warmed at 4 °C min$^{-1}$ to 310 °C, where it was held for 20 minutes, for a total run length of 86 minutes.

Because the signals in the aromatic fraction were rather weak, the attention was concentrated on the saturated hydrocarbon fraction which was run in SIM mode with increasing signal-to-noise ratio of 4 to target m/z ratios relevant to hopanes and steranes. Data acquisition and interpretations, and System Quality Control (QC) were achieved with a GC-MS software, Agilent MSD Productivity ChemStation for GC and GC/MS Systems Data Analysis Application. The individual biomarker compounds were determined using GC-MS in the selected ion monitoring mode (SIM) and using the appropriate mass chromatograms (e.g. *m/z* 57 for *n*-alkanes, *m/z* 191 for hopanes, *m/z* 217 for steranes) obtained by the GC-MS.

## RESULTS

The results of the Rock-Eval analysis for the studied outcrop samples are presented in Table 2. The thermal maturity index, Tmax measured from the pyrolysis ranges from 398 to 428°C. The Production Index (PI), the transformation ratio of Tissot and Welte (1984), varies from 0.01 to 0.20, and the bitumen to organic matter (EOM/TOC) ratio ranges from 0.004 to 0.047.

**Table 2:** Rock-Eval Analysis of the Representative Samples from Dorset, Southern England.

| **Outcrop / Formation** | ***TOC (%)** | **$S_1$** | **$S_2$** | **$S_3$** | **$T_{max}$ (°C)** | **HI** | **OI** | **PI** | ***EOM/ TOC** |
|---|---|---|---|---|---|---|---|---|---|
| Blue Lias | 8.14 | 0.79 | 46.33 | 2.07 | 417 | 569 | 25 | 0.02 | 0.004 |
| KCF | 3.22 | 0.14 | 10.75 | 0.55 | 428 | 334 | 17 | 0.01 | 0.031 |
| KCF | 10.98 | 1.47 | 65.82 | 1.33 | 419 | 599 | 12 | 0.02 | 0.041 |
| Gault Clay | 0.59 | 0.10 | 0.39 | 0.34 | 398 | 66 | 58 | 0.20 | 0.047 |
| Nothe Clay | 1.72 | 0.26 | 3.18 | 0.96 | 428 | 185 | 56 | 0.08 | 0.013 |
| Oxford Clay | 2.7 | 0.15 | 6.18 | 1.16 | 423 | 229 | 43 | 0.02 | 0.019 |
| Oxford Clay | 8.11 | 0.55 | 44.61 | 1.65 | 418 | 550 | 20 | 0.01 | 0.013 |
| PBS | 5.36 | 4.34 | 67.08 | 1.33 | 427 | 1251 | 25 | 0.06 | 0.036 |

$S_1$, and $S_2$ are in mg hydrocarbon / g rock, $S_3$ is in mg $CO_2$; HI, Hydrogen Index (HI = $S_2$ / TOC *100); OI, Oxygen Index (OI = $S_3$ / TOC *100); TOC, Total organic carbon (wt %), PI, Production Index (PI = $S_1$ /$S_1$+ $S_2$); KCF, Kimmeridge Clay Formation; PBS, Purbeck Black Shale; *EOM/TOC, EOM/TOC Data from Akande (2011)

**Table 3:** Biomarker-Based Parameters for Organic Matter Thermal Maturity Evaluation.

| Outcrop / Formation | Ts/Tm | Ts/ (Ts+Tm) | Hopane, $C_{31}$ 22S/(22S+22R) | Sterane, $C_{29}$ 20S/(20S+20R) |
|---|---|---|---|---|
| The Blue Lias | 0.13 | 0.11 | 0.29 | 0.24 |
| Kimmeridge Clay Fm | 0.31 | 0.24 | 0.20 | 0.23 |
| Kimmeridge Clay Fm | 1.05 | 0.51 | 0.29 | 0.29 |
| The Gault Clay | 1.23 | 0.55 | 0.50 | 1.43 |
| The Nothe Clay | 0.40 | 0.28 | 0.40 | 0.15 |
| The Oxford Clay | 0.40 | 0.29 | 0.33 | 0.18 |
| The Oxford Clay | 0.39 | 0.28 | 0.37 | 0.28 |
| Purbeck Black Shale | 1.65 | 0.62 | 0.53 | 0.64 |

The biomarkers of interest to this study are the *n*-alkanes, isoprenoids, hopanes, and steranes. The results of the GC-MS analyses show that the *n*-alkanes have unimodal distribution in the outcrop samples and they maximise at carbon–16 or carbon–18. The pristane/phytane (Pr/Ph) ratios range from 0.16 to 1.69. The thermal maturity indices obtained from hopane and sterane isomerization ratios are presented in Table 3.

## DISCUSSION OF RESULTS

The unimodal distribution of *n*-alkanes in the outcrop samples with a major mode at carbon – 16 or carbon – 18 indicates a predominant marine source with the major contribution probably from algae for the analyzed samples. The pristane/phytane (Pr/Ph) ratios (0.16 - 1.69) indicate anoxic to suboxic conditions of deposition (Didyk *et al*., 1978). This range of pristine/phytane ratios, typically less than 2.0, also lends credence to the marine origin of the organic matter in these rocks (Peters and Moldowan, 1993).

### Thermal Maturity Evaluation

The Rock-Eval $T_{max}$ values for the studied rock samples, typically less than 435°C, indicate that they are thermally immature and yet to enter the oil window. The ratio of extractable organic matter (extracted bitumen) to the total organic carbon content (Bitumen/TOC ratio) and Production Index (PI) are also parameters for evaluating source rocks thermal maturity (Peters and Moldowan, 1993). According to them, the Bitumen/TOC values of 0.05 - 0.10 indicate the beginning of the oil window, 0.15 – 0.25 range corresponds to the peak oil window and the values less than 0.05 indicate immaturity. Similarly, the PI values of 0.10, 0.25 and 0.40 approximately correspond to the beginning of oil window, peak oil window and end oil window, respectively.

On the basis of the above criteria, the PI value and EOM/TOC ratio for the Gault Clay sample which are 0.20 and 0.047, respectively, may indicate thermal maturity for this particular sample. However, these parameters seem to be unreliable in this case as it is obvious that this sample (aged: Lower Cretaceous – the Albian) has the lowest Tmax value of 398°C compared with other older samples (aged: Lower Jurassic to Upper Jurassic) with relatively elevated Tmax values and much deeper stratigraphic positions.

The biomarker-based thermal maturity parameter, hopane $C_{31}$ - 22S/22S+22R ratio indicates that the Gault Clay (0.50) and the Purbeck Black Shale (0.53) samples are at least marginally mature. Although the isomerization reactions of steranes under geological conditions are very complex (Seifert and Moldowan, 1978), the isomerization of $C_{29}$ (compared to $C_{27}$ and $C_{28}$

steranes) appears to be most useful in assessing the thermal maturity of both sedimentary rocks and crude oils (Mackenzie *et al.,* 1980; Seifert and Moldowan, 1980) since it extends well into the zone of hydrocarbon generation (Mackenzie and Maxwell, 1981). Thus, sterane $C_{29}$ – 20S/20S+20R ratios were computed for the samples under investigation (Table 3). The sterane $C_{29}$ – 20S/20S+20R ratios of 1.43 and 0.64 for the Gault Clay and the Purbeck Black Shale samples respectively also point to relatively high thermal maturity for these samples.

The trisnorhopanes, Ts/Ts+Tm ratios of 0.55 and 0.62 for the Gault Clay and the Purbeck Black Shale samples respectively, also lend support to the above interpretations. However, the sterane $C_{29}$ – 20S/20S+20R ratio of 1.43 for the Gault Clay appears to be anomalous and is suspected to not indicate the true thermal maturity status in this case.

Although there was no evidence of hydrocarbon staining in the sample during outcrop sampling, the shallow stratigraphic position of this rock compared to other rocks under investigation, as noted above, suggests that the sample probably contains huge chunks of reworked organic matter eroded from deeper, older, and more mature rocks, probably from the underlying Liassic shales or the Kimmeridge Clay beds in the basin. Alternatively, the spurious sterane isomerisation ratio for the Gault Clay may be attributed to analytical errors or low concentrations of steranes (e.g. $C_{28}$ – $C_{30}$ steranes) in the sample (Figure 3).

Thermal maturity parameters obtained from the biomarker analyses can be correlated with other indices of thermal maturity such vitrinite reflectance and pyrolysis Tmax (Waples and Machihara, 1991). The results of biomarker studies in this work are correlated with the pyrolysis Tmax and PI, and the plots produced are as shown in Figures 4 and 5.

An assessment of the organic maturation of potential source rocks and oils based on steranes and pentacyclic triterpanes of the hopanes as maturity indicators has the dynamic range of most of their ratios restricted to rather low maturation levels (Mackenzie and Maxwell, 1981) as there is a drastic decrease in the concentration of the biomarker molecules with increasing thermal maturity (Rullkŏtter *et al.,* 1984).

Thus, the results of this study show that there is a negative (slope) correlation between the biomarker parameter, hopane $C_{31}$ – 22S/22S + 22R ratio and the pyrolysis Tmax (Figure 4).

The plot shows that the Gault Clay sample underwent the least thermal stress among the samples under investigation, and hence it cannot be considered to be mature. The plot also shows that the data points correlate fairly well at low temperatures but become much scattered at elevated temperatures. Figure 5, however, shows a positive correlation between the biomarker parameter, hopane $C_{31}$ – 22S/22S + 22R ratio and the PI. The observed trend appears to be affected by facies as the shaley formations define the trend and the more clayey formations distribute uniformly about the trend line. Some evidences from gas chromatogram traces support that the Purbeck Black Shale is thermally mature.

The gas chromatogram trace (m/z 191) displayed in Figure 6, for example, shows a stair-step progression of hopane ($C_{30}$ – $C_{34}$) doublets distributions with 22S – stereoisomer configuration being more stable and in more abundance than the corresponding 22R– configuration which is a maturity signature. Another evidence to justify the thermal maturity of the Purbeck Black Shale is the quadruplet maturity signature at C-28 (Figure 7).

## CONCLUSION

The results of this study show that the Tmax values obtained for all the analyzed samples are less than the threshold Tmax value of 435°C quoted for the beginning of oil window in the literature. However, these results are not taken as gospels because the pyrolysis Tmax parameter is partly dependent on the type of organic matter (Peters and Moldowan, 1993) and can vary from one sedimentary basin to another. Thus, it is concluded on the basis of multiple lines of evidence from the biomarker studies that the Purbeck Black Shale is thermally mature and is already in the oil window. On the other hand, the thermal maturity shown by the Gault Clay sample is interpreted to be a result of allochthonous bitumen, probably from the underlying more mature, older Liassic shales or the Kimmeridge Clay beds in the basin.

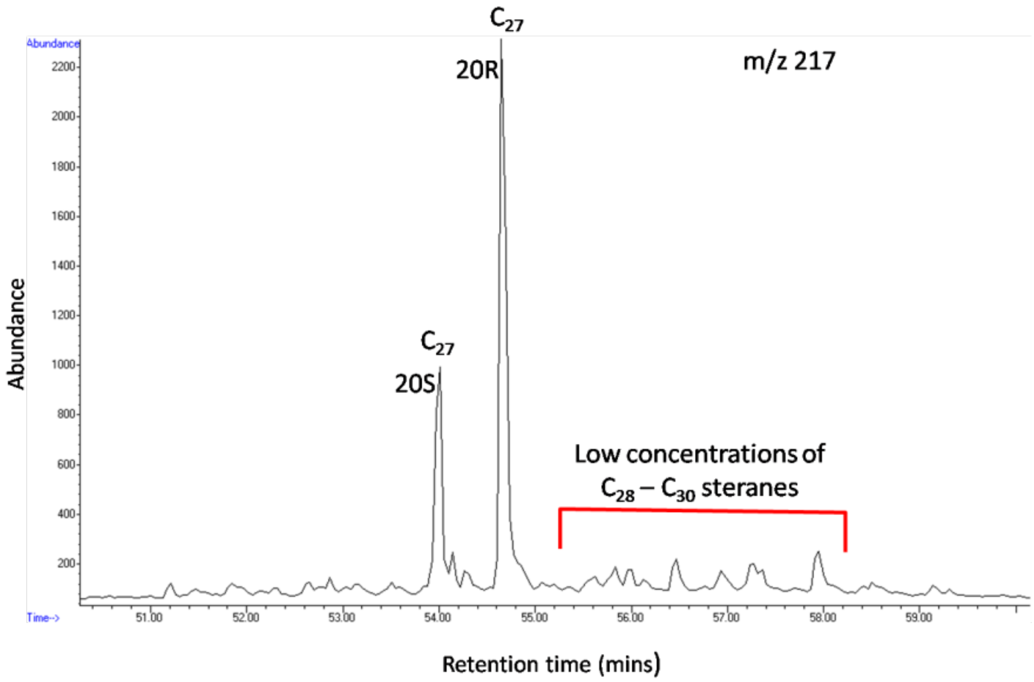


**Figure 3:** Gas Chromatogram (*m/z* 271) Trace Showing Steranes Distributions in the Gault Clay Sample.

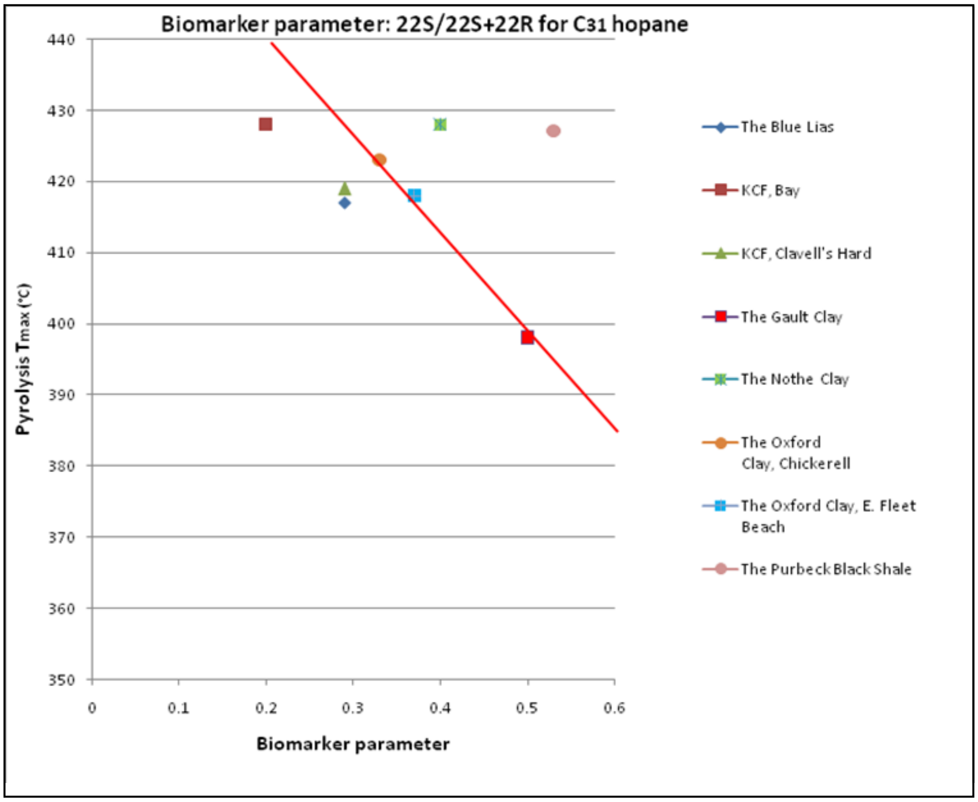


**Figure 4:** Correlation between Biomarker Parameter and Pyrolysis Tmax.

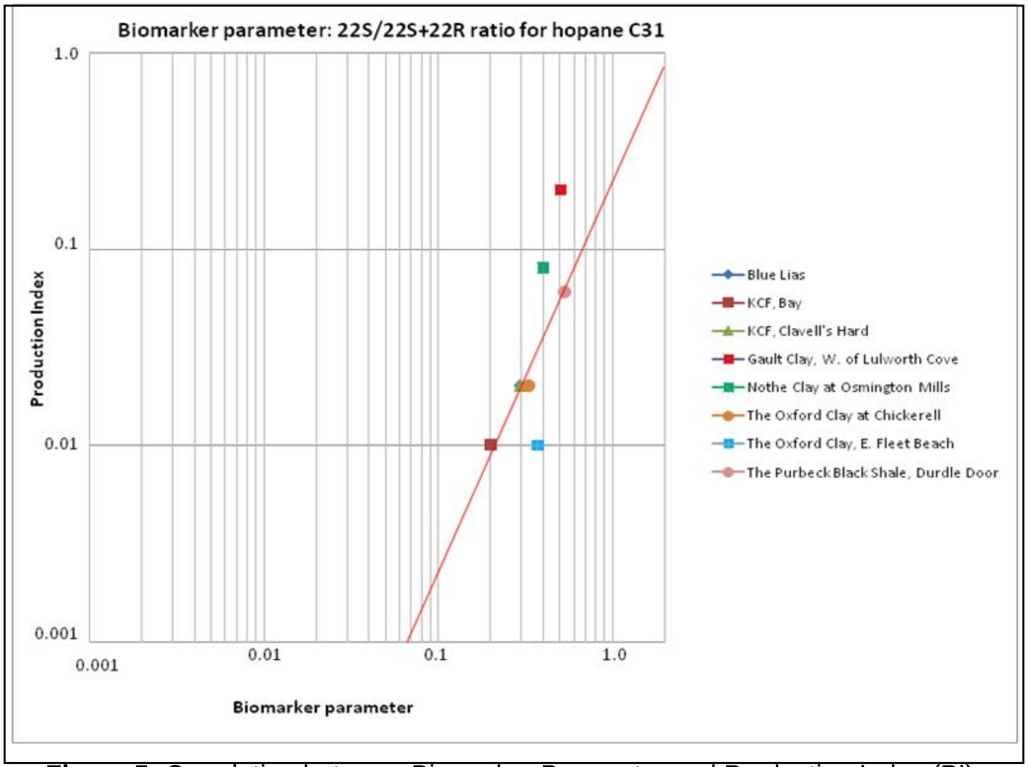


**Figure 5:** Correlation between Biomarker Parameter and Production Index (PI).

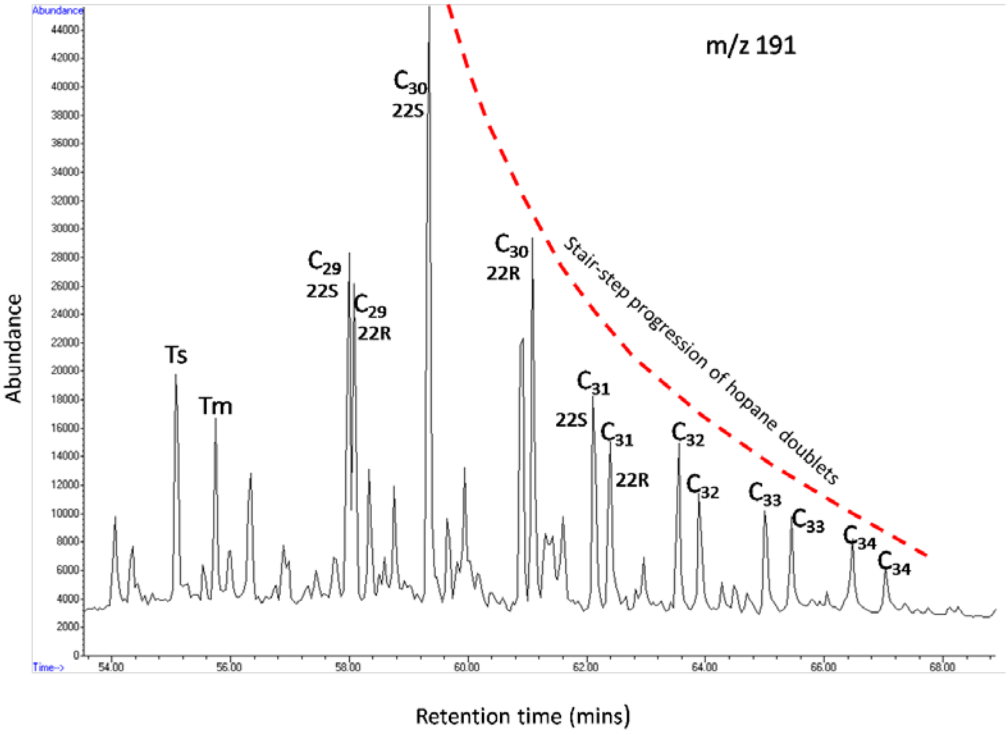


**Figure 6:** Gas Chromatogram (*m/z* 191) Trace Showing Hopanes Distributions in the Purbeck Black Shale Sample.

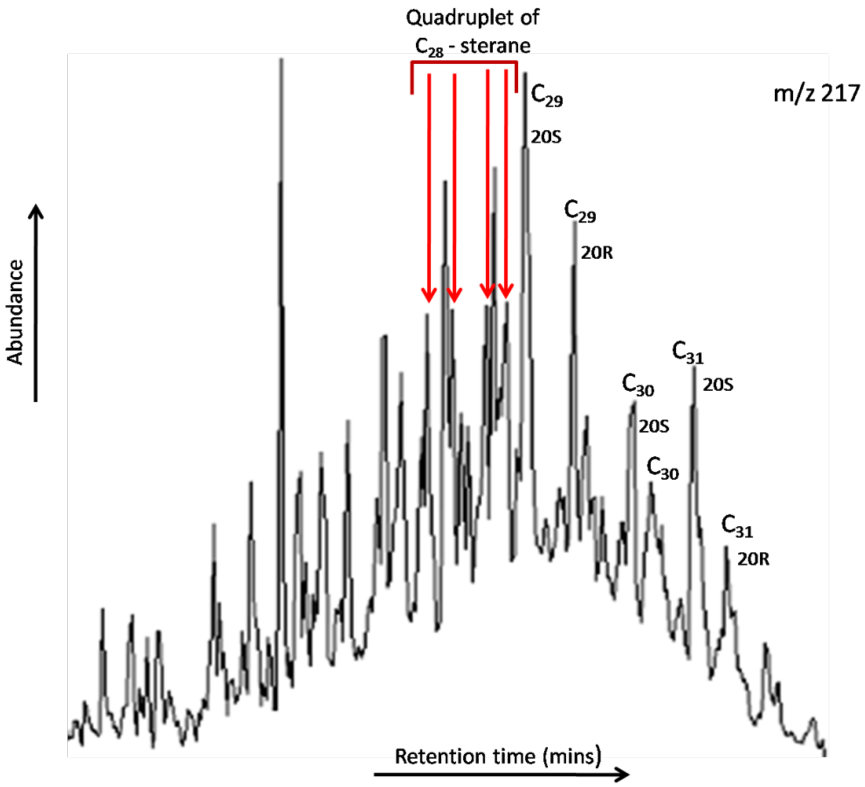


**Figure 7:** Gas Chromatogram (*m/z* 217) Trace Showing Quadruplet Signature in Sterane Distributions in the Purbeck Black Shale Sample.

The correlation of the biomarker parameter with the pyrolysis Tmax also confirmed that the Gault Clay sample has only experienced low thermal stress and therefore cannot be considered thermally mature. Other formations considered in this work such as the Kimmeridge Clay, the Blue Lias, the Oxford Clay, and the Nothe Clay Formations are thermally immature in the study area.

The present study reveals that the Purbeck Black Shale is thermally mature. Hence, it is recommended that besides the Kimmeridge Clay, the Blue Lias and the Oxford Clay Formations which are considered mature elsewhere in the basin, the Purbeck Black Shale should also be considered for hydrocarbon exploration in the study area.

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