Black shale deposition in an Upper Ordovician–Silurian permanently stratified, peri-glacial basin, southern Jordan

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1. Introduction

The deposition of marine black shale and the enhanced storage of organic carbon (OC) in the geological record indicate fundamental changes in the functioning of biogeochemical cycles and their feedbacks during extreme climate modes and transitions (Beckmann et al., 2005a; Page et al., 2007). Our understanding of the response of the marine environment during these climate states can only be gained from a study of deep time analogues.

The Upper Ordovician–Lower Silurian succession of North Africa and Arabia contains thick (~20 m), organic carbon (OC)-rich (up to 15% total organic carbon (TOC)) black shale, widely known as the “hot shales,” which are the source of ~30% of the world's oil (Lünig et al., 2000, 2006). The origin of these deposits remains contentious (Armstrong et al., 2005, 2006). The lower hot shale overlies glacial and glacio-marine sediments deposited during the Hirnantian glaciation (~445 Ma) and have been linked to either, nutrient enrichment of shallow marine environments during coastal upwelling (Lünig et al., 2000) or, freshening by deglacial meltwater (Armstrong et al., 2005).

Here we report data from Jordan that confirms the lower hot shale was deposited in a stratified, ice margin basin during Hirnantian to early Silurian deglaciation (Armstrong et al., 2005). We relate deglacial sea level rise (at Milankovitch timescales) and melt water flux to evidence for productivity changes, anoxia/euxinia and the increased burial of organic matter. Bulk δ13C and total organic carbon (%TOC) are taken to reflect productivity changes. The presence of isorenieratane (XXIII; see Appendix A for structure), a biomarker of green sulphur bacteria, is indicative of photic zone euxinia. Likely modern analogues are the seasonally isolated basins of east Antarctica. A similar interdisciplinary approach is necessary to elucidate the nature of these deposits elsewhere on the Gondwana margin.

2. Stratigraphical and geological context

The Lower Palaeozoic succession in southern Jordan includes some 750–800 m of well exposed Ordovician siliciclastic sediments deposited on the margins of the North African (Gondwana) in terrestrial to subtidal marginal marine and shelf environments (Amireh et al., 2001; Makhlouf, 1995; Powell et al., 1994; Fig. 1). During the Late Ordovician Jordan was located in a high latitude, east Gondwana setting, 60° S of the equator (Cocks and Torsvik, 2002), less than 100 km from the margins of a terrestrial ice sheet in northwest Saudi Arabia. This ice sheet was characterised by two major phases of ice advance and retreat (Vaslet, 1990) both marked by erosional unconformities (Vaslet, 1990; Figs. 1 and 2). The first major glacial ice incised into permafrost-hardened and glacially loaded, Tubeiylat shoreface and nearshore shelf deposits, preferentially excavating NW–SE trending major fault-controlled depressions, cutting a steep-sided U-shaped valley (Turner et al., 2005). This ice advance correlates with the first glacial advance in northwest Saudi Arabia (Vaslet, 1990; Miller and Mansour, 2007; Fig. 3), and was followed by deglaciation,
Fig. 1. Lithostratigraphy and chronostratigraphy for the Ordovician and Silurian of Jordan and Saudi Arabia, showing generalised depositional environments for outcrops in the Southern Desert region of Jordan (redrawn from Turner et al., 2005). Subdivision of the Ammar Formation into the Lower and Upper Ammar is based on Abed et al. (1993).

Fig. 2. Generalised section of the glacial and deglacial succession in the Southern Desert region of Jordan and northwest Saudi Arabia showing the stratigraphy and sediment fill of the glacially incised palaeovalley systems. Section A is located 0.5 km southwest of Jebel Umier (29° 34′ N, 35° 53′ E) and Section B is from Jebel Ammar (29° 34′ N, 35° 52′ E). Section C is from northwest Saudi Arabia and is based on Vaslet (1990); reproduced with permission from Turner et al. (2005).
a rise in relative sea level and transgressive filling of the palaeovalley. The latter is recorded by a thin, reworked bottom lag of glaciofluvial sandstones, overlain by thick, transgressive, shoreface sandstones. Late transgressive filling of the palaeovalley was interrupted by a second and possibly a third subsidiary glacial advance producing a glacially polished and grooved surface with intersecting glacial striations, indicating ice flow from the west and northwest (Turner et al., 2005).

The fourth glacial advance produced a regionally extensive lowstand tunnel valley beneath the ice sheet (Turner et al., 2002). This was subsequently preserved as a palaeovalley incised into the lower palaeovalley-fill deposits or, where this is missing, into the top of the Tubiliyat Formation. This ice advance correlates with the second major ice advance in Saudi Arabia (Vaslet, 1990), where the Sarah Tubeiliyat Formation similarly records a complex record of ice advance and subsequently preserved as a palaeovalley incised into the lower and upper subzones of the ascensus-acuminatus biozone (sensu Storch, 1990) and indicate an earliest Silurian age. Graptolites collected at 42.82 m (4.02 m above base of formation) and 41.57 m (5.27 m above the base of the formation) contain Normalograptus parvulus. N. parvulus ranges through the persculptus to acuminatus biozones of Hirnantian to Rhuddanian (Llandovery) age (Zalasiewicz and Tunnicliffe, 1994, Fig. 3). These age assignments allow the correlation of the transgressive fill of the upper palaeovalley in Jordan with the Hirnantian to early Llandovery (Rhuddanian) global eustatic sea level rise (Cocks and Rickards, 1988; Loydell, 1998).

Armstrong et al. (2005) concluded the base of the black shale is coincident with the maximum flooding of the first post-glacial highstand (cf. Lüning et al., 2000). The fill of the palaeovalley was considered as an “expanding puddle” as originally defined by Wignall (1991).

3. Materials and methods

Here we use carbon isotopic, biomarker and Rock-Eval analyses on black shale from the lower Batra Formation (Jordan) to establish water column redox conditions. Core samples were obtained from the immature (average $T_{\text{max}}$ value of 419 °C), OC-rich lower 18 m section of the Batra Formation in the type area of Wadi Batti el Ghul (well BG14; Table 1) from the Southern Desert region of Jordan (29°30′50.4″ N 35°57′41″ E; Armstrong et al., 2005).

3.1. Total organic carbon (TOC) and Rock-Eval pyrolysis

The TOC contents of the dried samples were measured using a calibrated LECO CS-244 elemental analyser. Each sample was analysed in duplicate and standard material was analysed after every 10 analytical samples to ensure that the analyser maintained its calibration. Rock-Eval pyrolysis was carried out using a Delsi Oil Show Analyser. Each sample was pyrolysed in duplicate so that the mean values of the amounts of hydrocarbons generated under the S1 and S2 peaks as well as the temperature $T_{\text{max}}$. Corresponding to the temperature at which the maximum of the S2 hydrocarbon generation occurs during pyrolysis, could be measured. Standard (5.51% TOC; 0.27 mg HC/g of rock S1; 13.59 mg HC/g of rock S2; and 430 °C $T_{\text{max}}$) and then blank samples were pyrolysed under these same conditions to ensure that the measurements of the unknown quantities were as precise as possible. The average standard deviations with respect to S1, S2 and $T_{\text{max}}$ were 0.05 mg HC/g of rock, 0.43 mg HC/g of rock and 2.3 °C respectively.

3.2. Bulk stable carbon isotope analysis

$^{13}$C/$^{12}$C ratios ($\delta^{13}$C) were measured on bulk sediments after removal of the inorganic carbonates with dilute HCl using automated online combustion followed by conventional isotope ratio-mass spectrometry in a VG TripleTrap and Optima dual-inlet mass spectrometer, with $\delta^{13}$C values calculated to the Vienna PeeDee belemnite (VPDB) scale using a within-run laboratory standard (cellulose, Sigma Chemical prod. no. C-6413) calibrated against NBS-19 and NBS-22. Replicate analysis of well-mixed samples indicated a precision of ±0.1‰ (1 S.D.).

3.3. Gas chromatography (GC)/gas chromatography–mass spectrometry (GC-MS)

The powdered black shales were Soxhlet extracted with dichloromethane/methanol (93:7 v/v) for 48 h. An aliquot of each total extract was separated by thin layer chromatography (TLC, Kieselgel 60G, 0.5 mm thickness) using light petroleum ether (boiling point from 40 to 60 °C) into aliphatic hydrocarbon, aromatic hydrocarbon and polar fractions. The aliphatic and aromatic hydrocarbon fractions were analysed using a Hewlett-Packard HP5890 gas chromatograph (GC) equipped with a flame ionisation detector and a fused silica capillary column.
(30 m×0.25 mm i.d.) coated with either HP-1 or HP-5 stationary phase (film thickness of 0.25 µm). The carrier gas was hydrogen, and the oven temperature was held at 50 °C for 2 min and then heated at a rate of 4 °C/min to 300 °C at which it was held for 20 min. The following oven temperature programme was used for the analysis of the aromatic hydrocarbons (primarily for the assignment of isorenieratane XXIII): the oven was held at 60 °C for 2 min and then heated at 4 °C/min to 300 °C, at which it was held for 20 min (Fig. 4).

GC-MS was performed using a Hewlett-Packard HP5890II GC coupled with a Hewlett-Packard 5972 mass spectrometer (ionising voltage of 70 eV and with the source temperature at 160 °C). The GC was fitted with a fused silica capillary column (30 m×0.25 mm i.d.) coated with either HP-1 or HP-5 stationary phase (film thickness of 0.25 µm). The carrier gas was helium, and for analysis of the aliphatic hydrocarbons (see Fig. 4) the oven temperature was held at 40 °C for 2 min and then heated at a rate of 4 °C/min to 300 °C at which it was held for 20 min. The following oven temperature programme was used for the analysis of the aromatic hydrocarbons (primarily for the assignment of isorenieratane XXIII): the oven was held at 60 °C for 2 min and then heated to 240 °C at 10 °C/min, further heated to 315 °C at 4 °C/min where it was held at the final temperature for 50 min.

Steranes and hopanes were identified using published mass spectra and relative retention times (e.g. Peters et al., 2005). Isorenieratane (XXIII) was identified by its mass spectrum and by GC-MS co-injection experiments on an HP-1 stationary phase (authentic standard of isorenieratane (XXIII) was supplied courtesy of S. Schouten, Netherlands Institute for Sea Research, Den Burg, Netherlands). The GC-MS results from the co-injection experiments are presented in Fig. 5.

4. Results

The presence of parallel laminations accompanied by an absence of bioturbation throughout the section indicates euxinic bottom waters during deposition. All samples typically contain acritarchs and graptolites (Keegan et al., 1990) indicating a primarily marine phytoplanktonic and zooplanktonic source of Type II kerogen. The percentage of total organic carbon (%TOC) increases as a function of height above the base of the Batra Formation (Fig. 6A). This section is OC-rich with a stepwise increase in %TOC from ~1% to ~3% and from ~3% to ~9% at 6.27 m and 12.94 m respectively above the base of the section (Armstrong et al., 2005). Figs. 6B and 7 show that the Rock-Eval hydrogen index (HI) values of the samples have a range of 156 to 402 mg HC/g TOC (mean value = 283 mg HC/g TOC). The δ13C of bulk organic matter show a range of ~30.8 to ~29.6‰, with fluctuations of up to 0.4‰ and a positive shift of 1.4‰ up the section with the largest increase (~1‰) apparent in the two uppermost samples (Fig. 6D).

The regular sterane carbon number distributions are such that C29>C27>C25>C23, where the average value of the C29/C27 steranes ratio is 0.27, which agrees with previous observations that generally this particular ratio is less than about 0.35 for samples older than Silurian (Grantham and Wakefield, 1988). Both the sterane and 17α-hopane distributions indicate thermally immature organic matter and do not vary significantly throughout the section. Maxima in the regular steranes/17α-hopanes (Frimmel et al., 2004) occur at ~6.5 m and 12.94 m above the base of the formation and coincide with the stepped increases in %TOC suggesting major contributions to the organic matter from plankton (Fig. 6C).

The mass chromatogram for m/z 133 from the aromatic hydrocarbon fraction (Fig. 7) reveals a pseudo-homologous series of aryl isoprenoids up to C26 (see I through to X in Fig. 7 and Table 2) as well as the presence of aryl isoprenoids with additional aromatic rings (see XI and XVI in Fig. 7 and Table 2). The relative amounts of the different components remains the same throughout the profile where the C17, C20, C21, C22

Table 1
Late Ordovician black shales from a Batra Formation (southern Jordan) borehole (BG14) giving sample # and position in the stratigraphical column

<table>
<thead>
<tr>
<th>Sample</th>
<th>Height above base of Batra Formation (m)</th>
<th>Depth in core (m)</th>
</tr>
</thead>
<tbody>
<tr>
<td>8402-77</td>
<td>16.76</td>
<td>30.08</td>
</tr>
<tr>
<td>8402-19</td>
<td>15.28</td>
<td>31.56</td>
</tr>
<tr>
<td>8402-18</td>
<td>15.14</td>
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<td>8402-17</td>
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<td>32.00</td>
</tr>
<tr>
<td>8402-16</td>
<td>14.34</td>
<td>32.50</td>
</tr>
<tr>
<td>8402-15</td>
<td>12.94</td>
<td>33.90</td>
</tr>
<tr>
<td>8402-14</td>
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</tr>
<tr>
<td>8402-52</td>
<td>3.77</td>
<td>43.07</td>
</tr>
</tbody>
</table>

Fig. 4. Total ion chromatogram (TIC) of aliphatic hydrocarbon fraction from Upper Ordovician black shale from 16.76 m (sample 8402-77) above the base of the formation in BG-14 borehole. Pr = pristane, Ph = phytane; numbers denote total number of carbon atoms in the n-alkanes.
members have reduced abundances. This distribution is similar to that observed in Upper Devonian sediments (Hartgers et al., 1994) but differs from that in much older mid-Proterozoic bitumens (Brocks et al., 2005). There are also less abundant C32, C33 and C40 diaryl isoprenoids both with and without an additional aromatic ring (see XVII through to XXIII in Fig. 7 and Table 2). Isorenieratane (XXIII) was present throughout the core and its identity was also confirmed by co-elution with an authentic standard on a range of stationary phases (Sinninghe Damsté et al., 2001).

5. Discussion

The deposition of sedimentary organic carbon in the lower Batra Formation is delimited by stepped increases (approximate doubling at

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**Fig. 5.** GC-MS ion chromatogram of m/z 133 of aromatic hydrocarbon fraction from Upper Ordovician black shale (sample 3402-15) taken at 33.9 m in the core, 12.94 m above the base of the formation. (A) after and (B) before co-injection with an authentic standard of isorenieratane (XXIII; courtesy of S. Schouten, Netherlands Institute for Sea Research (NIOZ), Den Burg, Netherlands) showing enhancement of peak after co-injection. (C) is mass spectrum of isorenieratane (XXIII) from the sample.
each step) in %TOC up succession. %TOC has been found to correlate well with more direct measures of photosynthetic primary productivity such as total chlorophyll-a or total steryl chlorine esters (Nara et al., 2005) and organic mass accumulation rate (Tyson, 1995; Vilinski and Domack, 1998; Twichell et al., 2002; Meyers and Arnaboldi, 2005). A longer-term trend towards more fractionated values up succession reflects a progressive increase in the sedimentation of $^{12}$C-enriched organic matter.

Hydrogen index (HI) is a measure of hydrogen-richness and depends on the nature of the original organic matter and the degree of preservation during diagenesis (Peters, 1986; Bordenave et al., 1993). Millimetre-scale laminations and presence of macroscopic pyrite through the section suggests euxinia and that organic matter preservation potential was high throughout deposition. Our bulk rock HI values are similar to those reported from Mediterranean, Pliocene–Pleistocene (< 3 myr old) immature sapropels which have HI values of ~400 mg HC/g TOC (Tyson, 1995). Bulk HI and organic matter $\delta^{13}$C values do not covary (Fig. 6B and D); and this with the unchanging nature of kerogen type through the section, suggests variations in HI reflect the changing extent of organic matter preservation (see Tyson, 1995).

Peaks in steranes/17α-hopanes ratio (>1) coincide with the stepped changes in %TOC, that provide a coherent signal of primary productivity. We therefore conclude the changes in %TOC reflect changes in plankton primary productivity in the photic zone. The decay of this organic matter through oxidative respiration resulted in anoxia and euxinia, which enhanced the potential for organic matter preservation.

Our bulk $\delta^{13}$C values fall within the range for modern phytoplanktonic algae (Schidlowski, 1988) and for bulk organic matter in the Southern Ocean at 0 °C (Rau et al., 1989; Bentaleb and Fontugne, 1998; Bentaleb et al., 1998; Lourey et al., 2004). Increasingly more fractionated bulk organic matter $\delta^{13}$C values occur up section with no change in kerogen type. In ice margin basins at the present day changes in $\delta^{13}$C are usually associated with a decreased CO$_2$ availability in response to (a) decreased supply by diffusive limitation, or (b) increased demand because of higher

![Fig. 6. Composite plot of bulk organic carbon, biomarker and bulk stable carbon isotopic data.](image)

![Fig. 7. Typical GC-MS summed mass chromatogram of m/z 133 + 134 from the aromatic hydrocarbon fraction isolated from the shale organic extract at 11.64 m above the base of the Batra Formation. This trace shows the C40 biomarker isorenieratane (XXIII). Roman numerals refer to compounds indicated in Appendix A and Table 2.)](image)
occur naturally in either intense phytoplankton blooms (Dunbar and Leventer, 1992) or within sea ice (Dunbar and Leventer, 1992; McMinn et al., 1999). Natural values for modern Southern Ocean phytoplankton can be as low as ∼25‰ (Rau et al., 1991). While highly fractionated values as high as ≥12‰ have been recorded from sea ice (Dunbar and Leventer, 1992; McMinn et al., 1999). Thus when sea ice melts it not only delivers a pulse of sediment and nutrients, but also organic matter that has a more fractionated δ13C signature (Gibson, 1999).

A pattern of long-term covariance of more fractionated δ13C and increasing δTOC has also been reported in Cretaceous (Late Cenomanian/Turonian) black shale in the North Atlantic, deposited during periods of enhanced continental run-off (Beckmann et al., 2005a,b). Here the pattern has been interpreted to indicate organic carbon sequestration to the sediment was cumulative through the depositional event (Kuypers et al., 2002).

We therefore consider the more fractionated δ13C values through the lower Batra section to represent CO2 limitation and increased productivity as nutrients and isotopically light carbon were supplied by melt water. The long-term trend towards more fractionated values reflects a progressive increase in the sedimentation of 12C-enriched organic matter during progressive deglaciation.

The presence of isorenieratene derivatives in black shales has been widely considered diagnostic of green sulphur reducing bacteria (Chlororhizaceae and Chromatiaceae) and is used as evidence for photic zone euxinia (e.g. Sinninghe Damsté and Köster, 1998). Further, pyrite is common through the section. In the modern ocean (Killops and Killops, 1993) the depth of the sulphate reduction zone depends on the amount of organic matter influx from the euphotic zone and may be relatively shallow (∼20 m) in highly productive areas, where sulphate is rapidly depleted (Brocks et al., 2005). The depth to the chemocline in the Batra Basin may have been shallower than the ∼50 m found in the Black Sea at the present day (Murray et al., 1989).

The greatest concentration of stratified water bodies in high latitudes, and possibly the world, is found in the Vestfold Hills of Antarctica. Here meromictic lakes, isolated marine basins and fjords occur (Burton, 1981; Burke and Burton, 1988a; Gallagher et al., 1989) and seasonal anoxia is developed in these settings. These basins formed following the retreat of the continental ice sheet ∼10 000 years ago, when isostatic rebound occurred at a faster rate than sea level rise, and the land emerged from the sea (Burke and Burton, 1988b). Seasonal stratification is maintained in these basins by an increase in salinity (Gibson, 1999) resulting from brine exclusion during sea ice formation. During the winter, a thermocline convection cell develops directly beneath the ice cover and penetrates progressively deeper into the basin throughout winter. At the end of the period of ice formation the convection cell breaks down and stratification of the surface water occurs. When the ice melts completely, lenses of relatively fresh water cap the basins; this reduces the effect of wind mixing, with a net result of stabilising the basin stratification, with anoxia developing at depth. The effect of, increasing water level in the basins or decreasing maximum ice thickness during the summer results in a shallowing of the mixoline and chemoline (Gibson, 1999). The nature of the stratification in these basins (Gibson, 1999; McMinn et al., 2001) is therefore similar to those reported from the many permanently stratified basins around the world including the Red Sea (Hartmann et al., 1998), the Cariaco Trench, the Black Sea, and fjords of Scandinavia (Skei, 1983; Lindholm, 1996).

We envisage the geological history of the Batra Basin and its included sediments to be directly controlled by ice margin processes. On ice melting and retreat the exposed upper palaeovalley became a conduit for ice meltwater. The basin was initially isolated from shallow marine waters, likely silled and glaciofluvial sediments were deposited. As the effects of isostatic rebound waned and sea level rose, the basin was flushed by marine waters. On short timescales we envisage the basin became stratified due to the formation and melting of sea ice (Fig. 8). The millimetre-scale laminations in the black shales may reflect seasonal to decadal (? millennial) changes in sea ice cover. On the longer term, periods of increased surface primary productivity occurred when prolonged ice free conditions prevailed and/or, fluxes of freshwater and nutrients entered the already stratified, euxinic marine basin. Stratigraphical evidence (Armstrong et al., 2005) is consistent with modelling results (Herrmann et al., 2003) and indicates the frequency of melting events during the late Hirnantian deglaciation was likely to have occurred on an obliquity (~40 kyr) timescale. The black shales of the Batra Basin record a few hundred thousand years of water column stratification and basin euxinia during deglacial hightstand. This represents one of the oldest peri-glacial permanently stratified basins yet described.

6. Conclusions

Sedimentology, geological setting and organic geochemical proxy data indicate the Batra Formation black shale in Jordan was deposited in a permanently stratified, ice margin, marine basin that existed for a few hundred thousand years. Euxinia extended into the photic zone enhancing sedimentary carbon preservation and sedimentation. Ice melting and/or, fluxes of freshwater and nutrients resulted in enhanced photic zone primary productivity.

![Fig. 8. Conceptual model of the open water (ice free), stratified water column during the deposition of the lower “hot shales” in the Batra Basin. This is based in part on that found in modern ice margin basins in the Vestfold Hills, east Antarctica (Gibson, 1999, Fig. 7).](image-url)
and organic matter sedimentation. The seasonally isolated basins and anoxic fjords of east Antarctica provide a likely modern analogue, though these are \(<10000\) years old. Black shale was patchily deposited along the entire northern Gondwana margin during the Silurian. Similarly detailed interdisciplinary studies are required to test whether coastal upwelling can be invoked to explain the origin of any of these deposits.

Acknowledgements

The National Resources Authority of Jordan provided access to the core. We acknowledge support from our host institutions and the Natural Environment Research Council for research funds including JREI awards. Dr David Loydell (University of Portsmouth) and Dr Mark Williams (University of Leicester) kindly identified the graptolites. TOC measurements were conducted by Dr D.M. Jones (University of Newcastle upon Tyne) and isotopic analyses by Prof. M. Leng (NERC Isotope Geosciences Laboratory). We thank Christine Jeans for the preparation of the Figures and B. Bowler for technical input. H.P. acknowledges the European Commission Research Directorates General for a Marie Curie Host Fellowship held at the University of Newcastle upon Tyne. A.B.M. was supported by the Petroleum Technology Development Fund, Nigeria. H.A.A. and B.R.T. acknowledge funding from the Natural Environment Research Council. This is a contribution to IGCP Project 503. We thank Lorenz Schwark and an anonymous referee for their helpful suggestions.

Appendix A. Structures of isorenieratene derivatives