FULL LENGTH ARTICLE

Mechanism of Late Campanian–Early Maastrichtian oil shale deposition and its sequence stratigraphy implications inferred from the palynological and geochemical analysis

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Geochemical analysis;
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Sequence stratigraphy

Abstract Understanding the organic rich sediment deposition mechanism is vital for the purposes of their exploration. This should reconsider the sequence stratigraphic framework and its associated paleoenvironmental setting. The palynological and geochemical aspects of the organic rich beds from the Duwi Formation conducted on six phosphate mines in the Eastern Desert of Egypt are reported in the present study and they were used to investigate the paleoenvironmental settings that existed during its deposition. The palynomorph assemblages were dominated by moderately diverse and abundant dinoflagellates and in El-Nakheil, Wasif, Umm Hueitat and Mohamed Rabah mines and generally scarce palynomorph assemblages were generally detected at El-Beida and Younis mines. The dinoflagellates are mainly peridinioids, namely; Alterbidinium acutulum, Cerodinium obliquipes, Palaeocystodinium australinum and Phelodinium tricuspis, in addition to some gonyaulacoid such as Kleithriasphaeridium readei, Hystrichosphaeridium sp. A, Hystrichosphaeridium sp. B and Spiniferites supparus. These dinoflagellate assemblages are indicating Late Campanian–Early Maastrichtian age. The palynofacies analysis revealed enrichments with amorphous organic matter (AOM) at El-Nakheil and El-Beida mines, while the phytoclasts enrichments were found to be at the Younis mine. The enriched AOM samples are of Type I and II oil prone kerogen while the enriched phytoclasts are of Type III gas prone kerogen. In line with, the resulted kerogen types agreed with rock eval pyrolysis analysis. The integration of rock eval pyrolysis and other geochemical parameters with the palynofacies analysis indicated that the deposition of low organic matter sediments (TOC/C24 = 0.04–1.77 wt%) took place in a low stand system-tract. On the other hand, the sediments of high organic matter content (TOC/C24 = 9.66–22.23 wt%) were deposited in a trans-

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1. Introduction

Duwi Formation is destined to pile up many valuable fortunes for Egypt. Since the 1950s it was the main source of phosphate minerals’ exploitation, which composed the bases for important industries such as agricultural fertilizers. In the more recent years, there is growing evidence about its capacity to store uranium [1,2]. Since 1980s, the organic matter rich sediments (carbonaceous oil shales) accompanied phosphorites have drawn the attention [3,4]. These oil shale beds have been reported to catch fire [5]. The average oil content of weathered black shale samples is 19 gallon/ton [6]. These oil shale beds in Duwi Formation have many relatives in the neighbor countries such as Jordan and occupied Palestine. The oil shale beds in these countries are exploited regularly and are used in many industrial applications such as electricity production and cement industry as direct combustion; they are also used to produce liquid fuels by retorting [7,8]. These oil shale beds are associated with one large environmental setting that is known as Parrish and Curtis [9] upwelling belt. This belt existed in the Late Campanian–Early Maastrichtian in the southeastern Levant Basin. The majority of oil shale workers in such countries adopt the same model mechanism for the deposition of the oil shale beds. Accordingly, organic matter rich sediments of the oil shale beds resulted exclusively from the high surface paleoproductivity accompanied by the upwelling high nutrient levels and oxygen poor bottom water during the Late Campanian–Early Maastrichtian in the southeastern Levant Basin [10–13]. The limitation, that is considered as Achilles’ heel in this Primary-Productivity-Driven model, is the weak concurrence between the oceanic primary productivity intensities and organic-carbon levels in sediment surface layers in modern oceanic upwelling zones (e.g., the Equatorial Pacific, Antarctic Divergences and the northwest African margin coastal upwelling) [14,15]. The outcome of dysoxic to anoxic conditions on the organic matter preservation is only graspable at slow sedimentation rates [16]. There are three factors controlling the total organic carbon content in the sediments, namely; organic matter input (productivity), organic matter preservation (oxygen deficiency) and dilution by mineralic sediment components. These factors are fully discussed in Katz [15] and Tyson [16]. The last factor is directly related to the sequence stratigraphic framework and its associated paleoenvironmental settings. The lower mineral sedimentation rates are associated with the flooding surfaces during the transgression system tracts.

The reconsideration of paleoproductivity as the major factor for the formation of the oil shale beds in the Upper Campanian–Lower Maastrichtian sediments is the central focus of the present study. The present paper aims to evaluate and explore the space relationship on the one hand between oil shale beds in the Duwi Formation in the Al-Qusseir area and on the other hand between these oil shale beds and their equivalents in adjacent countries in the perspective of the palynological and organic geochemical studies. This research study is based on twenty samples collected from some Egyptian localities and the geological knowledge available in the literature from the surrounding countries. The study of dinoflagellate biozonation and statistical distribution facilitate aging of certain dinoflagellate diversities and synchronize their excessive flux events in the different areas. These events were in turn exclusively connected to some intense paleoproductivity fluctuations in Parrish and Curtis [9] upwelling belt and deposition of the oil shale. In the present study, a revision of these concepts has been considered with referring to the recent studies of deposition of organic matter rich sediments in the Duwi Formation in the sequence stratigraphy framework and its associated paleoenvironmental settings.

2. Geological settings of the Duwi Formation

The Precambrian granite and metamorphic rocks (gneiss, schist) compose the basement complex in Egypt. They form a rough terrain at the Eastern Desert of Egypt along the Red Sea coast and Sinai. The Upper Cretaceous to Lower Cenozoic sedimentary rocks cover the basement complex in some areas (Fig. 1A). The Duwi Formation is a part of the Upper Cretaceous–Lower Cenozoic sedimentary sequence and is widely distributed in the Eastern Desert, Nile Valley and Western Desert areas. The Duwi Formation unconformably overlies the fluvial shale sequence of the mid Campanian Qusseir Formation, and conformably underlies the deep marine shales and marls of the mid Maastrichtian Dakhla Formation. Thus, deposition of the Duwi Formation represents an initial stage of the Late Cretaceous marine transgression in Egypt [10,17]. The Gebel Duwi region extends in a northwest direction along the western coast of the Red Sea from south of Al-Qusseir to Safaga, between latitude 25°50' and 26°67'N and longitude 33°45' and 34°25'E (Fig. 1A), covering an area of about 500 km² [18].

The general lithological compositions of the Duwi Formation in studied localities are presented in Fig. 2. The Duwi Formation is usually subdivided into three members by Said [19] and Temraz [18]. In [19], Said extended the use of the term Duwi Formation to laminated gray clays and chert phosphatic bands at Safaga and subdivided the whole section in the Red Sea area into three members, which are Atshan or “A”, middle Duwi or “B” member and lower Abu Shegela or “C” member. The Atshan or “A” member is separated from the middle Duwi or “B” member by an Oyster limestone bed 6–16 m in thickness; while the lower Abu Shegela, or “C” member, is separated from the middle member by a shale unit of variable thickness (6–10 m). In the present time, the Duwi Formation is subdivided into four members, which are the lower, the middle, the upper and the uppermost members by Baioumy and Tada [10] and Baioumy et al. [17]. The oil shale beds are concentrated in the Atshan or “A” and middle Duwi or “B” members.
3. Material and methods

3.1. Sample materials

Twenty samples were collected from the underground mines at six localities at the central part of the Eastern Desert of Egypt along Red Sea Coast. Details of the location latitudes and longitudes are shown in Table 1 and Fig. 1A. Fig. 1A shows the remote sensing land sat image that was processed by ENVI 4.5© for the study region. The samples were cut directly from the unweathered fresh rock exposed surface inside the mine just under the phosphate layers, which increases the chance to avoid collecting oxidized samples. Two to four samples, weighing about one kilogram each, were collected from each locality.
mine. The samples were then transferred to the laboratory for processing. The samples with relative high total organic matter (TOC wt%) are selected for palynological examination after they were processed by the rock eval pyrolysis.

3.2. Rock eval pyrolysis

Twenty samples were processed with the “Rock Eval 6” instrument. About 60 mg of each sample is progressively heated at 550 °C under an inert atmosphere using special temperature program. The details of the aforementioned methods are explained in Espitalié et al. [20], Teichmüller and Durand [21], Philip and Galvez-Sinibaldi [22] and Tyson [23]. During the heating course, hydrocarbons are progressively released from the sample. Pyrolysis was carried out to determine four parameters, which are $S_1$, $S_2$, $S_3$ and $T_{\text{max}}$ (°C). Free or adsorbed hydrocarbons ($S_1$) are the first to volatile without cracking of the kerogens in moderate temperature as measured
produced from combustion of hydrocarbons of (mg HC/g sample) and the other pass through a thermal conductivity detector (TCD) measuring \( (S_1) \) in (mg) of CO₂.

The maximum temperature \( (T_{\text{max}}) \), at which the hydrocarbon generation occurred during pyrolysis, is the fourth parameter measured during the pyrolysis and it is used mostly to determine the maturation stage. The fifth measured parameter is the total organic matter (TOC), which is used to measure the potential quantity of produced hydrocarbon. Generally, rock is considered as a potential source for hydrocarbons if it contains 1–2% TOC. \[24\] (Table 4). The previously mentioned parameters are used mathematically to calculate the hydrogen index (HI = \( S_2/\text{TOC} \times 100 \)) and oxygen index (OI = \( S_3/\text{TOC} \times 100 \)), which are cross-plotted in the well-known modified van Krevelen diagram. The latter diagram and HI versus the \( (T_{\text{max}}) \) diagram are used to control the potentiality of the source rocks to generate oil or gas by obtaining different types of hydrocarbon products and kerogens.

### 3.3. Palynological preparation

The initial intention for the palynological preparation was to extract all the dispersed organic matter (palynodebris) via applying the standard palynological technique. Forty grams of each sample were treated with hydrochloric acid (HCl) and hydrofluoric acid (HF) respectively in order to remove the carbonates and silicates from the sediments. The residue was sieved using a 10 µm mesh sieve. The residue was then divided into two portions. The first was oxidized by Schultz’s solution to remove the needless surplus organic matter and a couple of slides were prepared from this portion using UV adhesive as a mounting medium. The other portion will be used later for the palynofacies analysis. The palynomorphs were examined and photographed by Leica microscope that is equipped with a digital camera model Leica D280. The counting process involved all the examined palynomorphs in a pair of slides. Different dinoflagellates and sporomorphs were identified according to Schrank \[25–27\], El Beialy \[7\] and Hoek et al. \[28\]. The peridinioids/gonyaulacoids ratio (P/G ratio) is adapted in this study and it is a reliable indicator for upwelling \[23,29\]. Eshet et al. \[29\] suggested that the variations in the statistical peridinioid/gonyaulacoid (P/G) dinoflagellate ratio mainly reveal flux in productivity and upwelling intensity in their studied area, clearly high P/G values means an elevated productivity.

### 3.4. Preparation of palynofacies analysis and ternary plots

The palynofacies of the same samples were studied to interpret paleoenvironmental conditions under which the oil shales were deposited and to evaluate their hydrocarbon potentiality (oil or gas prone). The study involved the other portions of the palynological residues, which were left without oxidation and elimination of organic matters. They were mounted directly onto slides in order to get the palynomorphs subsequently examined, counted and classified into seven palynofacies classes. The classification followed the schema of Tyson \[23\]. This classification facilitates the using of Tyson type ternary plot of palynofacies \[23\]. The palynofacies classes included amorphous organic matter (AOM), black debris, degraded phytoclasts, structured phytoclasts (wood, parenchyma, and cuticle), marine palynomorphs (dinoflagellate cysts, acritarchs and foraminiferal test inner linings (FTL)), fungal remains

### Table 1 Details of the sample locations. The latitude and longitude of the mines arranged from north to south.

<table>
<thead>
<tr>
<th>Mine</th>
<th>Latitude</th>
<th>Longitude</th>
<th>Samples</th>
</tr>
</thead>
<tbody>
<tr>
<td>Mohamed Rabah</td>
<td>26°34’52.03”N</td>
<td>33°49’6.30”E</td>
<td>1</td>
</tr>
<tr>
<td>Umm El Huieit</td>
<td>26°29’46.64”N</td>
<td>34°0’23.60”E</td>
<td>2, 3</td>
</tr>
<tr>
<td>Wasif</td>
<td>26°27’23.81”N</td>
<td>33°51’22.06”E</td>
<td>4, 5</td>
</tr>
<tr>
<td>El Nakheil</td>
<td>26°11’23.28”N</td>
<td>34°2’47.05”E</td>
<td>6–8</td>
</tr>
<tr>
<td>Younis</td>
<td>26°7’58.90”N</td>
<td>34°13’48.18”E</td>
<td>9, 10</td>
</tr>
<tr>
<td>El Beida</td>
<td>25°57’35.63”N</td>
<td>34°13’30.40”E</td>
<td>11</td>
</tr>
</tbody>
</table>

### Table 2 Distribution of the dinoflagellate assemblages through the studied samples. The numbers refer to the percentages of each species from the total of the assemblage. The P/G refers to the peridinioids/gonyaulacoids ratios calculated from the absolute counts of dinoflagellate individuals.
(spores, hyphae and mycelia) and sporomorphs (spores and pollen). The counting process involved one thousand particles per sample, which is reliable for the statistical purposes and ternary diagrams. These counts exceed the 500 individuals that are required to carry out such plot [23]. The proximal–distal trends generally control the dominant proportions of the palynofacies classes in any basin. The phytoclasts will be dominant in the proximal parts (high terrestrial influence) and the AOM will be dominant in the distal parts (more marine influence) [23].

Another important environmental ternary diagram is suggested during the course of the present study, which is the prasinophyte–peridinioids–gonyaulacoids plot. This diagram is found to be useful in determining the influence of the environmental processes that are specifically involved in the mechanism of oil shale formation. This plot is suggested for the first time in this study and needs further investigations and modifications.

### Table 3
Percentages of the palynofacies components in the studied samples. Each of them is represented by bar diagram at right side to the values.

<table>
<thead>
<tr>
<th>Location</th>
<th>AOM (%)</th>
<th>Phytoclasts (%)</th>
<th>Palynomorphs (%)</th>
<th>FTL (%)</th>
<th>Dinoflagellate (%)</th>
<th>Pollen (%)</th>
<th>Spores</th>
</tr>
</thead>
<tbody>
<tr>
<td>Mohamed Rabah</td>
<td>82.32</td>
<td>12.1</td>
<td>0.96</td>
<td>0</td>
<td>0</td>
<td>0</td>
<td>0.96</td>
</tr>
<tr>
<td>Wasif</td>
<td>29.33</td>
<td>60.29</td>
<td>0.32</td>
<td>0</td>
<td>0.32</td>
<td>0</td>
<td>0</td>
</tr>
<tr>
<td>Umm Hueitat</td>
<td>92.77</td>
<td>6.22</td>
<td>0.99</td>
<td>0</td>
<td>0</td>
<td>0</td>
<td>0</td>
</tr>
<tr>
<td>El Nakheil</td>
<td>59.56</td>
<td>24.96</td>
<td>6.64</td>
<td>1.99</td>
<td>4.64</td>
<td>0.13</td>
<td>0</td>
</tr>
<tr>
<td>Umm Hueitat</td>
<td>93.62</td>
<td>0.84</td>
<td>0.42</td>
<td>0.33</td>
<td>0.42</td>
<td>0.08</td>
<td>0</td>
</tr>
<tr>
<td>El Beida</td>
<td>97.77</td>
<td>2.22</td>
<td>0</td>
<td>0</td>
<td>0</td>
<td>0</td>
<td>0</td>
</tr>
</tbody>
</table>

4. Results

4.1. Palynology

4.1.1. Palynomorph assemblages

The diversity and abundances of dinoflagellate assemblages are heterogeneous and varying through the different studied locations. The assemblages from El-Nakheil and Umm Hueitat mines are almost identical in their content and they are consisting totally of highly abundant and moderately diverse peridinioids and lesser abundant gonyaulacoids. The peridinioids are as follows: *Andalusiella polymorha*, *A. polymorha* subsp. *A*, *A. polymorha* subsp. *B*, *Alterbidinium acutulum*, *Chatangiella micracantha*, *Chatangiella williamsii*, *Palaeocystodinium australinum*, *Palaeocystodinium golzowense*, *Palaeocystodinium gabonense*, *Palaeoperidinium* sp. *A*, *Cerodinium obliquipes*, *Lejeune cysta hyaline*, *Senegalinium bicavatum*, *Senegalinium granulostriatum*, *Senegalinium laevigatum*, *Senegalinium* sp. 1,
Trithyrodinium robustum, Tarssipheridium gemitoporum and Phelodinium tricuspid. The gonyaulacoids are Adnatospheari-
um buccinum, Cleistosphaeridium huguoniitii, Exochosphae-
rium bifidum, Hystrichosphaeridium sp. A, Imbatodinium cf. I.
inflatum, Spiniferites supparus and Spiniferites ramosus. On
the other hand, in the Younis mine, the dinoflagellate assem-
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and Hystrichosphaeridium sp. A. The peridinioids are mainly
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sp. A. In the samples from El-Beida and Wasif mines, the dinof-
flagellate assemblages are composed of peridinioids such as P.
australinum and Alterbidinium acutulum and gonyaulacoids like
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the dinoflagellate assemblages through the studied samples is
presented in Table 2. The identified dinoflagellates are photo-
graphed and presented in Plate 1 for peridinioids and Plate 2
for gonyaulacoids respectively.

The sporomorph assemblages are very scarce and limited
to some grains of angiosperm pollen Classopollis sp. A, Echino-
monocolpites densus, Liliacidites sp. A, Tricolpites sp. A,
Tricolporopollenites sp. A and pteridophytic spore Cyathidites
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samples from Younis, Mohamed Rabah, Wasif and Umm
Hueitat mines. No sporomorphs were recovered from El-Beida
and El-Nakheil samples.

4.1.2. The peridinioids/gonyaulacoids ratio (P/G)
The calculation of the (P/G) ratio was possible in most of the
samples. Yet, the values are considered only statistically signif-
ificant in samples that contain dinoflagellate assemblage at
least fifty individuals (n = 50). The highest values of the (P/G)
ratio are found in El-Nakheil (P/G = 5.44) and Umm Hueitat
(P/G = 11.6) (Table 2). These elevated values are the result of
the dominance of the peridinioid dinoflagellates in these two
mines (El-Nakheil, sum = 185 and species count = 16; Umm
Hueitat, sum = 97 and species count = 10). The values at the
Younis mine (P/G = 0.35) are the lowest, because the domi-
ant dinoflagellate species are gonyaulacoids (sum = 54 and
species count = 8). The other values from El-Beida, Mohamed
Rabah and Wasif are in Table 2.

4.2. Palynofacies descriptions
The studied samples can be assembled into two palynofacies
types depending on the statistical characteristics of their
palynofacies components (palynomorphs, phytoclasts and
AOM). The relative changes in the samples from the different
mines are presented in pie diagrams given in Fig. 1(B). The ter-
nary plot of the palynofacies analysis in this study is presented in
Fig. 3, while the fields that are occupied by the samples and ker-
gen types are displayed in Fig. 1(C). The relative distributions
of the palynofacies classes are obtainable in Table 3. The palynofacies
types are:

4.2.1. Palynofacies type 1
It includes the samples from Umm Hueitat, Wasif, El-Nakheil
and El-Beida. It is distinguished with relatively high content of
the amorphous organic matter (AOM) (≥92.00%) and rela-
tively low phytoclasts and with/without high abundances of
marine palynomorphs (dinoflagellates and foraminiferal test
lining). Some samples from Umm Hueitat (65.33–98.65%), El-
Nakheil (97.77%), El-Beida (94.72%), Wasif (92.77%) and
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organic matter and these samples are located in fields of IX and
VIII in the Tyson type ternary plot [23]. The nature of the AOM
is slobbery structure-less homogeneous with diffuse ends, Plate 2
(17 and 19). Some samples namely; El-Nakheil, El-Beida and
Mohamed Rabah mines from this palynofacies type contain
abundant prasinophytes.

<table>
<thead>
<tr>
<th>Table 4</th>
<th>Guidelines for pyrolysis parameters of quality, quantity and thermal maturity (adapted and modified after both Tyson, [23] and Peters and Cassa [24]). In this table, we suggest to add the kerogen type obtained from Tyson, 1993 ternary diagram in a new field in the original Peters and Cassa [24] table. The parametric values of El-Nakheil and El-Beida samples are highlighted by the dark gray in their comparative cells.</th>
<th>433</th>
</tr>
</thead>
<tbody>
<tr>
<td>Quantity</td>
<td>TOC (wt%)</td>
<td>S1 (mg HC/g rock)</td>
</tr>
<tr>
<td>Poor</td>
<td>0-0.5</td>
<td>0-0.5</td>
</tr>
<tr>
<td>Fair</td>
<td>0.5-1</td>
<td>0.5-1</td>
</tr>
<tr>
<td>Good</td>
<td>1-2</td>
<td>1-2</td>
</tr>
<tr>
<td>Very good</td>
<td>2-4</td>
<td>2-4</td>
</tr>
<tr>
<td>Excellent</td>
<td>&gt;4</td>
<td>&gt;4</td>
</tr>
<tr>
<td>Quality</td>
<td>HI (mg HC/g TOC)</td>
<td>S2/S1</td>
</tr>
<tr>
<td>None</td>
<td>&lt; 50</td>
<td>&lt;1</td>
</tr>
<tr>
<td>Gas</td>
<td>50-200</td>
<td>1-5</td>
</tr>
<tr>
<td>Gas and oil</td>
<td>200-300</td>
<td>5-10</td>
</tr>
<tr>
<td>Oil</td>
<td>300-600</td>
<td>10-15</td>
</tr>
<tr>
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<td>&gt;600</td>
<td>&gt;15</td>
</tr>
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Mohamed Rabah mines from this palynofacies type contain
abundant prasinophytes.
Plate 1  The depth and the specimen diameter are indicated. All the specimens are stored in the biostratigraphy laboratory in the Exploration Department in the Egyptian Petroleum Research Institute (EPRI). All magnifications are x400, unless otherwise is indicated.  
4.2.2. Palynofacies type 2

The Phytoclasts are relatively high and exceed the AOM in the samples taken from Younis (88.45% and 79.90%) and in one of the samples of Wasif (67.02%). The AOM ranged between 19.29% and 32.61% and the dinoflagellates are scarce. The samples of this type of palynofacies are located in field II in the Tyson type ternary plot.

Plate 2

<table>
<thead>
<tr>
<th>Image</th>
<th>Description</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>Exechosphaeridium bifidum, Sample 4, Umm Huweitat, 55 μm.</td>
</tr>
<tr>
<td>2</td>
<td>Cleistosphaeridium huguoniotii, sample 4, Umm Huweitat, 51 μm.</td>
</tr>
<tr>
<td>3</td>
<td>Hystrichosphaeridium sp. A, sample 4, Umm Huweitat, 56 μm.</td>
</tr>
<tr>
<td>4</td>
<td>Spiniferites supparus, sample 4, Umm Huweitat, 39 μm.</td>
</tr>
<tr>
<td>5</td>
<td>Xenascus ceratioides, sample 4, Umm Huweitat, 42 μm.</td>
</tr>
<tr>
<td>6</td>
<td>Tarsispheridium geminiporatum, sample 4, Umm Huweitat, 89 μm.</td>
</tr>
<tr>
<td>7</td>
<td>Adnatosphaeridium buccinum, sample 8, El-Nakheil, 61 μm.</td>
</tr>
<tr>
<td>8</td>
<td>Florentinia cooksoniae, sample 10, Younis, 55 μm.</td>
</tr>
<tr>
<td>9</td>
<td>Florentinia mantelli, sample 10, Younis, 56 μm.</td>
</tr>
<tr>
<td>10</td>
<td>Cluster of the gonyaulacoids dinoflagellates, 231 μm.</td>
</tr>
<tr>
<td>11</td>
<td>Cluster of the peridinioids dinoflagellates, 235 μm.</td>
</tr>
<tr>
<td>12</td>
<td>Prasinophyte phycoma, sample 6, El-Nakheil, 212 μm.</td>
</tr>
<tr>
<td>13</td>
<td>Structuraless amorphous organic matter, sample 6, El-Nakheil, 234 μm.</td>
</tr>
<tr>
<td>14</td>
<td>Amorphous organic matter with diffused edges, Umm El Huweitat, 120 μm.</td>
</tr>
<tr>
<td>15</td>
<td>Foraminiferal test linings, sample 4, Umm Huweitat, 69 μm, 75 μm.</td>
</tr>
</tbody>
</table>

4.3. The prasinophyte-peridinioids-gonyaulacoids ternary plot

The studied samples are distributed in three fields according to the proportions of the prasinophyte and peridinioids as well as gonyaulacoids dinoflagellates (Fig. 4).

Field A: it includes the samples from the Wasif and Younis mines and they are characterized by the absence of prasin-
Figure 3 Tyson type ternary plot of the palynofacies distribution interpretation. All the samples are plotted in the fields IX and VIII except two samples from Wasif and Younis are plotted in filed II. Field I = gas prone type III; field II = gas prone type III; field IV = gas prone type III; field II = gas prone type III; field III = gas prone type III or IV; field IV = gas prone type III or II; field V = gas prone type III > IV; field VI = oil prone type II; field VII = oil prone type II; field VIII = oil prone type II > I; field IX = highly oil prone type II or I.

Figure 4 Peridinioids–gonyaulacoids–prasinophyte phycoma ternary diagram is proposed in this study to determine which environmental parameter has a greater influence in the mechanism of oil shale formation.
ophytes, Plate 2 (15 and 16), low peridinioids and abundant
gonyaulacoids. The TOC content of the samples from this
field is interpreted as a result of the effect of low oxygen
content in the depositional environment (anoxic condi-
tions), which could accompany the water stratification
and absence of upwelling when prasinophytes increase.

Field C: it includes samples from Umm El Hueitat,
El-Nakheil and Wasif. They are characterized by the abun-
dant peridinioids, low gonyaulacoids and absence of prasin-
ophytes. The concentration of the TOC in this field resulted
from the high water surface productivity accompanied by
the upwelling events.

Field D: it includes samples from the Mohamed Rabah,
El-Nakheil and El-Beida, which are characterized by the
abundant prasinophyte, low peridinioids and gonyaulac-
oids. The TOC content in this field resulted from the depo-
sition in distal basin under low sedimentation rates.

4.4. Geochemical analyses

The parameters measured are presented in Table 5, while their
graphical presentation, cross plotting interpretations (modified
van Krevelen diagram, HI versus OI and HI versus $T_{\text{max}}$) and

Table 5 Geochemical parameters that measured by the rock eval pyrolysis for the studied oil shale samples. The sample numbers referred to the palynological samples. The samples without indicative numbers are omitted from the palynological investigations.

<table>
<thead>
<tr>
<th>Mine name</th>
<th>Palynological samples number</th>
<th>TOC</th>
<th>$S_1$</th>
<th>$S_2$</th>
<th>$S_3$</th>
<th>$T_{\text{max}}$</th>
<th>PI</th>
<th>HI</th>
<th>OI</th>
<th>Kerogen type ($S_1/S_3$)</th>
<th>Production yield ($S_1 + S_3$)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Mohamed Rabah 1</td>
<td>9.66</td>
<td>1.3</td>
<td>58.25</td>
<td>4.04</td>
<td>416</td>
<td>0.02</td>
<td>603</td>
<td>42</td>
<td>14.42</td>
<td>59.55</td>
<td></td>
</tr>
<tr>
<td>Wasif 2</td>
<td>1.36</td>
<td>0.1</td>
<td>0.51</td>
<td>1.24</td>
<td>415</td>
<td>0.16</td>
<td>38</td>
<td>91</td>
<td>0.41</td>
<td>61</td>
<td></td>
</tr>
<tr>
<td>3</td>
<td>8.39</td>
<td>0.98</td>
<td>42.73</td>
<td>6.86</td>
<td>421</td>
<td>0.02</td>
<td>506</td>
<td>82</td>
<td>6.23</td>
<td>43.71</td>
<td></td>
</tr>
<tr>
<td>Umm Hueitat 4</td>
<td>3.02</td>
<td>0.23</td>
<td>12.93</td>
<td>1.17</td>
<td>409</td>
<td>0.02</td>
<td>428</td>
<td>39</td>
<td>11.05</td>
<td>13.16</td>
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</tr>
<tr>
<td>5</td>
<td>2.6</td>
<td>0.32</td>
<td>11.63</td>
<td>1.6</td>
<td>427</td>
<td>0.03</td>
<td>447</td>
<td>62</td>
<td>7.27</td>
<td>11.95</td>
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<tr>
<td>El Nakheil 6</td>
<td>19.18</td>
<td>4.18</td>
<td>130.19</td>
<td>8.17</td>
<td>418</td>
<td>0.03</td>
<td>679</td>
<td>43</td>
<td>15.94</td>
<td>134.37</td>
<td></td>
</tr>
<tr>
<td>7</td>
<td>21.94</td>
<td>4.05</td>
<td>151.98</td>
<td>6.79</td>
<td>418</td>
<td>0.03</td>
<td>693</td>
<td>31</td>
<td>22.38</td>
<td>156.03</td>
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</tr>
<tr>
<td>8</td>
<td>22.23</td>
<td>4.5</td>
<td>159.22</td>
<td>6.54</td>
<td>418</td>
<td>0.03</td>
<td>716</td>
<td>29</td>
<td>24.35</td>
<td>163.72</td>
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</tr>
<tr>
<td>Younis 9</td>
<td>1.77</td>
<td>0.14</td>
<td>1.66</td>
<td>1.68</td>
<td>421</td>
<td>0.08</td>
<td>94</td>
<td>95</td>
<td>0.99</td>
<td>1.8</td>
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<tr>
<td>El Beida 11</td>
<td>11.61</td>
<td>1.97</td>
<td>65.61</td>
<td>7.1</td>
<td>419</td>
<td>0.03</td>
<td>565</td>
<td>61</td>
<td>9.24</td>
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<tr>
<td>11.45</td>
<td>2.01</td>
<td>64.81</td>
<td>7.75</td>
<td>418</td>
<td>0.03</td>
<td>566</td>
<td>68</td>
<td>8.36</td>
<td>66.82</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

Figure 5 Van Krevelen-type diagram (van Krevelen, 1993) of hydrogen/oxygen indices (HI/OI) plotted for the studied samples. The plots show a predominance of type I and II kerogen.

Figure 6 HI versus $T_{\text{max}}$ diagram (modified from Tyson [23]).
the thermal maturity are shown in Figs. 5–7 respectively. All the samples that were geochemically analyzed from the different locations varied between poor and excellent production potential according to the standard guidelines of Peters and Cassa [24] (Table 4). The samples from El-Nakheil (19.18–22.23 wt%) have the highest TOC values and they are considered as excellent producers. The samples from El-Beida (11.45–11.61 wt%), Mohamed Rabah (9.66 wt%) and Wasif (8.39 wt%) are much lower than El-Nakheil however still excellent producers. The samples from Umm Hueitat (2.6–5.18 wt%) have a lower potentiality as it is ranging from very good to excellent. The samples from Younis mine (0.04–1.77 wt%) have the lowest production potentiality of all samples and are considered as poor to good. The ($S_1$) values are generally low in the samples of Mohamed Rabah (1.30 mg HC/grock), Wasif (0.10–0.98 mg HC/grock), Umm Hueitat (0.23–0.39 mg HC/grock) and are mainly related to low maturity of these samples despite their high content of TOC. Whereas, the sample from Younis (0.14 mg HC/grock) is mainly related to low maturity of the rock samples and low TOC Content. The low values of ($S_1$) should reduce production potentiality ($S_1 + S_2$). The values of $S_1$ of the samples from El-Nakheil (4.05–4.18 mg HC/grock) and El-Beida (1.97–2.01 mg HC/grock) are relatively high, which in turn indicate that they are very good to excellent producers. The values of Hydrogen index (HI) in the samples from El-Nakheil (679–716 mg HC/g TOC), Mohamed Rabah (603 mg HC/g TOC) and El-Beida (566 mg HC/g TOC) indicated high quality oil prone kerogen type I. The other samples are oil prone as well; however, type II kerogen was detected. Two
samples from Wasif (38 mg HC/g TOC) and Younis (94 mg HC/g TOC) indicated gas prone type IV kerogen. The measured $T_{\text{max}}$ (409–427 °C) in all samples indicated immature kerogens to yield hydrocarbons (Figs. 6 and 7).

5. Discussion

5.1. Palynostratigraphy

Although it is economically mined, the accurate age of the Duwi Formation is questionable and requires additional investigations. The age of the Duwi Formation was recorded in several previous works, which depended on different types of fossils. El Deftar et al. [30] and Issawi et al. [31] assigned the age of the Duwi Formation as Maastrichtian depending on ammonites and bivalves, while Awad et al. [32], Issawi [33], Abd El-Razik [34] assigned its age as Late Campanian depending on the same fossil groups. Glenn and Arthur [35] considered the age as Late Campanian–Early Maastrichtian depending on the nanofossils, this age is coinciding with previous palynological studies (e.g. El Beialy [7]; Schrank [25,27]; Ganz et al. [36]; Schrank and Perch-Nielsen, [37]). The phosphate workers preferred and considered the later age [10,17,38]. The previous palynological studies examined samples from some mines investigated in the present study as Mohamed Rabah, Younis, Umm Hueitat and El-Nakheil (as have been reported by El Beialy [7], Schrank and Perch-Nielsen [37] and Schrank [27]). El Beialy [7] did not consider the El-Nakheil mine in his study because the samples were barren. The palynostratigraphy in these studies lacked a distinctive zonation and the Campanian/Maastrichtian boundary remains uncertain. This boundary is defined at the top of the dinoflagellate species *Xenascus ceratioides* in extensive palynostratigraphic study as studied by Hoek et al. [28] on Campanian–Maastrichtian sequence from Beer Sabaa Valley and Shefela Basin.

The recovered dinoflagellates showed moderate abundances in El-Nakheil, Umm Hueitat, Younis, and Wasif and poor abundance at El-Beida and Mohamed Rabah. The dinoflagellate assemblages investigated in the present study are very similar to those described from the *X. cerastioides* Interval Zone of the latest Campanian age [28]. This zone ranges from the last occurrence of *Odontochitina costata* in Campanian to the first occurrence of *Diphyes colligerum* in Maastrichtian. No *X. cerastioides*, *D. colligerum* or *O. costata* individuals were observed in the studied samples. The occurrences of *P. australinum* and *T. robustum* compensated the absence of the marker species and should indicate Late Campanian–Early Maastrichtian according to Hoek et al. [28]. The *P. australinum* was recorded in the Early Maastrichtian to early Eocene from Australia, Argentina, Turkey and Morocco [39–42], the late Maastrichtian from Spain [43] and it was recorded in the Late Campanian in Palestine [28]. Schrank and Perch-Nielsen [37] and Schrank et al. [27] reported the first occurrence of the *P. australinum* in Early Maastrichtian Dakhla Formation because they did not record it in the older sediments of the Duwi Formation. The *P. australinum* was recorded in samples from Duwi Formation at the Hamrawein mine [7], and El-Nakheil and Umm Hueitat in the present study. *T. robustum* was recorded in the Early Maastrichtian from The Netherlands, Demark, Maryland U.S.A. Albany [44–46], in the latest
Campanian to Early Maastrichtian of the Atlantic Coastal Plain and Palestine [28,47] and the Maastrichtian of Morocco [42,48]. Our findings support the Late Campanian–Early Maastrichtian age for the studied oil shale samples. The oil shale beds in the Mishash and Gharib formations deposited in the time interval of *X. cerastioides* Interval Zone [28]. There are great similarities in the recorded dinoflagellate assemblage from this zone and that recorded from the oil shale samples from El-Nakheil and Umm Hueitat in the present study. Both have a high abundance of the peridinioids (*Palaeocystodinium* spp., *Cerodinium* spp. and *Andalusiella* spp.). These similarities indicate a clear relationship and the resemblance in the depositional conditions or corresponding sedimentation. Both locations belong to the southern Levant upwelling system of Parrish and Curtis [9] (Fig. 8).

5.2. Paleoenvironmental interpretation

The palynofacies analysis is an insightful tool for the paleoenvironmental interpretation in terms of sea and oxygen levels [23]. The palynofacies interpretations are integrated in the present study with the peridinioids/gonyaulacoids ratio (P/G) to indicate the paleoproductivity (Table 6). The palynofacies type 1 is composed of the slobbery structure-less homogenous AOM, abundant prasinophytes, and low terrestrial phytoclasts and sporomorphs input. These compositions accomplish the characteristics of IX and VIII fields on the Tyson type ternary diagram. The samples plotted in the field IX with low dinoflagellate abundances or low (P/G) ratio are interpreted by the deposition in the suboxic/anoxic distal part of deep basin low sediment supply (sediment starvation or condensation) [23]. This situation exists during the rising in sea level (transgressive system tract); particularly during the flooding or transgressive surfaces [23,49]. The sediment starvation prevents dilution of AOM [23,50]. Whereas, the samples that are plotted in the IX and VIII fields with moderate dinoflagellate abundance and high (P/G) ratio are interpreted by deposition in shallower basin under suboxic to anoxic conditions and relatively high productivity (active upwelling). This situation exists during high sea level with moderate terrestrial influx (high stand system tract) (Figs. 9 and 10).

The samples of palynofacies type 2 contain much more phytoclasts, low dinoflagellate and no prasinophytes. The samples are plotted in the II field in the Tyson type ternary diagram. They deduce proximal marginal basin with dysoxic conditions. These samples are mostly deposited in the low sea levels (low stand system tract) with higher terrestrial input (Figs. 9 and 10).

5.3. Kerogen types and hydrocarbons generation potentiality

The kerogen is carefully defined as the organic content of the sedimentary rock, which is not soluble in the ordinary organic solvents [51]. The kerogen can be discriminated into four types as follows: type I (algal kerogen), type II (liptinitic kerogen), type III (humic or coaly kerogen), and type IV (inertinitic

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**Figure 9** Schematic diagram for the processes that are involved during the deposition of the oil shale and their possible combinations and interrelations.
The kerogen types can be evaluated separately from the palynofacies examination under the microscope and from the geochemical analysis by rock eval pyrolysis. The palynofacies investigations in the present study match organic geochemical analysis (Table 6). The kerogen of highly oil prone type I and type II is encountered in samples from El-Nakheil, Umm El Hueitat and Mohamed Rabah mines with elevated TOC, AOM and rich algal prasinophytes. This indicates the high potentiality to generate hydrocarbons in the studied samples. The presence of abundant prasinophytes is typical in the oil prone type I kerogen [23]. The kerogen gas prone type III and IV came across atWasif and Younis mines with abundant phytoclasts and low TOC. The abundant phytoclasts are typical in the palynological assemblages of these kerogen types [23]. These samples are of less potentiality to produce hydrocarbons than the samples from the other mines.

All the examined kerogen in the studied samples were generally immature, so that they cannot yield hydrocarbons as pointed from $T_{\text{max}} (<435 \, ^\circ C)$. However, they are still economically worthy for direct combustion mining and they can be artificially treated by retorting for fuel extraction.

5.4. Mechanism of oil shale deposition and its sequence stratigraphy implications

The High dinoflagellate (P/G) ratio values indicate the high paleoproductivity rates, which in turn are the precursor of active upwelling (Eshet et al., [29] and discussion therein). The phosphate and oil shale beds in the Duwi Formation are part of the upwelling belts existed down the southern Tethys margin during the Late Campanian and Early Maastrichtian (Fig. 8) [9]. This was confirmed by the similarity in the dinoflagellate associations in some of the studied samples compared to those conducted from other areas in the neighboring countries in the upwelling belt (e.g. Eshet et al. [29] and Hoek et al. [28], Mishash-Palestine) as previously mentioned. The same is found in case of TOC content in the studied samples, which showed remarkable high values (22.23 wt%) as the samples from the other areas in the Parrish and Curtis [9] upwelling belt. For example, the TOC values of the Oil Shale Member in Mishash Formation are ≤10 wt% [8], 18 wt% [12], 15–20 wt% [52] and up to 22.25 wt% at lower Muwaqqar Formation, Jordan [11]. These high TOC values are “exclusively” interpreted by the high paleopродuctivity rates and the existence of the very reducing environment or oxygen minimum zone (OMZ), (Duwi Formation, Egypt: Ganz [3], Ganz et al. [53], Schrank [26]; Oil shale member, Mishash Formation, Palestine: Bein et al. [52], Ashkenazi-Polivoda et al. [12], Schneider-Mor et al. [13], and lower part of Muwaqqar Formation, Jordan: Abed et al. [11]). This interpretation has some weaknesses (Katz [15] and Tyson [16] and the discussion therein).

The main weakness is that, the high productivity surface water or highly anoxic bottom water conditions are not necessarily accompanied with elevated organic matter content, in fact they are absent, in the recent upwelling zones or highly anoxic seas [14,16]. This interpretation also neglected the effect of low sedimentation rates, which certainly has a very effective and controlling influence on the concentration of organic matter.
[54–56]. Normally, the rate of organic matter sedimentation increases primarily with the sedimentation rate until a critical point is reached; the organic matter content becomes more diluted by mineral sediments and consequently decreases [57]. The low sedimentation rates are usually related to the formation of basin-ward condensed sections. The condensed sections are thin laminated stratigraphic units consisting of pelagic to hemipelagic sediments characterized by low sedimentation rates of less than 1 cm/year [54]. The condensed sections are relevant to the flooding surface at the peak of the sea level transgression and it is placed normally at the transgressive system tract. The main weakness of this interpretation, as a fundamental motive for the organic matter concentration, is that organic rich sediments can occur in other parts of the system tracts other than transgressive system tract [15]. Consequently, no sole and stand-alone model can interpret the deposition of organic rich sediments.

Therefore, an integrated model in which the three interpretations are combined together was suggested by Katz [15] and Tyson [16]. In reference to that model, the formation of the organic rich sediments is resulted of the interplay between these three factors (Fig. 9). The present study provides evidence of the low sedimentation rates that existed during the deposition of the studied oil shale beds from the Duwi Formation, which is the presence of abundant prasinophyte association. Plate 2(15 and 16). This is not suggested as an isolated single factor that led to concentration of the organic rich samples encountered in the present study, but as a main factor that was occasionally accompanied by high surface productivity rates and bottom anoxic conditions. The suggested prasinophyte-gonyaulacoids ternary plot, presented in Fig. 4, measures the influence of each of these three factors that might cooperate to produce the organic rich sediments, determines the most important influence and attempts to interpret the related environmental conditions. The abundant prasinophyte in the palynological association is indicating either water stratification or low sedimentation rates, depending on the abundances of the associated dinoflagellates [23]. This relationship was the key for the suggested ternary plot. The dominant peridinioids will indicate the active productivity while the prasinophyte will indicate the low sedimentation rates. On the other hand, the anoxia will be indicated from absence of both later palynomorph groups and the presence of gonyaulacoids. The anoxia resulting from the water stratification will be indicated by the abundant presence of both prasinophyte and gonyaulacoids. The abundant prasinophyte assemblage was recorded previously from the oil shale beds in the Duwi Formation, nevertheless their environmental value as an indicator for the rate of sedimentation did not grab high attention [27]. The same samples contain abundant AOM and are plotted in the field IX in the Tyson type ternary diagram, this field is typically interpreted by the deposition in sediment starved basin areas. The rate of sedimentation in the Duwi Formation could be obtained from the analysis of the complete lithologic section from Baioumy and Tada [10]. The later indicated that the oil shale bed deposition took place at the transgressive and high stand system tracts and spanned time interval between 76.6 and 75.00 Ma with in-situ comprising sedimentation rate of a 1.68 cm/ky (Fig. 10). Which is considerably lower than the sedimentation rate (2.4 cm/ky) of the oil shale beds at Mishash Formation that spans the time interval between 71.6 and 69.85 Ma [12]. Yet, both sedimentation rates relatively are not low enough to represent a condensed section. Despite this high rate, the high TOC values in the lower part of Mishash Formation could be resulted from the low sedimentation rate (see the oil shale lithologic section in Ashkenazi-Polivoda et al. [12]). Tyson [16] stated that, the very high TOC values (> 10%) are not common and generally reflect a combination of higher preservation (dysoxia–anoxia) and low dilution. Since the sedimentation rate of the oil shale beds in the Mishash Formation is reconsidered by the following: (1) it was calculated for the entire section, (2) the sedimentation rate could vary over time and (3) maximum TOC values are located just above a condensed layer. It can be postulated that the deposition of at least the high TOC at the lower part of the section took place under a relatively low sedimentation rates. These rates are continued from the younger condensed bed, although the palynological evidence is still unexamined. The oil shale of lower part of Muwaqqar Formation appeared in the condensed deposits in the distal part of the Arzaq and El Lajjun basins [11], which might also indicate that the rate of sedimentation played an important role in the deposition of the organic matter rich layers.

According to what has been mentioned before, the deposition of the condensed section can be accompanied with organic rich sediments, which consequently have a strong affinity to exist in specific position in the stratigraphic sequence cycle. The case will not change too much when the dominant influence in the organic rich sediments deposition is belonging to the high productivity or anoxia. Each has a specific position in the stratigraphic sequence cycle. These three factors (sedimentation rate, bottom water oxygen and surface productivity) will interact differently in each of system tracts. This interaction would produce organic rich sediments that vary in palynological content, kerogen type, organic carbon content and oil proneness. The relationship between the system tract position and the kerogen type in the studied samples is presented in Figs. 9 and 10 and Table 5. The samples with high TOC, high AOM and abundant prasinophyte are deposited during a flooding surface of the marine transgression (transgressive system tract). Likewise, the samples with high TOC, high AOM and high (P/G) ratio are deposited during the high stand system tract. On the other hand, the Low TOC, low AOM and abundant phytoclasts are deposited in the low stand system tract. The deposition of the organic rich sediments in the Duwi formation is stratigraphically related and can be arranged according to the position in the sequence cycle (Fig. 10 and Table 5). The Late Campanian transgression is well known in the Egypt [7,10,17,27]. This research findings support the deposition of the oil shale beds at the Duwi Formation during the sequence cycle of this transgression and at its maximum flooding system. This was followed by the high stand system tract accompanied with a period of active upwelling and high productivity. The deposition of the organic matter rich sediments or the oil shale beds in the Parrish and Curtis [9] upwelling province initially started at the Campanian transgression and its low sedimentation rates and high productivity rates came in the following high stands. The accumulation of the organic matter (high TOC) is under the influence of both the low sedimentation rates and the elevated paleoproductivity.

The future studies on the oil shale deposits should concentrate on the variation of the sedimentation rates through time and the related changes in the TOC wt% values, especially at
the initial stages of the accumulation of the large concentration of organic matter.

6. Summary and conclusions

The present study focused on the geochemical and palynological examinations of samples collected from oil shale beds underneath the phosphate layers in the Duwi Formation at El-Nakheil, El-Beida, Mohamed Rabah, Umm El Hueitat, Wasif and Younis mines. The palynological study included palynological dating of the studied samples and quantitative palynofacies examination. These examinations expounded the sequence stratigraphy framework and its palaeoenvironmental settings. The examinations revealed the following:

1. The identified dinoflagellate association abundance and diversities are fluctuating in the studied samples. The dinoflagellate assemblages from El-Nakheil, Umm Huetat are almost identical in their content and they are consisting totally of highly abundant and moderately diverse peridinioids and minor abundant gonyaulacoids. In the samples from El-Beida, and Wasif, the dinoflagellate assemblages are composed of peridinioids and gonyaulacoids. In the Younis mine, the dinoflagellate assemblage is composed mainly of gonyaulacoids. The spromorph assemblages are scarce in all samples and are not palynostratigraphically significant.

2. The dinoflagellate assemblages indicated Late Campanian–Early Maastrichtian and showed great similarities with the dinoflagellate assemblages from the oil shale beds of the same age in the Parrish and Curtis [9] upwelling province.

3. The geochemical analysis revealed that the sample from the El-Nakheil mine has an excellent potentiality to produce oil; their kerogen is immature oil prone type I or II. The samples from Mohamed Rabah, El-Beida, and Wasif are excellent oil prone type I or II. The samples from the Younis mine are poor to good gas producers; their kerogen is immature oil prone type I. The samples from El-Beida, and Wasif, the dinoflagellate assemblages are totally of highly abundant and moderately diverse peridinioids. The sporomorph assemblages are scarce in all samples and are not palynostratigraphically significant.

4. The palynofacies examination results strongly agreed with the geochemical analysis results.

5. The integration of the results from the palynological, palynofacies and geochemical analysis indicated that the organic rich sediments or oil shale beds in the Duwi Formation are deposited during latest Campanian–earliest Maastrichtian sequence. The samples with low organic matter are deposited in the low stand system tract. The samples with high organic matter content are deposited in the transgressive and high stand system tracts. The samples that deposited during the transgressive system tract are affected with the mineral sediment starvation. The high paleoproduction and upwelling were active during the high stand system tracts.

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