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Reconnaissance Lithological-Geochemical Exploration for Organic Matter and Total Organic Carbon in the Late Campanian-Paleocene Black Shale Belt, Upper Egypt

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Abstract

The provided factual data about the organic matter bearing black bituminous and oil shale in Western Desert, Nile Valley and Red Sea coastal zone of Egypt point to a promising potential source of energy. These black shales in Egypt belong to three stratigraphic formations namely Duwi at the base overlain by the Dakhla and the lower Calcareous Member of the Esna Formation. The objectives of the present study concern the stratigraphic position, lithological characteristics, hosted total organic matter (O. M.) and total organic carbon (TOC) contents in relation to depositional environments of the formations forming the black shale belt. Lithologicaly these shales are distinguished by presence of mud bands and goethite cubes pseudomorph after pyrite favoring deposition in oscillating marine environment from shallow neritic to inner neritic (littoral to sub-littoral) reducing environment supplied with muddy argillaceous sedimentation. The recorded organic matter in Duwi Formation is ranging from 22.33 wt. % (El Sebaiya) to 0.73 wt. % (Kom-Mir) in the Nile Valley, 28.08 wt. % (El Nakheil) to 8.12 (Zug El Bahar) in the Red Sea Coastal Zone, in the Dakhla Formation 13.49 wt. % (Eldeir) to 1.60 wt. % (Kom-Mir) in the Nile Valley, 16.91 wt. % (W. Abu Shigeila),1.61 wt. % (G. Duwi) in the Red Sea Coastal Zone, 14.61 wt. % to 3.49 wt. % (G. Gifata) in the Nile Valley and from 12.42 wt. % (W. Abu Shigeila) to 2.27 wt. % (G. Duwi) in the Red Sea Coastal Zone. The total organic carbon content ranges from 22.15-0.22 wt. % in the Duwi Formation, 0.82-0.13 in the Dakhla Formation and 0.14-0.11 in the Esna Calcareous Shale. The determined Total Organic Carbon contents in twenty samples are potentially suitable for petroleum: (Excellent-2 samples, very good-5 samples, good-9 samples, fair-3 samples, poor 1). The impact of the depositional environments of each formation on the O. M. and TOC contents is obvious.

Keywords: ***Black Shale, Depositional Environment, Organic Matter, Total Organic Carbon, Egypt.***

1. Introduction

Nearly all sediments contain detectable organic matter, the general average being about 2 %; in black shale the percentage is commonly a few percent but may rise much higher. Black shale in fact, grade into pure organic materials, into coal on the one hand and into bitumen on the other. The provided factual data about the organic matter bearing black bituminous and oil shale in Egypt point to a promising potential source of energy. These black shales in Egypt were considered by several authors belong to two stratigraphic formations namely, Duwi at the base conformably overlain by the Dakhla of Campanian-Early Maastrichtian and Late Maastrichtian - Danian age respectively. However; Samieh (1989) and Hassaan *et al*. (1987) recorded in the Lower Calcareous Shale Member of the Esna Formation in El-Deir-Mahrousa area, south west Qena presence of organic matter ranging from 19..13 to10.31 % calculated from the abnormally high loss on ignition values (L. O. I.) relative to CaO and MgO values . Therefore, the Upper Paleocene-Lower Calcareous Shale Member of the Esna Formation will be considered in this article a part of this belt.

The Early Campanian–Late Paleocene tremendous black shale belt bearing organic matter (Fig. 1) existing in Upper Egypt extends in the Southern part of the Western Desert from Dakhla Oasis to Sinn El-Kaddab Scarp, Nile valley district from Idfu to Esna and the Red Sea coastal zone from Qusseir to Safaga is considered by Hassaan and Ezz-Eldin (2007) promising source for organic matter.

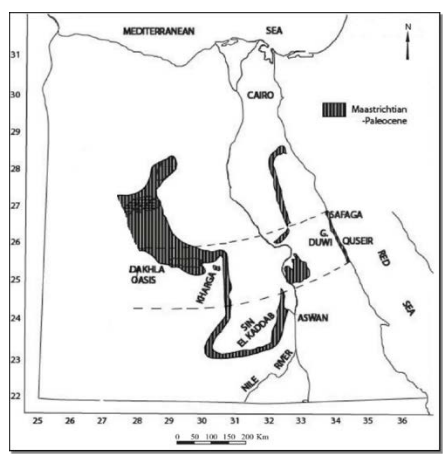

Fig. 1: The black shale belt in Egypt (After Hassan and Ezz Eldin, 2007).

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The present article deals with the stratigraphic position, lithological characteristics and distribution of the total organic matter (O. M.) in relation to depositional environments of these formations forming the black shale belt existing in the Upper Egypt. The objectives of the present study will be reached by undertaking field study and determination of both organic matter and total organic carbon contents in several representative sites of this belt distributed in these districts.

2. Field Study

The field studies undertaken during five field trips accomplished by studying and sampling fifteen sites representing the Western Desert, Nile Valley and Red Sea coastal zone where 48 shale samples were collected (Fig 2).

compared to other methods. The difference in weight of each dried sample at 110° C before and after ignition at 375° C for 16 hours represents the amount of the O. M. present in the sample according to the following equation:

$$\text{O. M. wt. \%} = \frac{pre..ignition..weight - post..ignition..weight}{pre..ignition..weight} \times 100$$

The slow temperature increase of 5° C/min was taken into consideration till the furnace has reached to the desired temperature (375° C).

In the present study this procedure was used to determine the O. M. % after ignition of each dried sample at 110° C for 2 hours at 375° C to re-estimate the time of ignition for save of coasts then weighed after cooling to estimate the probable amount of O. M. Moreover; each sample was ignited for 12 hours and weighed as a second step and finally for 16 hours.

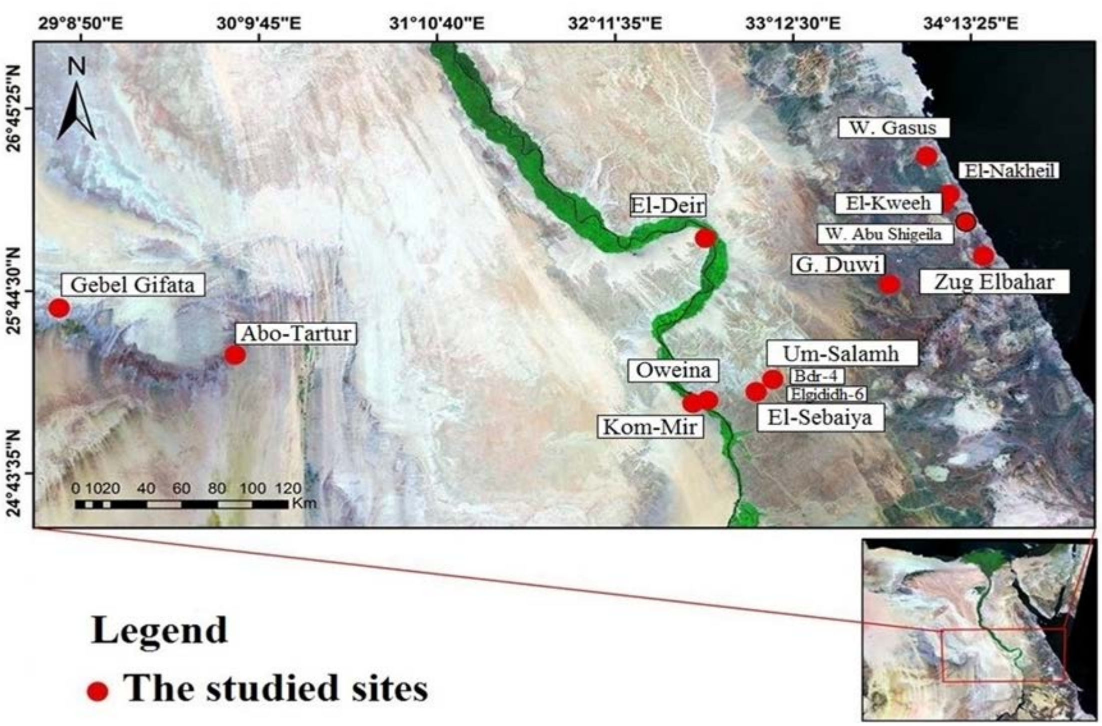

Fig. 2: Location map showing the representative sites.

3. Laboratory Work

The laboratory work was accomplished by determination of O. M. % in all collected samples and TOC in the samples containing promising O. M. content.

The organic matter (O. M.) in the 48 shale samples were determined using the Loss on Ignition (L.O.I.) method proposed by Sarah (2011) as a relatively simple procedure

The TOC was determined using a LECO Carbon Analyzer in the Geochemical Laboratory of the Egyptian Petroleum Researches Institute. Sample is combusted in an oxygen atmosphere and any carbon present is converted to CO_2. The sample gas flows into a non-dispersive infrared (NDIR) detection cell. The NDIR measures the mass of CO_2 present. The mass is converted to carbon percent based on the weight of dried sample.

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4. Lithological characteristics of the studied sites

The comprehensive study attempted by Issawi *et al*., (1999 and 2009) divided the huge and extensive Cretaceous sediments into eight facies; of the the Nile Valley Facies in which these black shales are included (Fig. 3).

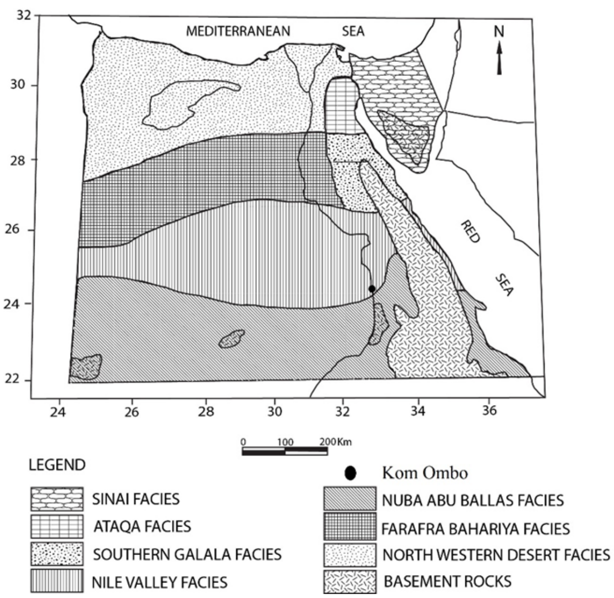

Fig. 3: Different Cretaceous facies in Egypt. (After Issawi *et al*., 1999, 2009).

The Nile Valley facies were deposited during the gradual transgression of the Tethys with time which covered a huge tract of Egypt during the Late Cretaceous. Probably at the same time the Indian Ocean, invaded Egypt during the Cretaceous (Issawi *et al*. 1999 and 2009) but certainly the deposits of this Sea reached only south Egypt up to the latitude of Kom Ombo (Lat. 24° 30' and 32° 30' N and long. 32° 50’ and 33° 25’ E, Fig. 3). This transgression resulted into deposition of the arenaceous Nuba-Abu Ballas facies (Taref Sandstone Member) that gradually changed in lithology and thickness into variegated shale of the Quseir Clastic Member during the Campanian to phosphatic beds interbedded with shale and occasionally Oyster limestone of the Duwi Formation till the Early Maastrichtian, followed by deposition of proper Dakhla shales (sandy south Kom Ombo) during the Late Maastrichtian-Early Paleocene. The Tarawan Chalk Formation and finally the Calcareous Shales Member of the Esna Formation considered a part of this belt were deposited during the Late Paleocene.

The results of field study of these formations and description of collected hand specimens of the studied localities representing the three main districts show the following stratigraphic position, lithological characterization and depositional environment of this black shale belt:

4.1 Western Desert District

The two studied locations representing this district are Gebel Gifata and Misr Company Abu-Tartur open-pit phosphate mine. The two sampled exposed sections are 16, 19 m. thick respectively. This difference in thickness is due to coverage of the lower part of the Dakhla Formation by weathered shale and aeolian sand at Gebel Gifata located to the west of the Nile Valley. The color of its shale beds varies from pale grey at the base of the section to dark grey due to presence of discontinuous mud bands (Fig. 4) via pale yellowish grey with goethite cubes pseudomorph after pyrite at the top. The yellowish coloration is a product of oxidation of the pyritic cubes.

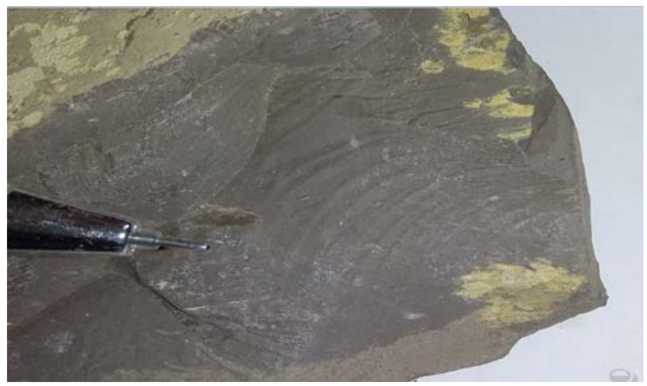

Fig. 4: Mud bands in shale, Abu-Tartur.

The following is the stratigraphic correlation chart showing the two studied sections representing the Dakhla Formation cropping out in the Western Desert as well as the locations of the collected samples (Fig. 5).

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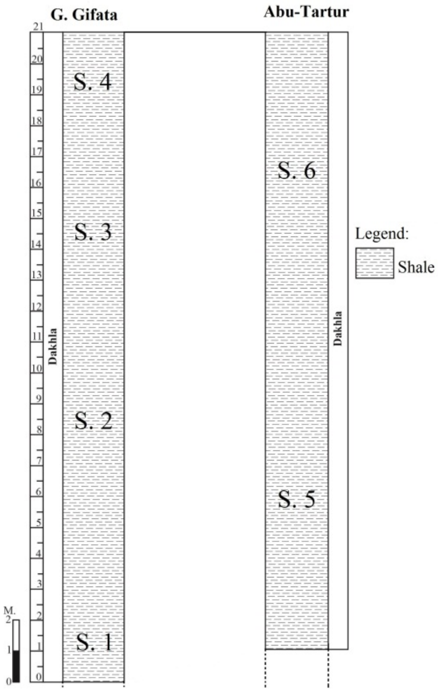

Fig. 5: Lithostratigraphic Correlation of the two studied sections, Western desert.

4.2 The Nile Valley District

The shale beds of the Duwi Formation at El Sebaiya open-pit mine are generally compact, the lower bed (Fig. 6) is yellowish grey (S. 11) while the middle bed is dark blackish grey (S. 12). The upper shale bed (S. 13) is pale yellow with occasional presence of anhydrite a product of oxidation of the pyrite and black cubes of goethite pseudomorph after pyrite. The shale beds of the Duwi Formation at um Salamah open-pit mine is generally compact. The lower shale bed (S. 16, 17) is dark blackish grey with black cubes of goethite pseudomorph after pyrite with yellow rim (S. 17).The upper shale bed (S. 18, 19) is yellowish grey.

At Kom-mir the color of the shale beds of the Dakhla Formation varies from dark blackish grey in the lower bed (S. 9) to light grey in the upper one (S. 10).

At El-Deir the color of the lower part (S. 31, 32) is yellowish red to pale yellowish red while it is yellowish dark grey to pale grey in the upper part (S. 33, 34).

The lithofacies, the fossil content and the spatial distribution of the Duwi Formation indicated according to Issawi, (1999 and 2009) a littoral environment of deposition probably oscillating with inner to outer sublittoral conditions resulting into the various types of lithotopes ranging from phosphate, carbonate to shale beds.

These variations in color from base to top of each succession confirm the oscillating depositional environment from littoral inner to outer sublittoral conditions with time and spatially gets deeper on going from the west till the extreme eastern part of Nile Valley district. The depositional environment characterized by variable pH values (from 5.5 to > 7) and Eh values (from < -0.1 to > -0.3 volts).

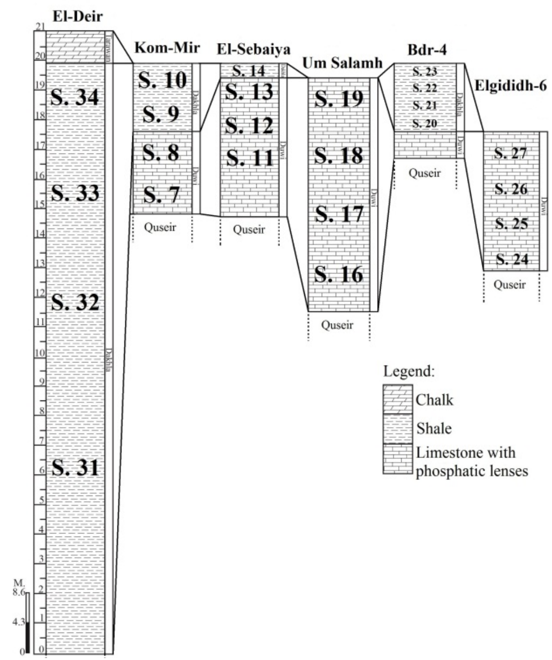

Fig. 6: Lithostratigraphic Correlation of the six studied sections, Nile Valley District.

The variation in color of the shale beds of the Duwi Formation East of the Nile Valley from yellowish grey at the base to pale yellow (with goethite cubes pseudomorph after pyrite) at the top via; dark blackish grey confirm the oscillating depositional environment from littoral to inner to outer sublittoral conditions. However, the color of the shale beds of the Duwi Formation West of the Nile Valley varies from yellowish dark grey to yellowish red pale grey from base to top mostly due to oxidation of fine-grained pyrite which formed hematite and limonite iron oxides and the black goethite cubes.

Regarding to physico-chemical characteristics prevailed during deposition of the Duwi Formation; the environment can be dedicated from the present day black mud data given by Krauskopf, (1979) where the pH is ranging from 5.5 to 7. He mentioned that below the surface of the black mud, where the decomposition of organic material has gone on longer, the pH tends to rise and the Eh to become

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more reducing. In this respect sulfur from original protein of died organisms is largely converted to hydrogen sulfide. Some of the sulfur combines with iron to form iron sulfide, perhaps first as fine-grained, black hydrotroilite (FeS.nH2O), but this compound changes quickly to the more stable pyrite. This explains the recorded goethite cubes with yellowish rim pseudomorph after pyrite (Fig. 7) and the associated anhydrite formed by combination of the liberated SO3 – by the oxidation of pyrite to goethite as well as hematite and limonite stainings.

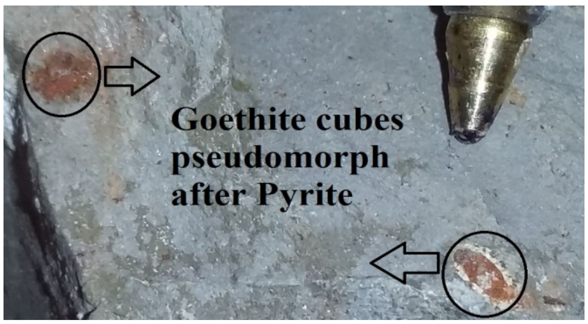

Fig. 7: Goethite cube with yellowish rim in shale..

4.3 The District of the Red Sea Coastal zone

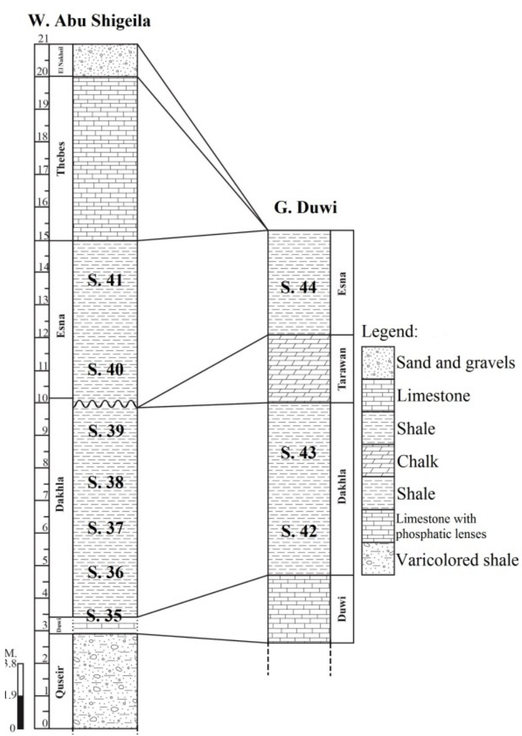

Fig. 8: Correlation chart of the two lithostratigraphic succession of the studied sections, Red Sea Coastal zone.

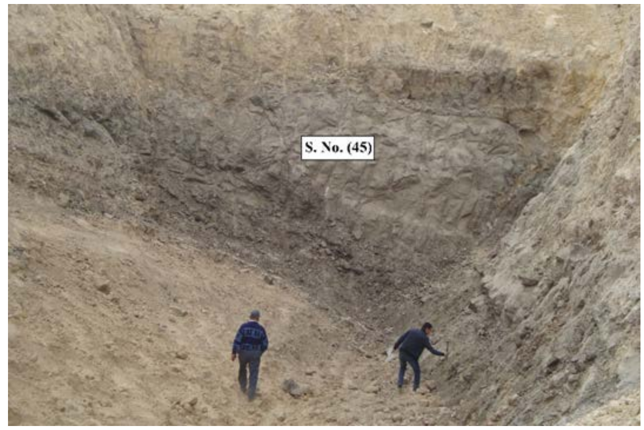

Fig. 9: Photograph showing Fresh black shale bed within the Duwi Formation, Gasus open-pit phosphate mine.

Fig. 10: Photograph showing blocks of the black shale, El Nakheil mine.

The Dakhla shale at the sampled sections: Gebel Gifata, Badr-4, El-Deir, Abu-Shigeila and G. Duwi are generally characterized by disseminated red to black cubes of goethite pseudomorph after pyrite.

The Dakhla Shale was deposited under weak short-lived current quite shallow neritic to inner neritic zone of Folk (1962, 1968) of marine environment. The outer neritic marine environment prevailed at interval of time of deposition of the Dakhla shale at Gebel Duwi was warm reducing water during deposition. The O. M. content recorded in the Dakhla shale beds at G. Duwi by Mohammed *et al*. (1992) is 4.66 %. Its presence in association with the calcite and anhydrite favor deposition in very shallow warm, reducing marine environment supplied by local accidents of argillaceous sedimentation incorporated with lenses and pockets of un-decayed organic matter existed in the accumulating muds and silts. The recorded mudstone bands by Issawi *et al*, (1999 and 2009) and the studied sections of the present study (Fig. 4) point to deposition within oscillating not constantly

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advancing but phases of retreat within the transgressive sea episode during the Late Maastrichtian- Early Paleocene.

The recorded goethite cubes pseudomorph after pyrite confirm prevalence of environment of redox potential $Eh < -0.3$ volts as the decomposition of the organic matter below the surface of the deposited mudstone tends the Eh to become more reducing. However the topmost beds of the Upper Argillaceous Limestone Member of the Esna Formation at Gebel Duwi are devoid of organic matter as mentioned by Mohammed *et al*, (1992).

The geochemical distribution of the chemical components in the Esna Shale reveal deposition of the carbonate beds at Gebel Duwi in shallower and warmer marine environment (high) in comparison with that prevailed during deposition of the Wasif Esna Shale. The distribution of the chemical components Viz; high MgO, Cl^- and SO_3^{--} percentages favors prevalence of very shallow, warm and evaporational marine environment at interval of time. Moreover, the presence of organic matter associated with calcite, evaporates and phosphate favors deposition of the Esna Shales at Gebel Duwi in reducing marine environment supplied by argillaceous material.

Mudstone bands recorded in the Maastrichtian part of the Duwi Formation in some studied mines east of the Nile Valley.as well as; the overlaying lower part of the Dakhla Formation of Late-Maastrichtian mostly shale with mudstone bands in the east support the seeming conformity mentioned by Issawi *et al*. (1999, and 2009) between both formations. The reefal limestone near the top of the Dakhla Formation indicating shallower conditions appearing the unconformity at the top of the Maastrichtian section points to the transition to the Lower Paleocene part of the formation.

5. Organic Matter Distribution

The results of L.O.I ignited at 375° C for 2 hours representing the amount of O.M. in the collected samples from the fifteen sites representing three districts recorded in (Table 1) point to the following:

In the Upper Calcareous Shale Member of the Esna Formation in the Red Sea Coastal Zone the O. M. contents representing ≈ 2.5 wt. % in average.

The Dakhla Formation in the Western Desert samples Low O. M. values (≈ 2 wt. %) except sample no. (4.23 wt. %) are recorded. While in the Nile Valley and the Red Sea Coastal Zone: O.M. ≈ 1.5 wt. % and ≈ 1.6 wt. %. In average respectively.

In the Duwi Formation Nile Valley; the O. M. is ranging from 1.92 – 0.3 wt. %.

These low O. M. percentages in the three formations of the three districts could be considered exploratory criteria pointing to that the studied shales of the three formations are organic matter bearing. Regarding to Sarah's Method of organic matter determination the samples ignited for 12 and 16 hours followed by weighing after each ignition time.
The O. M. values in wt. % after 12 and 16 hours recorded the results given in (Table 1) compared to 2 hours ignition recorded increase several times where the range of the ratio of both values for are extraordinary high as follows:

Fm.		12 h. /2 h.	16 h. /2 h.
Esna		1.33-1.11	1.33-1.18
Dakhla	(Gifata)	7 -1.37	7.01-1.37
	(Red Sea)	12.77-4.02	12.90-4.03
Duwi	(Nile Valley)	31.77-1.04	45.25-0.55
	(Red Sea)	7.57-1.06	7.57-1.08

Moreover, the recorded O. M. values on ignition after 16 hours compared to that after 12 hours are approximately matching. Consequently the ignition for 16 hours with gradual increase of temperature with time is to be the recommended method for determination of total organic matter, however the obtained results of ignition for 2 hours favor its use for exploration for probable presence of O. M. in shale.

Summing up the sampled shales of the Duwi Formation, Dakhla and Lower Calcareous Shale Member of the Esna formations in the three districts: Western Desert, Nile Valley and Red Sea Coastal Zone are bearing promising organic matter percentages. These values favored estimation of the total organic carbon content to evaluate the sampled shales as source for energy.

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Table 1: The Organic Matter (O. M.) % after 2,12and 16 hs.

Locality	Fm.	S. No.	O. M. wt. % after (2) hs.	O. M. wt. % after (12) hs.	O. M. wt. % after (16) hs.
a.) Western Desert district					
Gebel Gifata	Dakhla	1	2.201	11.632	11.632
		2	2.538	3.485	3.485
		3	2.543	8.576	8.576
		4	2.084	14.591	14.609
Abu - Tartur		5	4.232	4.303	4.578
		6	1.925	13.034	13.080
b.) The Nile Valley district					
Kom-Mir (West)	Duwi	7	0.301	0.658	0.728
		8	0.778	3.030	3.059
	Dakhla	9	1.513	3.192	3.202
		10	1.062	1.353	1.602
El-Sebaiya (East)	Duwi	11	0.925	2.159	2.459
		12	0.994	5.142	5.291
		13	0.702	22.301	22.326
	Dakhla	14	1.917	2.005	2.047
		15	1.812	7.750	7.899
Um Salamah (East)	Duwi	16	0.223	0.443	0.443
		17	1.718	3.320	3.415
		18	1.41	5.114	5.412
		19	1.267	6.603	6.715
Badr-4 (East)	Dakhla	20	1.503	9.684	9.790
		21	1.021	5.751	5.947
		22	1.532	1.909	2.108
		23	1.403	11.265	11.265
Elgididh-6	Duwi	24	1.341	4.835	4.866
		25	0.835	1.051	1.051
		26	0.574	2.118	2.169
		27	1.301	12.903	12.903
Oweina (East)	Duwi	28	1.542	1.834	2.087
		29	1.209	3.319	3.450
		30	0.947	6.019	6.107
Eldeir (West)	Dakhla	31	1.345	13.486	13.492
		32	1.705	1.966	2.122
		33	0.582	10.397	10.397
		34	1.296	7.461	7.461

Table 1. cont…

c.)-The Red Sea Coastal zone					
W. Abu Shigeila	Lower part of Dakhla	35	2.304	2.425	2.425
	Dakhla	36	1.311	16.748	16.907
		37	1.067	13.588	13.588
		38	1.792	13.928	13.928
		39	1.992	8.005	8.024
	Esna	40	3.031	3.538	4.037
		41	10.93	11.962	12.418
G. Duwi	Dakhla	42	1.396	1.473	1.611
		43	0.424	8.938	8.938
	Esna	44	1.920	2.134	2.273
W. Gasus	Duwi	45	8.555	9.879	10.135
El Nakheil		46	20.130	27.993	28.083
Elkweeh		47	1.709	12.929	12.929
Zug ElBahar		48	7.494	7.930	8.121

The determined total organic carbon (TOC) of twenty representative shale samples containing the highest O.M. % are given in table (2).

Table 2: The total Organic carbon content in (20) shale samples

S. No.	TS wt.%	TC wt.%	TOC wt.%	Petroleum potential
1	0.31	6.1	3.3	Very good
4	0.26	3.96	2.2	Very good
5	0.34	1.04	0.82	Fair
6	0.09	2.9	2.1	Very good
7	0.39	0.6	0.6	Fair
9	0.08	0.66	0.66	Fair
15	0.30	1.3	1.3	Good
17	1.55	1.23	1.05	Good
23	2.37	1.7	1.7	Good
25	0.36	1.1	1.1	Good
28	0.88	0.23	0.23	Poor
31	0.004	3.63	1.5	Good
36	0.01	4.40	1.8	Good
38	1.42	5.11	1.5	Good
40	0.21	0.11	1.1	Good
41	0.01	4.80	1.4	Good
43	0.01	5.69	2.8	Very good
45	1.97	21.65	20.69	Excellent
46	2.76	22.76	22.15	Excellent
48	0.07	6.3	2.2	Very good
Petroleum potential: Poor (0-0.5 TOC Wt. %) - Fair (0.5-1 TOC Wt. %)- Good (1-2 TOC Wt. %)- Very good (2-4 TOC Wt.)- Excellent (>4 TOC Wt. %)				

According to the Petroleum potential (quantity) of an immature source rock of Peters and Cassa (1994, Table 2), there are seven samples exhibiting Fair to Excellent

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Petroleum potential which can be considered promising oil shales. The graphic presentation of O. M. and TOC content in shown in Figs. (11:22).

The determined total sulfur generally ranges between 0.9-0.004 in fifteen samples except five samples exhibiting high sulfur content 2.8-1.4 (Table 2). Of these (3) samples (No. 17, 45 and 46) represent Duwi Formation are characterized by notable amount of goethite cubes pseudomorph after pyrite and (2) samples (No. 23 and 38) representing Dakhla Formation.

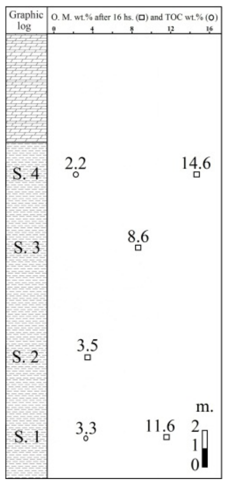

Fig. 11: Gebel Gifata, Western Desert District.

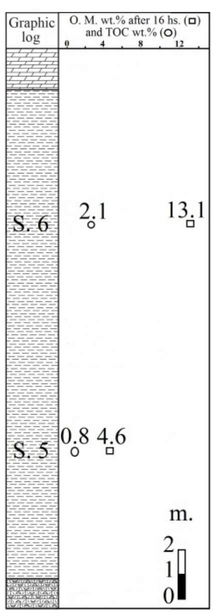

Fig. 12: Abu-Tartur, Western Desert District.

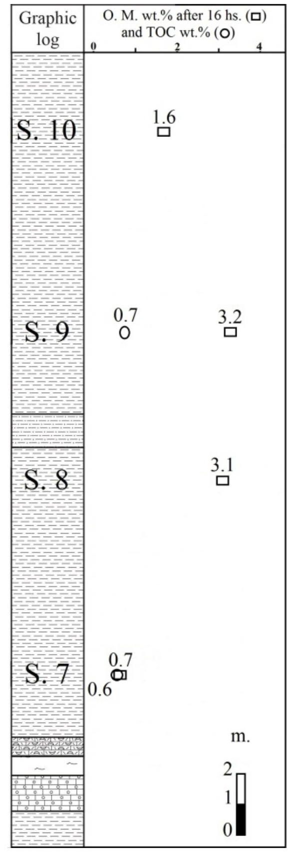

Fig. 13: Kom-Mir, Nile Valley District.

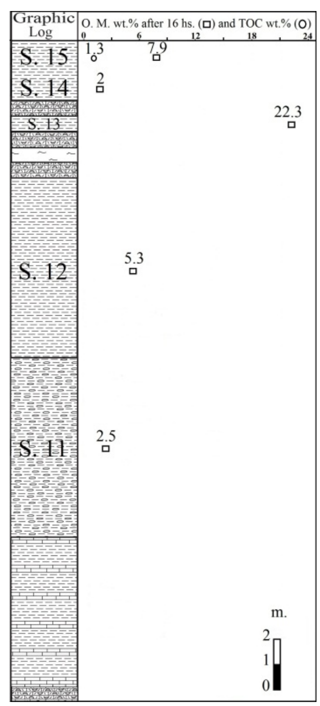

Fig. 14: El-Sebaiya, Nile Valley District.

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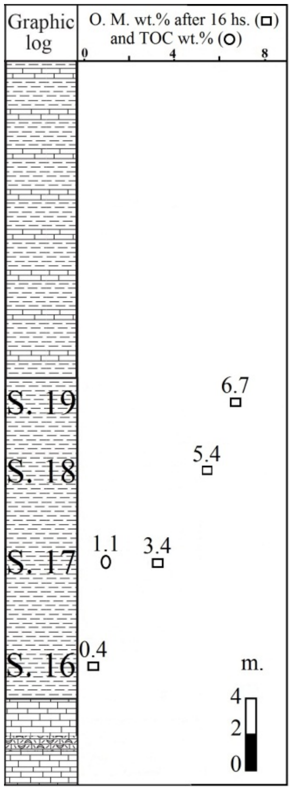

Fig. 15: Um Salamh, Nile Valley district.

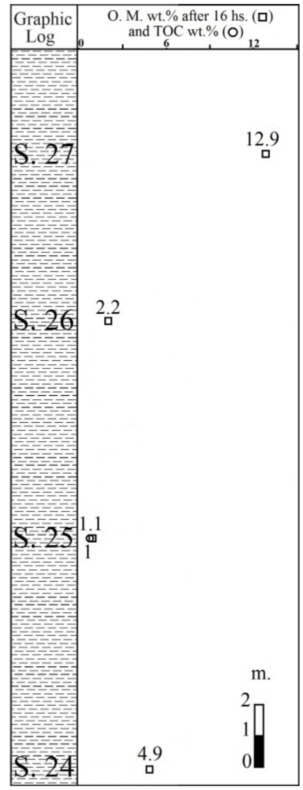

Fig. 17: Elgidida-6, Nile Valley district.

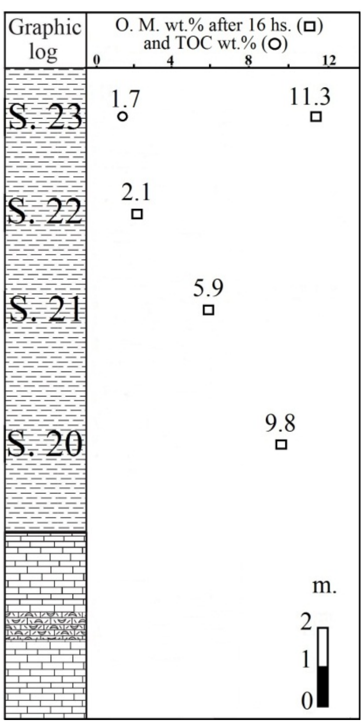

Fig. 16: Badr-4, Nile Valley District.

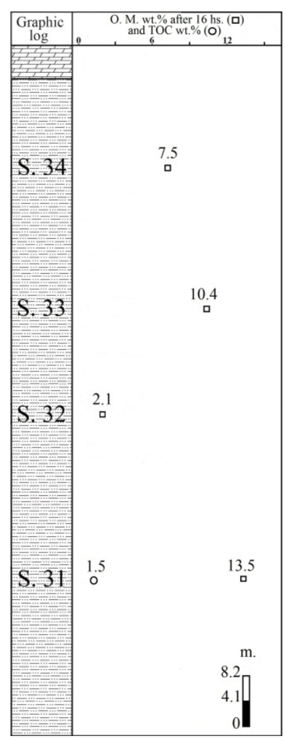

Fig. 18: Eldeir, Nile Valley District.

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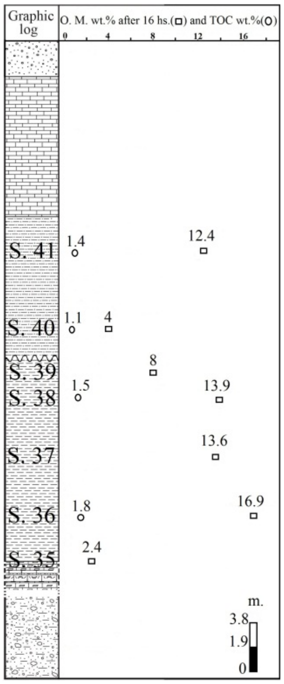

Fig. 19: W. Abu Shigeila, Red Sea Coastal zone.

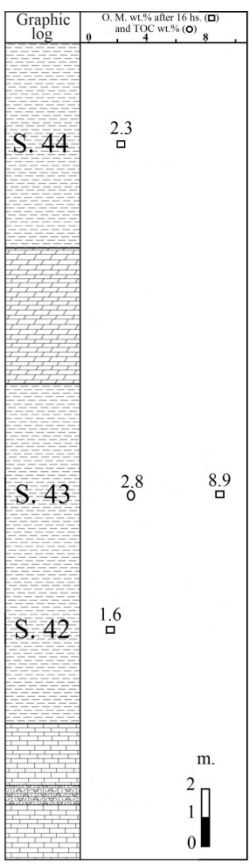

Fig. 20: W. Gebel Duwi, Red Sea Coastal zone.

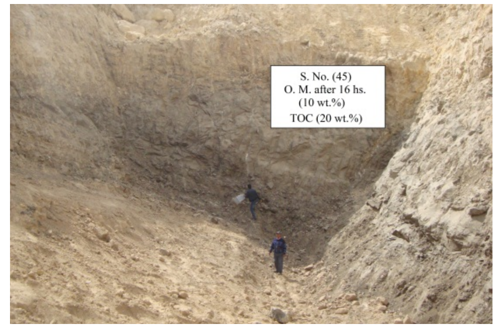

Fig. 21: W. Gasus, Red Sea Coastal zone.

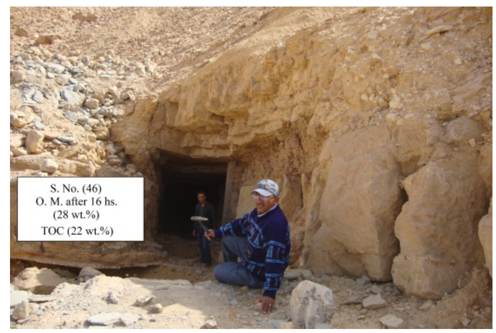

Fig. 22: El Nakheil, Red Sea Coastal zone.

4. Conclusions

The black shale of Duwi, Dakhla formations and the Lower Calcareous shale Member of the Esna Formation are Lithologicaly distinguished by presence of mud bands and goethite cubes pseudomorph after pyrite favoring deposition in oscillating marine environment from shallow neritic to inner neritic (littoral to sub-littoral) reducing environment supplied with muddy argillaceous sedimentation. The prevailed environment was highly reducing (Eh -0.3 volts and pH > 7) during deposition of the Campanian-Maastrichtian Duwi and the Lower part Early Paleocene Dakhla Formation. This environment change to be weakly reducing during deposition of the lower shale Member of the Esna Formation of Paleocene age as no goethite cubes are recorded.

The physico-chemical conditions prevailed during deposition of these black shale formations controlled distribution of O. M. from a formation as well as to another. The recorded organic matter in Duwi Formation is ranging from 22.33 wt. % (El Sebaiya) to 0.73 wt. % (Kom-Mir) in the Nile Valley, 28.08 wt. % (El Nakheil) to 8.12 (Zug El Bahar) in the Red Sea Coastal Zone, in the Dakhla Formation 13.49 wt. % (Eldeir) to 1.60 wt. % (Kom-Mir) in the Nile Valley, 16.91 wt. % (W. Abu Shigeila) 1.61 wt. % (G. Duwi) in the Red Sea Coastal Zone, 14.61 wt. % to 3.49 wt. % (G. Gifata) in the Nile Valley and from 12.42 wt. % (W. Abu Shigeila) to 2.27 wt. % (G. Duwi) in the Red Sea Coastal Zone. The total organic carbon content ranginges from 22.15-0.22 wt. % in the Duwi Formation, 0.82-0.13 in the Dakhla Formation and 0.14-0.11 in the Esna Calcareous Shale. The determined Total Organic Carbon contents in twenty samples are potentially suitable for petroleum: (Excellent-2 samples, very good-5 samples, good-9 samples, fair-3 samples, poor 1).

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