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Full Length Article

Upper Cretaceous Duwi Formation shale's oil potentiality Safaga-Quseir, Red Sea, Egypt

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ABSTRACT

Duwi Formation shale deposits at Safaga – Quseir area were assessed by rock eval pyrolysis for its shale oil potentiality. The studied shale's organic matter are mainly of type I, type II, mixed type I/II in addition to some of type III. The studied shale is described as very good source rock with total organic carbon (TOC > 2 wt%). The kerogen maturation in the studied samples is immature according to PI and Tmax. Extracted shale oil from the studied samples as bitumen was fractionated by liquid chromatography (LG) into saturated hydrocarbon, aromatic hydrocarbon, resin and asphaltene. The saturated hydrocarbon was described by gas chromatography (GC), the pristane/phytane, isoprenoids/n-alkane and CPI ratios show that, samples were deposited in marine environment with some input of terrestrial environment and are characterized by immature level.

The X-Ray Diffraction (XRD) analysis for the studied shale samples show their bulk minerals are mainly quartz, calcite, dolomite, gypsum, hematite and pyrite in addition to apatite and fluorapatite. While their clay minerals are mainly smectite and kaolinite. The chemical composition of the studied samples showed high concentration of trace elements such as Co, Cr, Cu, Ni, V and Zn which in turn through more lights on the depositional environment of the organic matter.

1. Introduction

Safaga – Quseir is important area along the Red Sea Coast in Egypt. Its importance was gained since phosphate ore were mined from the Gabel Duwi Range. The study area extends in a northwest direction along the western coast of the Red Sea from Quseir to Safaga, between Latitude 25° 50′ and 26° 67′ N and Longitude 33° 45′ and 34° 25′ E, covering an area of about 500 km^2 Fig. 1. The Duwi Formation is spread over several hundreds of kilometers, from the Western Desert up to the River Nile Valley, and in the Eastern Desert up to the Red Sea Coast.

Black shale is restricted with phosphate bed in the Upper Cretaceous and recorded at the upper parts of Qusier Formation, Duwi Formation and the lower part of Dakhla Shale. Black shale has been deposited in Quseir- Safaga land stretch and shoreline of the Tethys during Upper Cretaceous.

The main objectives of this study are characterizing and evaluating the black shale of Duwi Formation. The evaluation includes their organic matter content, its type, maturity, depositional environment and their potential to generate unconventional shale oil and shale gas hydrocarbon.

2. Geologic setting

The Red Sea is initiated in the Late Oligocene–Early Miocene as relatively young and active continental rift system [1,2]. It is resulting from the northeast separation of the Arabian plate away from the African plate, with part of the plate movement taken up by opening of the Gulf of Suez rift during the Late Oligocene-Early Miocene [3]. Part of the extension was taken by the sinistral strike-slip motion along the Aqaba-Dead Sea transform during Mid-Miocene– Quaternary time [4,5].

In the Safaga-Quseir area, rotated extensional fault blocks had exposed pre- and syn-rift strata together with large areas of the Precambrian basement which form the Red Sea Hills on the rift margins. The stratigraphic units of the Egyptian Red Sea at the area between Safaga and Quseir consist of the following rock units from base to top [6] Fig. 2:

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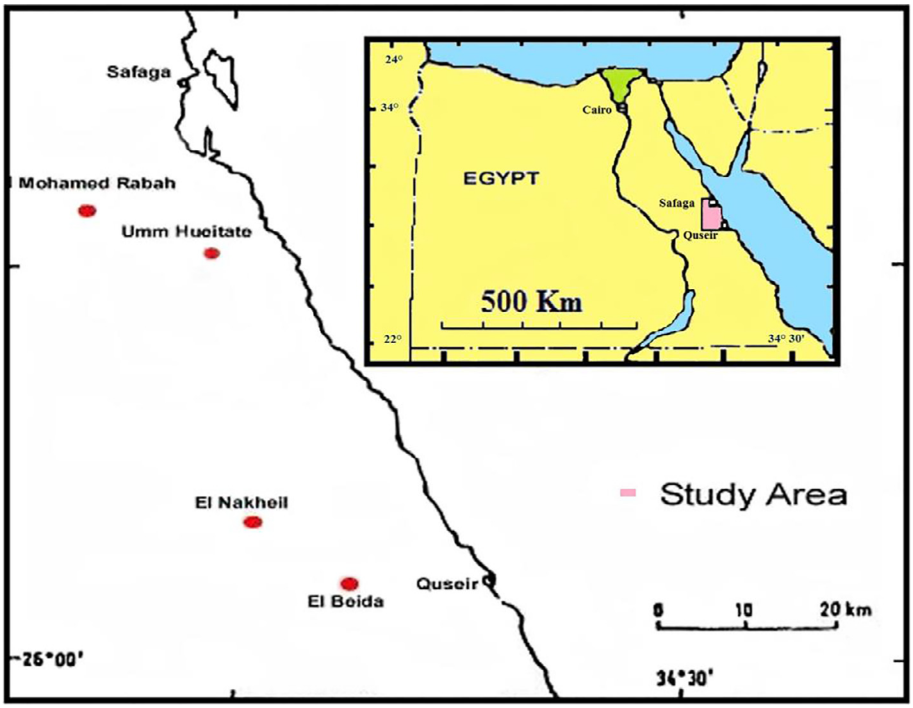

Fig. 1. Location map of the study Area.

1. Precambrian crystalline basement (metavolcanics, meta-sediments and granitoid intrusive.
2. Mesozoic-Cenozoic pre-rift sediments.
3. Late Oligocene-Miocene to Recent syn-rift sediments.

The basement contains strong fabrics (faults, fractures, shear zones and dykes) oriented WNW, NNW, NS and ENE. The basement is unconformably overlain by a 500–700 m thick section of pre-rift strata that ranges in age from the Late Cretaceous to the Middle Eocene. The lower part of the pre-rift section is the 130 m massive-thick bedded, siliciclastic Nubia Formation [7]. This is overlain by a 220–370 m thick sequence of interbedded shales, sandstones and limestones of the Quseir, Duwi, Dakhla and Esna Formations [8,9]. The uppermost pre-rift strata consist of 130–200 m of competent, thick bedded limestones and cherty limestones of the Lower to Middle Eocene Thebes Formation.

The Late Oligocene to Recent syn-rift strata unconformably overlie the Thebes Formation and vary in thickness from less than 100 m onshore to as much as 5 km in offshore basins [10]. The lowermost syn-rift strata are dominantly coarse-grained clastics (Nakheil and Ranga Formations). These clastics are overlained by reefal limestones, clastics and evaporates (Um Mahra, Sayateen and Abu Dabbab Formations). Late Miocene carbonates and reefs; and the Pliocene to Recent syn-rift clastics overlie the evaporites in the coastal outcrops [11].

3. Methods

Twelve oil shale samples were collected from Duwi Formation from four mines (Mohamed Rabah, Um Huetate, El Nakheil and El Beida phosphate mines) at Safaga–Quseir area, Red Sea Coast, Egypt. The weathering impressions on the samples were brushed off, deried at 60oc and powdered to less than 63 μm using sieving analysis. The samples were analyzed by Rock-Eval pyrolysis which was described by [12], as a rapid method for characterization of kerogen types and determination of its maturity.

Shale samples were analyzed by soxhlet apparatus for bitumen extraction. The extracted bitumen is fractionated and separate to their constituents as asphaltene, saturates, aromatic and resins. This is done through a column liquid chromatography using n-heptane, n-hexane, benzene and chloroform respectively. The saturated fraction is isolated from bitumen then they are analyzed with avarian model 3400 gas chromatography (GC) instrument fitted with a quardex 50 m fused silica capillary column. The GC is programmed from 40 °C to 340 °C at 10 °C/ minute with 2 min hold time at 40 °C and 20 min hold time at 340 °C.

The samples were examined for the bulk and clay minerals using Philips X-ray diffraction PW 9901 of copper anode tube.

The chemical composition of trace elements is measured by using Flame atomic absorption instrument (Spectrometer-ZEE nit 700P-analytic jena-Germany). Disintegrate sample to 100–200 mesh then take 0.5 g of the sample in Teflon then add to it 5 ml H2O2 + 10–25 ml HF then put it on hot plate until dry then add 25 ml HCl (1:1) then put on hot plate and steer it until formation a precipitate then take the solution in measuring flask and add distilled water to known volume.

4. Results and discussions

4.1. Organic richness

The organic carbon richness of the oil shale (fine grained sedimentary rock containing organic matter rich in hydrogen, known as kerogen [14]) samples is expressed by the weight percent of TOC, which give fast picture in the evaluation of sediments as a source for hydrocarbons production and generation.

[15] presented a scale for assessment of source rocks used in a wide scale and is applied in this work; a content of < 0.5 wt% TOC as a poor source, 0.5–1.0 wt% as a fair source, 1.0–2.0 wt% as a good source and more than 2.0 wt% TOC as very good source rock. Based on the organic carbon content sediment can classify to muddy rocks containing TOC less than 1 wt%, black shale has TOC up to 20 wt%, and oil shale contain TOC ranged from 20 wt% to 50 wt% [13].

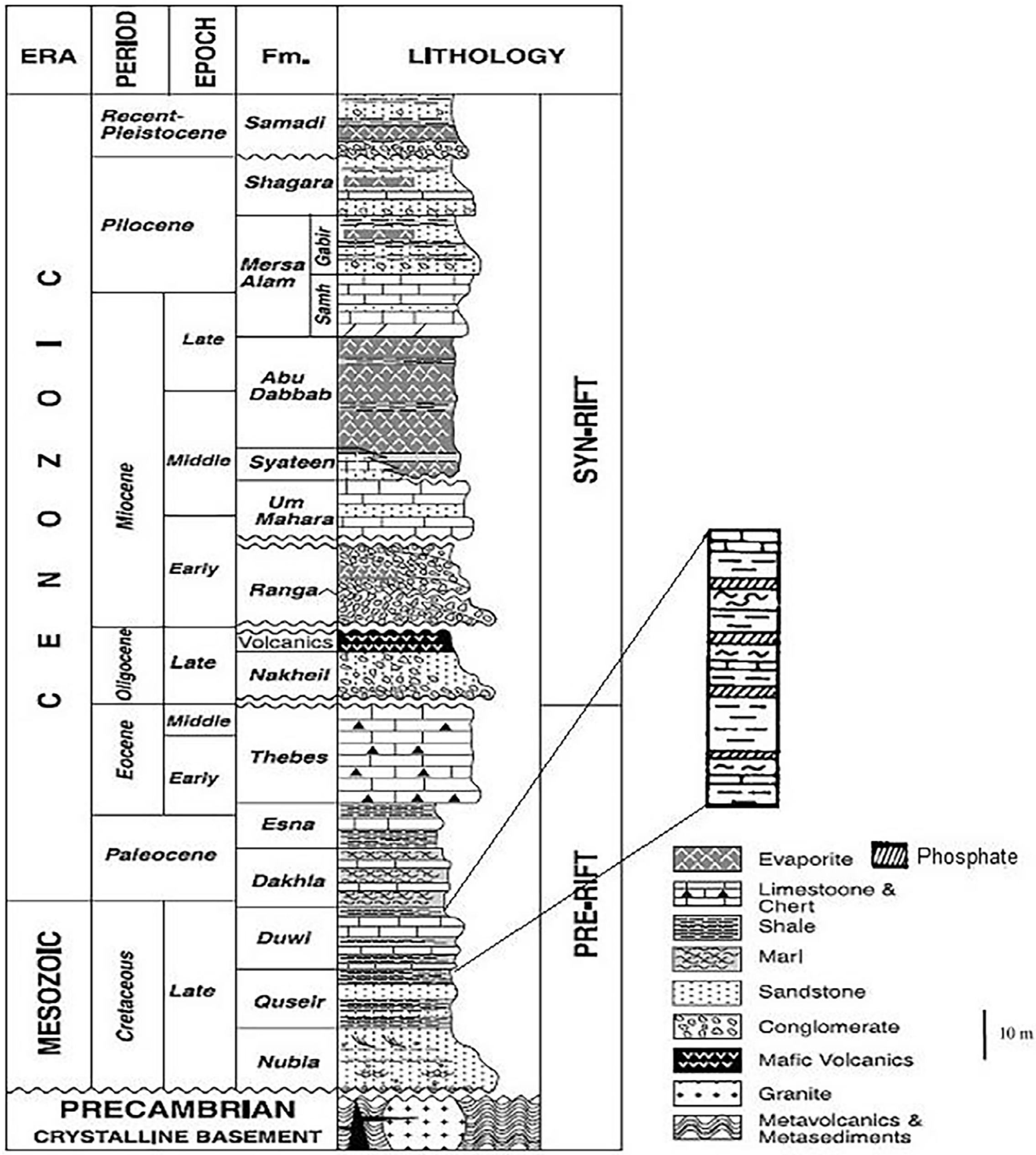

Fig. 2. Summary of the stratigraphic sections of the northwestern part of Red Sea coast, (Khalil et al., 2009).

As shown in Table 1 and according to [13,15] TOC values of Umm Huetat mine samples range between 2.6 wt% and 5.18 wt% with average TOC of 3.6 wt%, indicating very good source rock and called black shale. In El Nakheil mine samples, TOC values range between 19.18 and 22.23 wt% with an average TOC of 21.12 wt%, which considered very good source rock and called oil

Table 1
Rock Eval pyrolysis data of the analyzed samples from the studied mines.

Mine Name	Sample Number	TOC	Average TOC	Shale Type [13]	S1	S2	S3	Tmax	PI	HI	OI	Kerogen Type QI	Production Yield S1 + S2
Um Huetat	1	3.02	3.6	Black Shale	0.23	12.93	1.17	409	0.02	428	39	11.05	13.16
	2	2.6			0.32	11.63	1.6	427	0.03	447	62	7.27	11.95
	3	5.18			0.39	24.37	2.73	412	0.02	470	53	8.93	24.76
Mohamed Rabah	4	1.77	2.88	Black Shale	0.14	1.66	1.68	421	0.08	94	95	0.99	1.8
	5	0.04		Muddy Rock	–	–	–	–	–	–	–	–	–
	6	9.66		Black Shale	1.3	58.25	4.04	416	0.02	603	42	14.42	59.55
	7	0.05		Muddy Rock	–	–	–	–	–	–	–	–	–
El Beida	8	11.61	11.53	Black Shale	1.97	65.61	7.1	419	0.03	565	61	9.24	67.58
	9	11.45			2.01	6481	7.75	418	0.03	566	68	8.36	66.82
El Nakheil	10	19.18	21.12	Oil Shale	4.18	130.2	8.17	418	0.03	679	43	15.94	134.4
	11	21.94			4.05	151.9	6.79	418	0.03	693	31	22.38	156.1
	12	22.23			4.5	159.2	6.54	418	0.03	716	29	24.35	163.7

shale. In El Beida mine samples TOC values range between 11.45 wt% and 11.61 wt% with average TOC of 11.53 wt% it is mainly very good source rock and called black shale. At Mohamad Rabah mine TOC values range between 0.04 wt% and 9.66 wt% with average TOC of 2.88 wt%, indicating poor to very good source rock and considered black shale and muddy rock.

4.2. Organic matter type

[12] Used the pyrolysis yield to differentiate between the types of organic matter by plotting the hydrogen index (HI = S2/TOC) versus the oxygen index (OI = S3/TOC) on a modified Van Krevelen diagram Fig. 5 as follows

Type I: mainly oil-prone organic matter with minor gas.

Type II: mixed oil and gas-prone organic matter.

Type III: mainly gas-prone organic matter with minor oil.

The relationship between hydrogen index (HI), versus oxygen index (OI) Fig. 3 shows that the type of organic matter at El Nakheil mine is mainly of type I (oil prone), while in Umm Hueitate and El Beida mines are mainly of type II (oil prone). In Mohamed Rabah mine the type of organic matter range from type I, II and mixed type II/III.

4.3. Organic matter maturation

[15,16] used Tmax and PI = S1/ (S1 + S2) from the Rock-Eval pyrolysis, as a good tool for the maturation of organic matter evaluation. [16] used Tmax to divide the maturity of organic matter into:

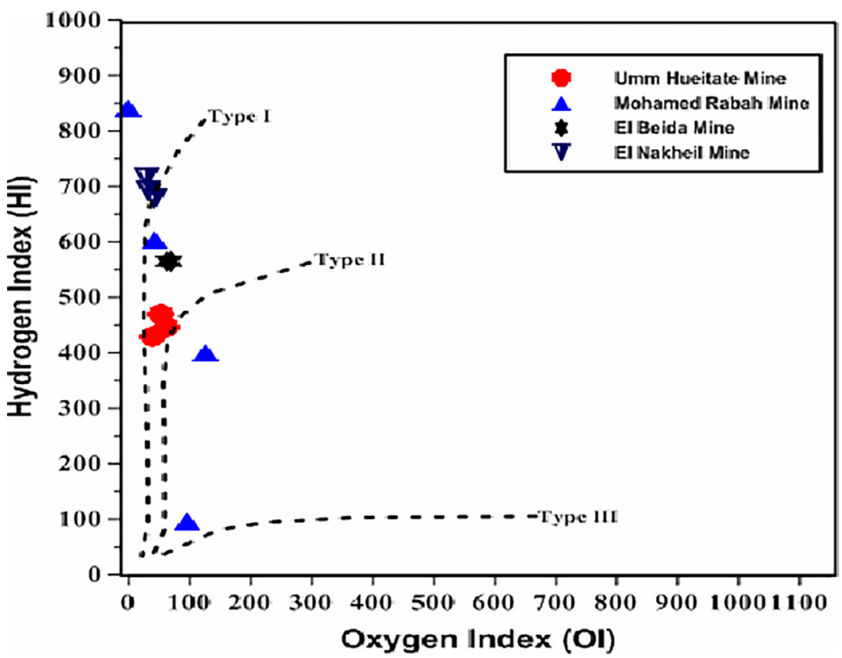

Fig. 3. Modified Van Krevelen diagram (Espitalie et al., 1977).

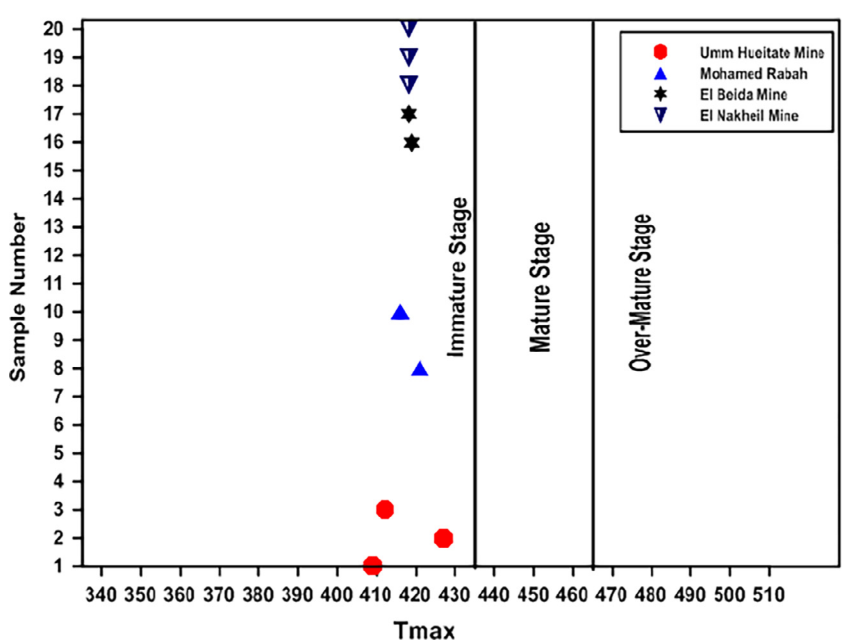

Fig. 4. Maturation of organic matters present in Duwi Formation based on rock-eval pyrolysis data as Tmax, (Peters et al, 1994).

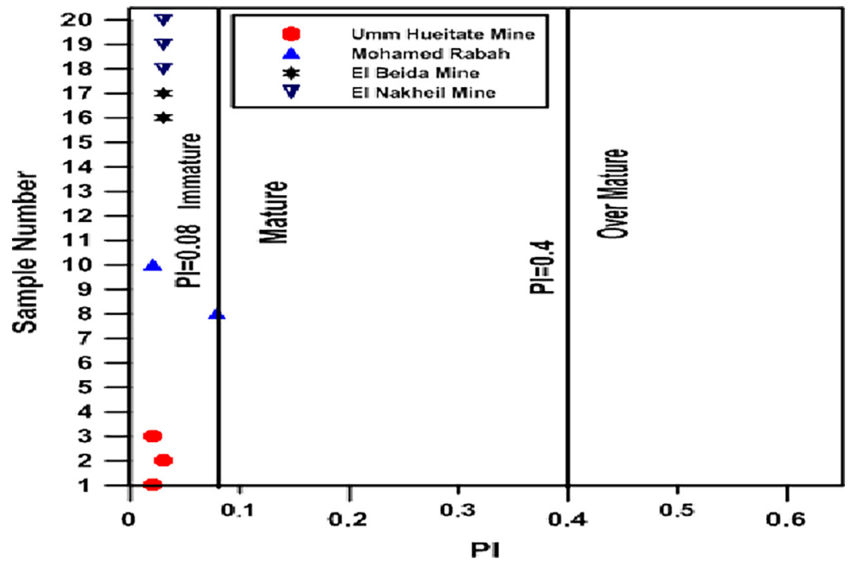

Fig. 5. Maturation of organic matter present in Duwi Formation based on rock-eval pyrolysis data as production index (PI) (Peters, 1986).

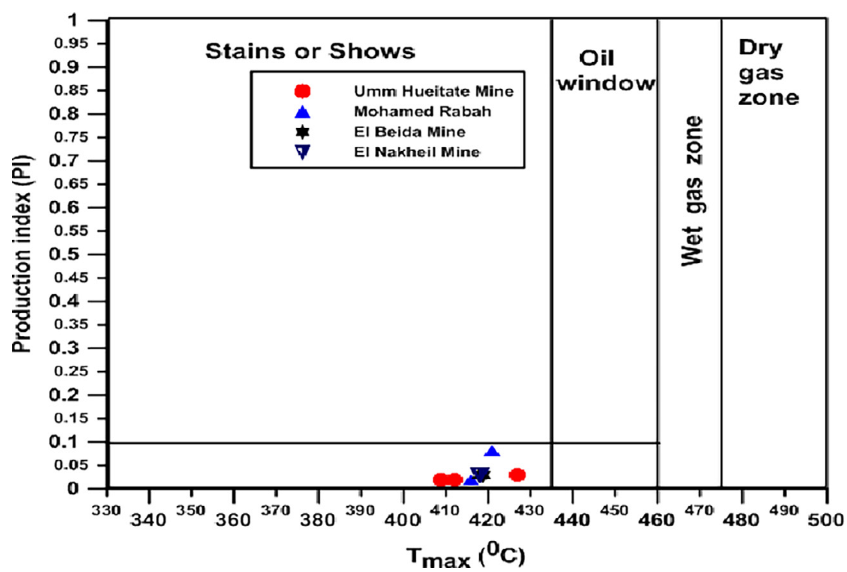

Fig. 6. Maturation potential of the studied mines based on rock-eval pyrolysis data Tmax and PI (Ghori and Haines, 2007).

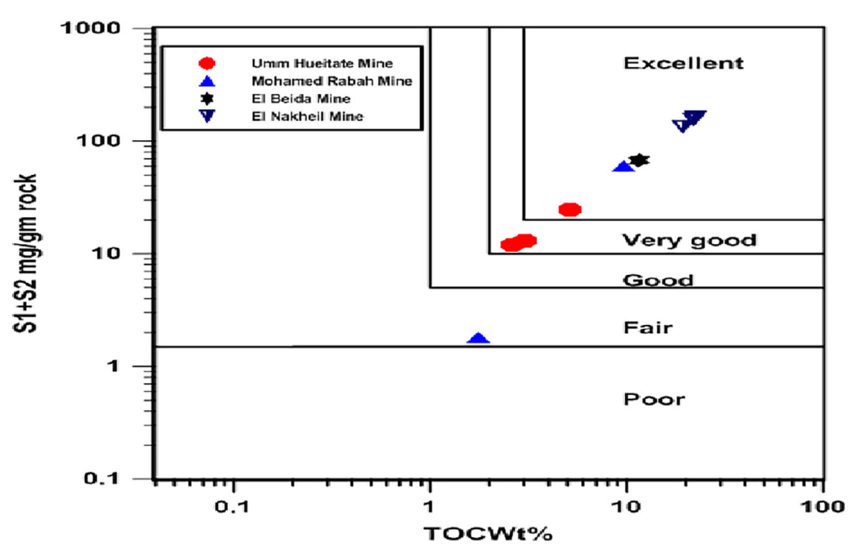

Fig. 7. Generation potential of Duwi Formation in the studied mines, (Tissot and Welte, 1984).

Tmax <435 °C indicates immature stage, Tmax between 435 and 445 °C indicate early mature stage, Tmax between 445 and 450 °C indicate peak of maturation stage, Tmax between 450 and 470 °C indicates late maturation stage, while Tmax >470 °C indicate over mature stage. According to [16], the measured Tmax of the analyzed samples from all mines occur in immature stage, (Tmax <435 °C) Fig. 4.

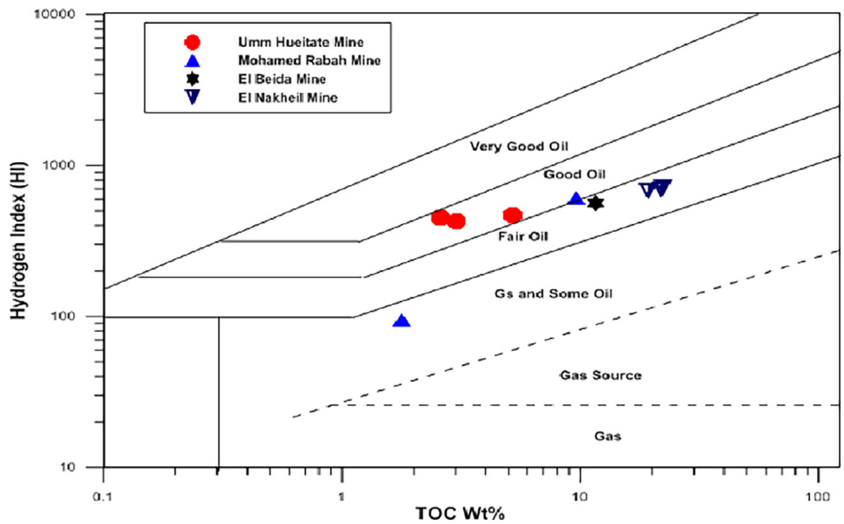

Fig. 8. -1. The relationship between organic carbon richness (TOC) and the hydrogen index (Jackson, et al., 1985).

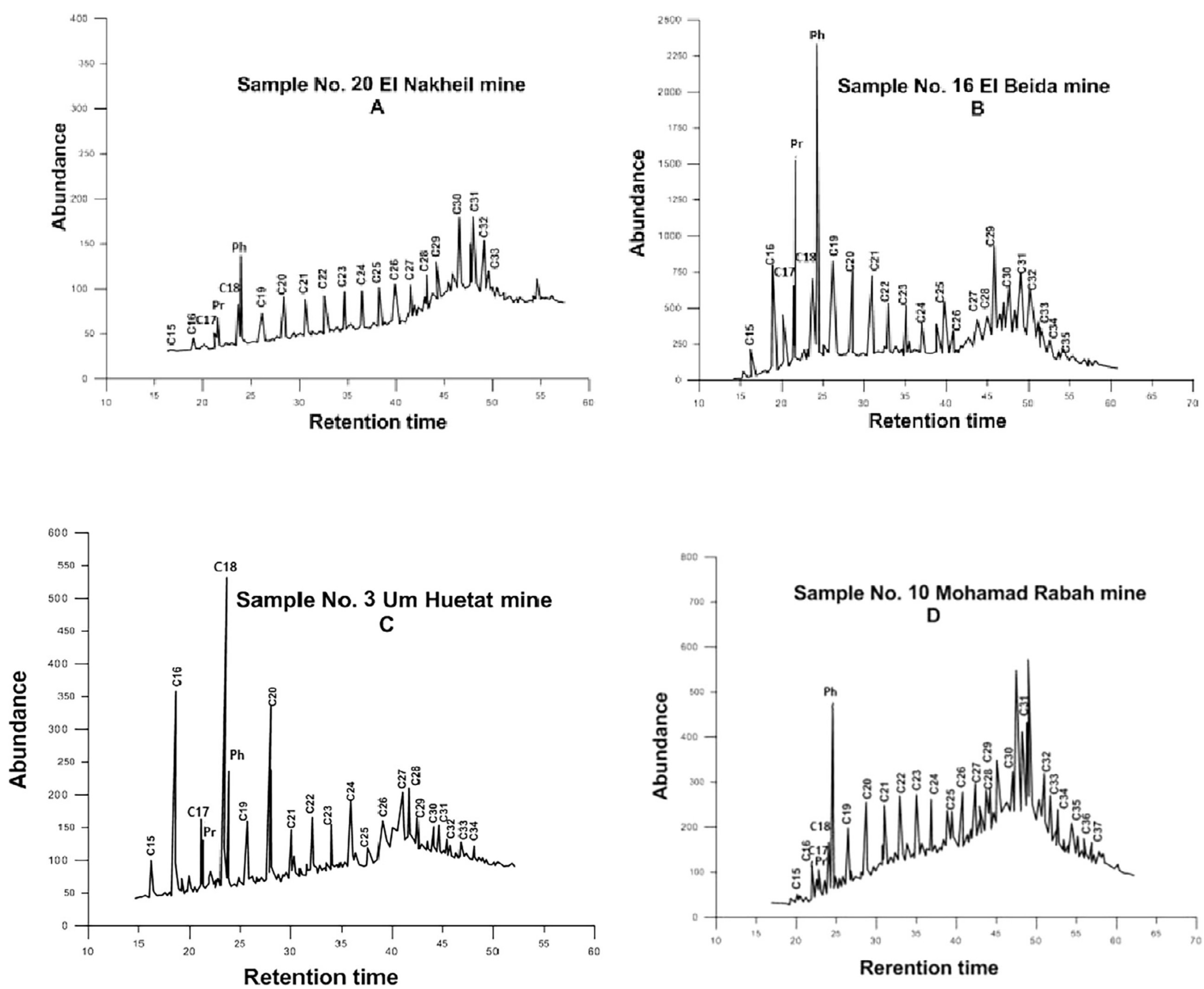

Fig. 8. -2. Gas chromatography charts of Carbon number distribution of the studied extracted bitumen samples (Peter et al., 2005).

[15], refered to PI as a maturation indicator for organic matter present in the oil shale. He refered to oil generation window (maturation stage) between 0.08 and 0.4. It is clear from Fig. 5 that, most of the analyzed samples are outside the limit of the maturation stage.

[17], constructed a relationship between the maturation parameters Tmax and production index (PI) obtained from Rock-Eval pyrolysis data to define the maturation of the existing sedimentary organic matter. We find that all the samples not reached to maturation stage as shown in Fig. 6.

4.4. Generation potential

The generation potential of oil shale is identified by the sum of S1 + S2 obtained from pyrolysis analysis. It ia also known as the generation potential (GP) [18]. According to [17,18] source rocks or oil shales with GP < 2 are poor, from 2 to 5 are fair, from 5 to 10 are good and >10 are very good generation potential. The relation between GP and TOC after [17] in Fig. 7 shows that El Nakheil and El Beida mines are excellent generation potential and in Umm Hueitate mine is varied from very good to excellent generation potential. Mohamed Rabah mine is ranged from fair to excellent generation potential.

4.5. Hydrocarbon products type index (QI).

This method is used for determining the type of hydrocarbon products (oil or/and gas) generated from a source rock or oil shale. The QI is equal S2/S3 [15,16,19].

As summarized by [19], the hydrocarbon type index (QI) is <2 for gas-prone organic matter, >5 for oil-prone and between 2 and 5 for oil/gas prone. The results of the analyzed samples from the studied mines reflected that, oil and black shale of El Nakheil, El Beida and Um Huetat mines are mainly oil-prone in which the values of S2/S3 are more than 5. While Mohamad Rabah mine the hydrocarbon products are range from oil, gas/oil and gas Table 1.

[20] Used the relationship between organic carbon richness (TOC) and the hydrogen index (HI) Fig. 8-1 to define the hydrocarbon quality. The occurrence of different types of organic matter lead to production of different hydrocarbon types of various quality ranged from good oil as in Um Huetate mine to fair oil in most of the analyzed samples obtained from the evaluated mines as El-Nakheil, El-Beida and some from Mohamad Rabah, whereas one sample from Mohamad Rabah can produce gas and some oil.

4.6. Composition of extracted hydrocarbons (Bitumen) n-alkane

The gas chromatography analysis is performed on 4 extracted samples selected from the studied mines, one from each. N-alkanes are an integral part for determining the source of the organic matter. If the n-alkane distribution is unimodal, this indicates a single source of organic matter; while a bimodal distribution of n-alkanes indicates a secondary source of organic matter [21]. The preference for odd n-alkanes comes from terrigenous input. Contamination from higher plants will show an odd n-alkane preference and an even n-alkanes preference usually implies aqueous input [21]. The gas chromatography charts of the samples from the Duwi Formation indicated that, At El-Nakheil

Table 2
Ratios derived from bitumen analysis calculating from GC.

Mine Name	Cmax	CPI	Wax%	n-C19/n-C31	Pr/Ph	Pr/nc17	Ph/nc18
El-Nakheil	C18	0.868	50.67	0.24	0.337	0.462	0.470
El-Beida	C18	0.881	25.90	3.87	0.946	0.357	0.234
Mohamed Rabah	C18	0.730	92.30	1.08	0.263	0.421	0.287
Um Huetate	C18	0.886	25.10	3.98	0.228	1.458	1.717

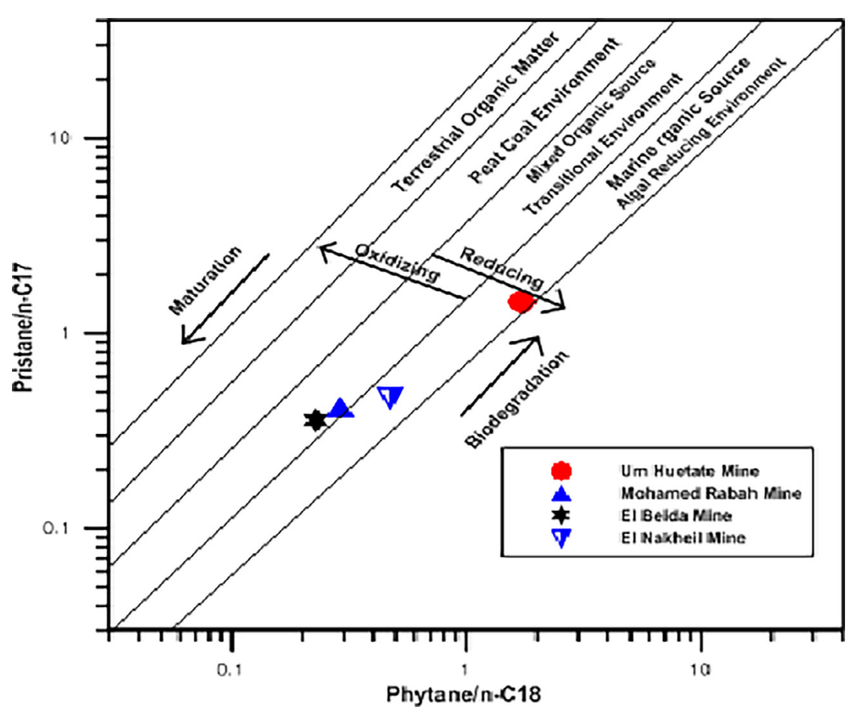

Fig. 8. -3. The relationship between Pr/n-C17 and Ph/nC18 to define type and maturation of kerogen in Duwi Formation (Shanmugam, 1985).

mine it did not produce high enough abundances of bitumen to effectively determine whether the n-alkane distribution unimodal or bimodal as shown in Fig. 8-2, but the gas chromatography chart shows humping, this reflect the biodegradation of the present hydrocarbons. The carbon number distribution (Fig. 8-2) indicates that, the extracted bitumen contain a mixture of marine organic matter in association with terrigenous input. At El-Beida mine the gas chromatography chart (Fig. 8-2) reflects that, n-alkane distribution is bimodal and there is a biodegradation lower than that present in El-Nakheil mine, this is reflected from the high intensity of the normal alkanes and it indicates also the presence of terrigenous organic matter input. Gas chromatography chart and carbon number distribution of the normal alkanes which separated from Umm Hueitate oil shale (Fig. 8-2) shows unimodal carbon number distribution toward the short chain; this indicates marine source organic matter. It also reflects that it is exposed to

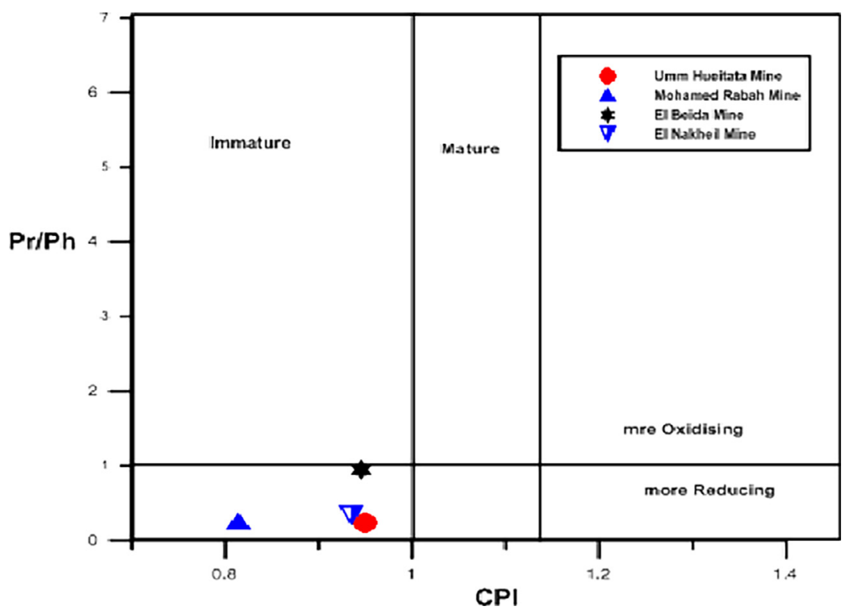

Fig. 8. -4. Pristane/Phytane ratio (Pr/Ph) versus carbon preference index (CPI) (Akinlua et al., 2007).

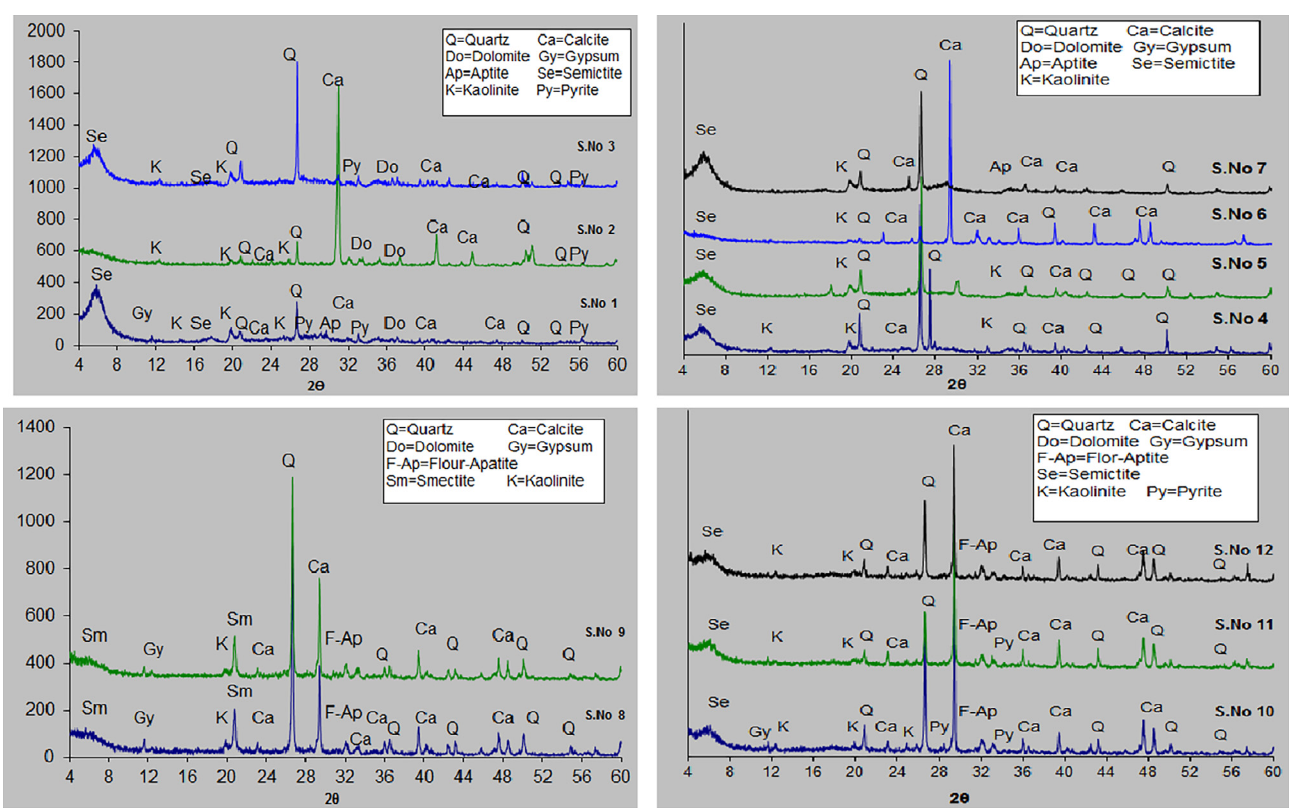

Fig. 9. XRD of bulk minerals of studied samples.

biodegradation as indicated from the humping of the chart and decrease of the intensity of the different components. At Mohamad Rabah mine the gas chromatography chart and carbon number distribution for sample No 10, show that the extracted bitumen is suffering from biodegradation as shown from humping of the chart and it is also unimodal towards the high molecular weight. These features reflected that the organic source is a mixture of marine and terrestrial organic matter.

The calculated parameters on the basis of the distributions and the abundance of the n-alkanes as well as ispronoids pristine (Pr) and phytane (Ph) of all investigated samples are presented in Table 2.

The carbon preference index (CPI) values range from 0.730 to 0.866 in the studied samples (Table 2) which indicate that samples are originated from algae and bacteria and are deposited in reducing environment [22]. In Mohamed Rabah mine, the degree of waxiness is 92.30 reflecting high waxy nature and suggestsvterrestrial origin. But at El Nakheil mine, the degree of waxiness is 50.67 indicates moderate waxy nature and suggests marine origin with terrestrial input [23,24].

4.7. Pristane and phytane

The Pr/Ph values are less than 1.0 suggest deposition in a reducing environment while Pr/Ph values more than 1.0 suggest deposition in oxidizing environment [23,24]. The Pr/Ph ratios of studied samples indicate deposition in reducing environment.

According to [25] the relationship between Pr/n C17 and Ph/c C18 (Fig. 8-3) is used to define the nature of organic matter, depositional environment and maturation. The studied samples according to this relationship are mainly marine and mixed organic matter, deposited under reducing environment and immature.

From the relationship between Pristane/Phytane ratio (Pr/Ph) versus carbon preference index (CPI) Fig. 8-4 and according to [26], it is clear that the kerogen in the studied samples are mainly immature deposited under more reducing environment.

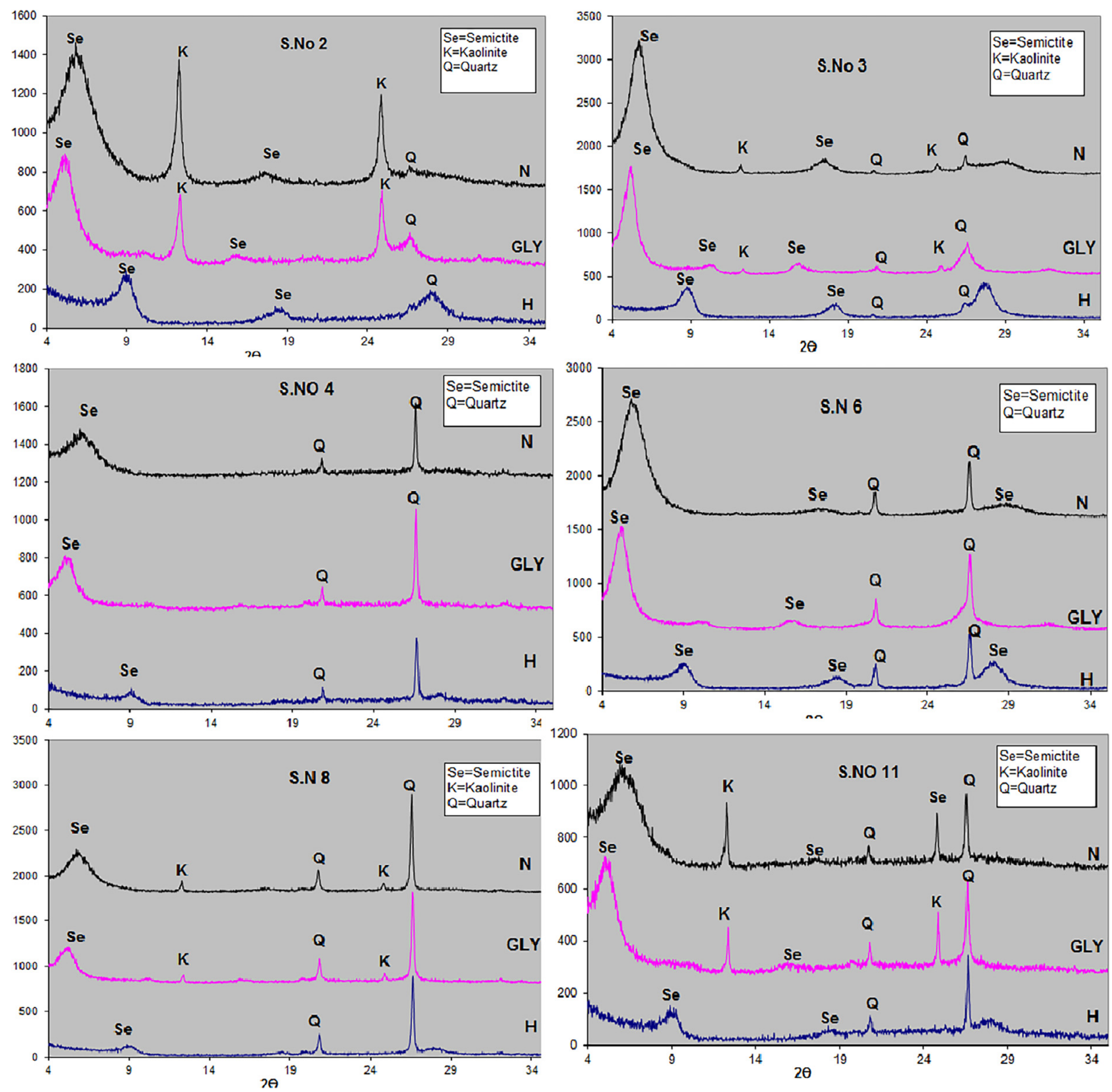

Fig. 10. XRD of clay minerals in studied samples.

Table 3
The trace elements in studied samples.

Zn ppm	V ppm	Ni ppm	Cu ppm	Cr ppm	Co ppm	Sample number	Mine name
330	240	116	20	139	129	1	Um Heuitate
235	125	117	53	210	150	2	
460	182	112	28	119	161	3	
424	330	148.5	33	190	137	6	Mohamed Rabah
850	61	180	41	235	124	8	El Beida
487	258.4	136.7	58	188	118	9	
195	360	247	97	226	133	11	El Nakheil
425.9	222.3	136.7	47.1	186.7	136	average	

4.8. Mineralogy

The bulk minerals in the studied samples composed mainly of quartz, in all the samples followed by carbonate and clay minerals Fig. 9. In some of the studied samples gypsum and pyrite were identified by its XRD characteristic lines. The presence of pyrite indicates reducing depositional environment.

Phosphate minerals such as apatite and carbonate fluorapatite minerals are occurred in most of the studied samples. The carbonate and phosphate minerals indicate their deposition in a marine environment [27]. The dominant of smectite and kaolinite clay minerals in the studied shale samples Fig. 10 indicate active chemical weathering of rocks under humid temperate to tropical climates, in continental weathering environments, smectite and kaolinite are generally formed [28].

4.9. Significant of trace elements

The following Table 3 show trace elements of the bulk rock samples from the studied mines.

The Vanadium average value 222.3 ppm in the studied shales is more than those of [29,30], which suggested that some V may be complexes within the organic matter. The Nickel (Ni) and Chromium (Cr) average values (136.02 and 186.7 ppm) is more than Ni and Cr of [29,30] indicate an association of these elements mainly with the organic matter and may relate to their basement complex provenance (mafic to ultramafic) for the sedimentary origin [31]. The enrichment of Cr and Ni in shales further reflects the incorporation of Cr and Ni ions into clay particles during the weathering of ultramafic rocks containing chromite and other Cr and Ni- bearing minerals [31].

5. Conclusion

Rock – Eval pyrolysis studies of surface samples from the studied phosphate mines at Safaga – Quseir area, Red Sea Coast, Egypt. Show that the samples have fair to very good TOC. The hydrocarbon source potential indicates predominantly immature that are oil prone. Bitumen analysis also, showed that the studied samples are immature and deposited under reducing environment. The (XRD) analysis for the studied shale samples show their bulk minerals are mainly quartz, calcite, dolomite, gypsum, hematite and pyrite in addition to apatite and fluorapatite. The association of smectite and kaolinite is referring to detrital origin and deposition in open marine environment. The chemical composition of the studied samples showed high concentration of trace elements such as Co, Cr, Cu, Ni, V and Zn. The V enrichment indicates deposition under reducing environment, Cr strong positive correlation with Cu, Zn and TOC indicates an association of these elements with the organic matter.

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