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STRATIGRAPHIC, MINERALOGIC AND GEOCHEMICAL CHARACTERIZATION OF GÜRÜN OIL SHALES, CENTRAL ANATOLIA, TURKEY

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A Middle Miocene playa-lake sedimentary sequence containing oil shales and trona is divided into the Gökpınar (the lower oil shale unit) and the Terzioğlu (the upper oil shale unit) Members in the Gürün Basin in eastern Turkey. Thermal decomposition of Gürün oil shales was studied by thermogravimetry (TGA), differential thermal analysis (DTA), and Rock Eval pyrolysis.

Na_2O content of oil shales reaches 0.2%, SiO_2 content reaches 31.5%, and CaO content ranges from 29.8 to 52.7%. Content of Sr, Cu, Zn, Ba and Zr (ppm) exceeds that of other trace elements. In the lower oil shale unit, TOC reaches 9.03 wt%, whereas in the upper oil shale unit, the maximum TOC value is 1.54 wt%, generally being even lower. Oil shale contains mainly calcite, aragonite and montmorillonite minerals. DTA curves of oil shale samples show that exothermic peaks are compatible with TOC values. Weight loss is compatible with chemical properties, DTA results and TGA curves of oil shale. Organic geochemical analyses indicate that Gürün oil shale contains sufficient amounts of kerogen of good quality to generate both oil and gas upon pyrolysis.

Introduction

Oil shale represents for many countries a valuable potential source of liquid hydrocarbons and energy [1]. Turkey's reliance on imported energy is high. Production of shale oil from oil shale is not one of the available energy generation alternatives in Turkey. However, the continuing decline of

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petroleum supplies, accompanied by increasing costs of petroleum-based products may present opportunities for oil shale to supply some of Turkey's energy needs. Turkish oil shale deposits are currently estimated to be approximately 1.865 million tons [2–6]. Oil shales of mainly Tertiary age such as Seyitömer-Kütahya, Beypazarı-Ankara, Hatıldağ-Bolu, Bahçecik-İzmit, Ulukışla-Niğde have been investigated for a long time by several researchers [2–6]. The Gürün Basin is a new deposit that has not been investigated in detail [7].

Geochemical, mineralogic and thermogravimetric analyses have been carried out on outcrop samples from the Gürün Basin south of Sivas (Gürün is a town of Sivas in the Central Anatolia). Oil shale occurs in a calcareous bituminous limnic sequence of Miocene age. Bituminous rocks occur in two zones: in the lower part of the Gökpınar Member and in the upper part of the Terzioğlu Member.

There is no information on thermal decomposition of Gürün oil shale. Information on its geochemical characteristics is also scanty. In this paper, we present the results of thermal and organic geochemical analyses of the Gürün Basin oil shales.

Experimental

Oil shales samples

Oil shale samples used in this work were taken from the Gürün Basin in the southern part of the Sivas-Turkey (Fig.1). The samples were crushed to <60 mesh. Thickness of Gürün oil shale bed varies from 0.2 cm to 5 m, and its color from grey to dark gray. It is medium hard and compact, brittle and thinly laminated.

Major and minor elements in Gürün oil shale

The content of major and minor elements in eight samples of Gürün oil shale was determined by X-ray fluorescence spectroscopy analysis (XRF).

Organic geochemical analysis

The quantities of organic matter in oil shale samples were determined basing on the total content of organic carbon (TOC) measured by LECO analyzer and on genetic potential (GP) (the sum of S_1 and S_2 values) established by Rock Eval pyrolysis. Hydrogen index HI (S_2/TOC) and oxygen index OI (S_3/TOC) were used to characterize the kerogen type. A plot of these indices on a van Krevelen diagram shows the type of organic matter present in oil shale samples. Production index $S_1/(S_1+S_2)$ and T_{max} were used to characterize the maturity of kerogen.

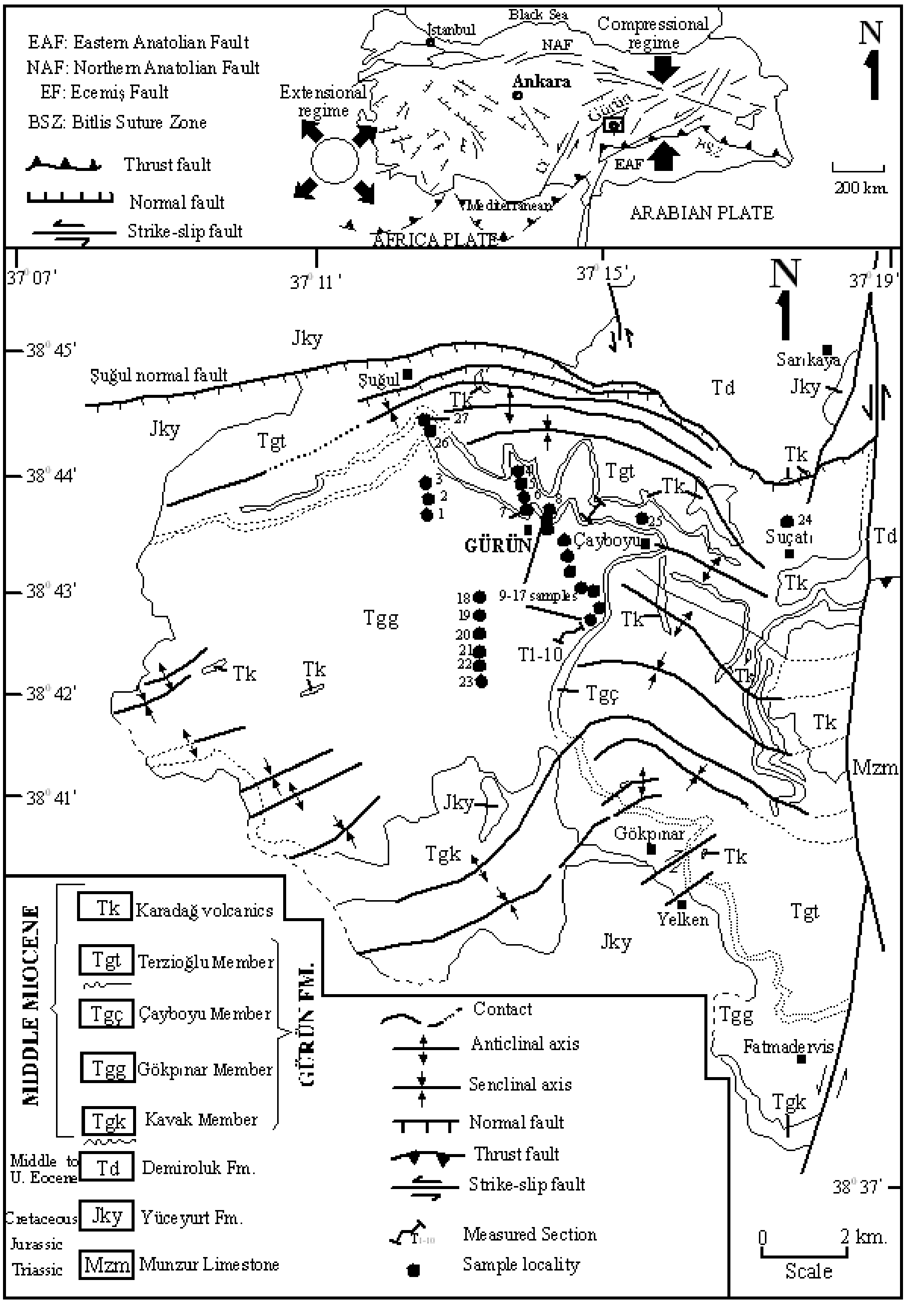

Fig. 1. Geologic map of the Gürün Basin indicating sample location (Modified from Önal et al. [7])

X-ray diffraction analysis

XRD analysis was carried out by RadB X-Ray diffractometer at scanning speed 1°/min from 2 to 60^o/ 2θ.

Thermal analysis

Differential thermal analysis (DTA) and thermogravimetric analysis (TGA) were performed with Shimdzu, DTA-50 and a TGA-50 thermal analyzers, respectively. DTA measurements were made under nitrogen at a heating rate of 10 °C/min in the air, whereas the TGA measurements were made at a heating rate of 10 °C/min.

Stratigraphy, lithology and tectonic setting

The Gürün Basin (Sivas), which is located in the heart of the Eastern Taurus Region, is bordered by the Suçatı and Şuğul faults in the east and the north, respectively (Fig. 1). The basin occupies approximately 100 km^2. The Gürün Basin, extending in an east-west direction, contains a volcanic-sedimentary sequence as thick as 1200 m [7]. Spore and pollen fossil assemblages in the sedimentary rock sequences have been determined to be of middle Miocene [8, 9] which is subdivided into sedimentary rocks belonging to Kavak, Gökpınar, Çayboyu and Terzioğlu Members of the Gürün Formation and Karadağ volcanic rocks [9]. All together they consist of clastic, calcareous, bituminous and evaporitic sediments and volcanic rocks [7]. The Karadağ volcanics consists of alternating pyroclastic breccia, tuff, cal-alkaline theoleitic basalt and basaltic andesite lavas, and agglomerate of Middle Miocene age. Volcanics located in the eastern and northern parts of the basin are intertwined with the upper part of the Terzioğlu Member, and also cut into the whole rock units [7].

In the region, gravity tectonic, which is characterized by the growth faults, began to develop in the beginning of middle Miocene. The growth faults, which were developed at the northern margin of the basin, controlled the deposition during Middle Miocene. The Gürün Basin was affected by the extensional tectonic regime changed to a compressional regime during Late Miocene. During this new tectonic phase, NE-SW compressional regime occurring in the region originated probably from the movement of the East Anatolian Fault (Fig. 1) [7].

Pre-Miocene rock units

The pre-Miocene basement rocks are composed of Munzur Limestone, Yüceyurt and Demiroluk Formations, which are Triassic-Jurassic-Cretaceous and Middle-Upper Eocene in age (Fig. 1), consisting of limestones and flysch-like sediments.

Middle Miocene rock units

The Gürün Formation already stated consists of conglomerate, sandstone, siltstone, limestone, oil shale, gypsum, lignite and tuff, which were deposited in fluvial, alluvial, lacustrine and playa-lake environments. The total thickness of the formation exceeds 1200 m [7].

The unit rests on the basement rocks with angular unconformity, but it is in fault contact with the basement rock in the northern and eastern sides of the Gürün Basin. Oil shale was identified in two members of the formation: the lower Gökpınar Member and upper Terzioğlu Member.

The lower oil shale unit (Gökpınar Member)

The Gökpınar Member is composed mainly of conglomerates, sandstones, siltstones, clayey limestones, limestones and mudstones with interbedded oil shales. The type section of the member is located about 750 m northwest of Gökpınar spring. At the reference section, the lower oil shale unit is 70 m thick. (Fig. 2a) and can be traced laterally about 5 km. The thickness of oil shale beds ranges from 2 cm to 5 m. Oil shale layers within the Gökpınar Member thin to the east and west. The oil shales were deposited during episodic expansions of the lake (Gürün middle-Miocene lake). The geological resource of the lower oil shale is estimated to be 15 million tons.

The upper oil shale unit (Terzioğlu Member)

The Terzioğlu Member of the Gürün Formation consists of limestones, clayey limestones, silicified limestone agglomerates, tuff, intraformational conglomerates and siltstones with interbedded oil shales ranging in thickness from 2 cm to 2.5 m. Leaching of trona is common in the lower and middle part of the Terzioğlu Member [7]. The upper part of the member includes micritic lacustrine limestone. The oil shale is yellow to dark grayish-greenish in color, moderately consolidated and thinly laminated, and is placed in the lower part of the member. The type section of the unit is located on Terzioğlu hill. At the type section, the member is 152 m thick (Fig. 2b) and is laterally continuous for about 4 km. Oil shales within the Gökpınar Member thin to the east and west. The member includes a transgressive and regressive sequence, and is gradational with the underlying Çayboyu member in the deeper parts of the basin, but is also in local unconformity with some members in the shallower parts of the basin [7]. Composition, sedimentologic features and fossil assemblages indicate that it was deposited in a playa-lake environment. Oil shales were deposited in a shallow brackish-water lake environment during seasonal flooding. The geological resource of the upper oil shale is estimated at 75 million tons.

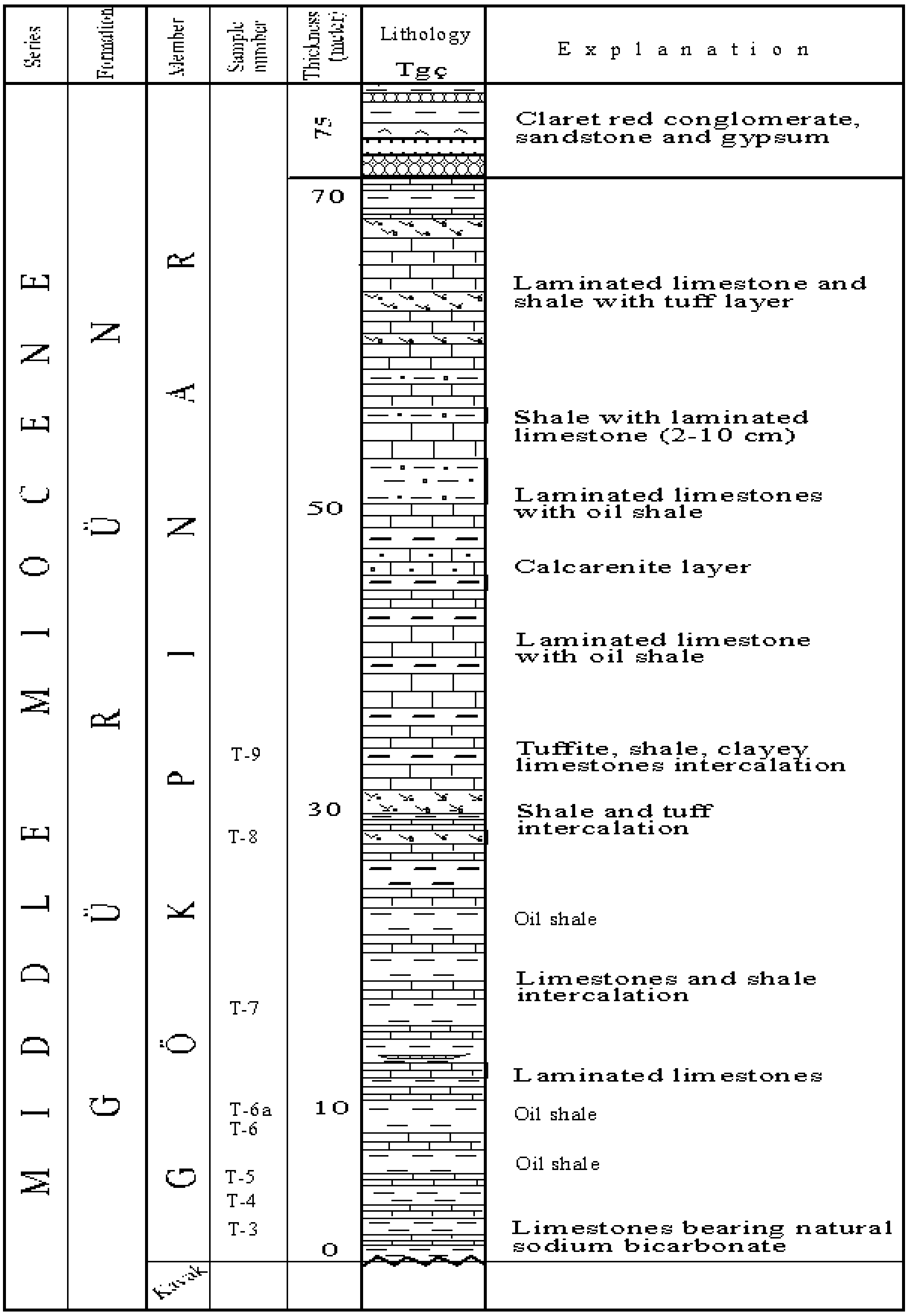

Fig. 2a. Measured section of the lower oil shale unit in the Gökpınar Member

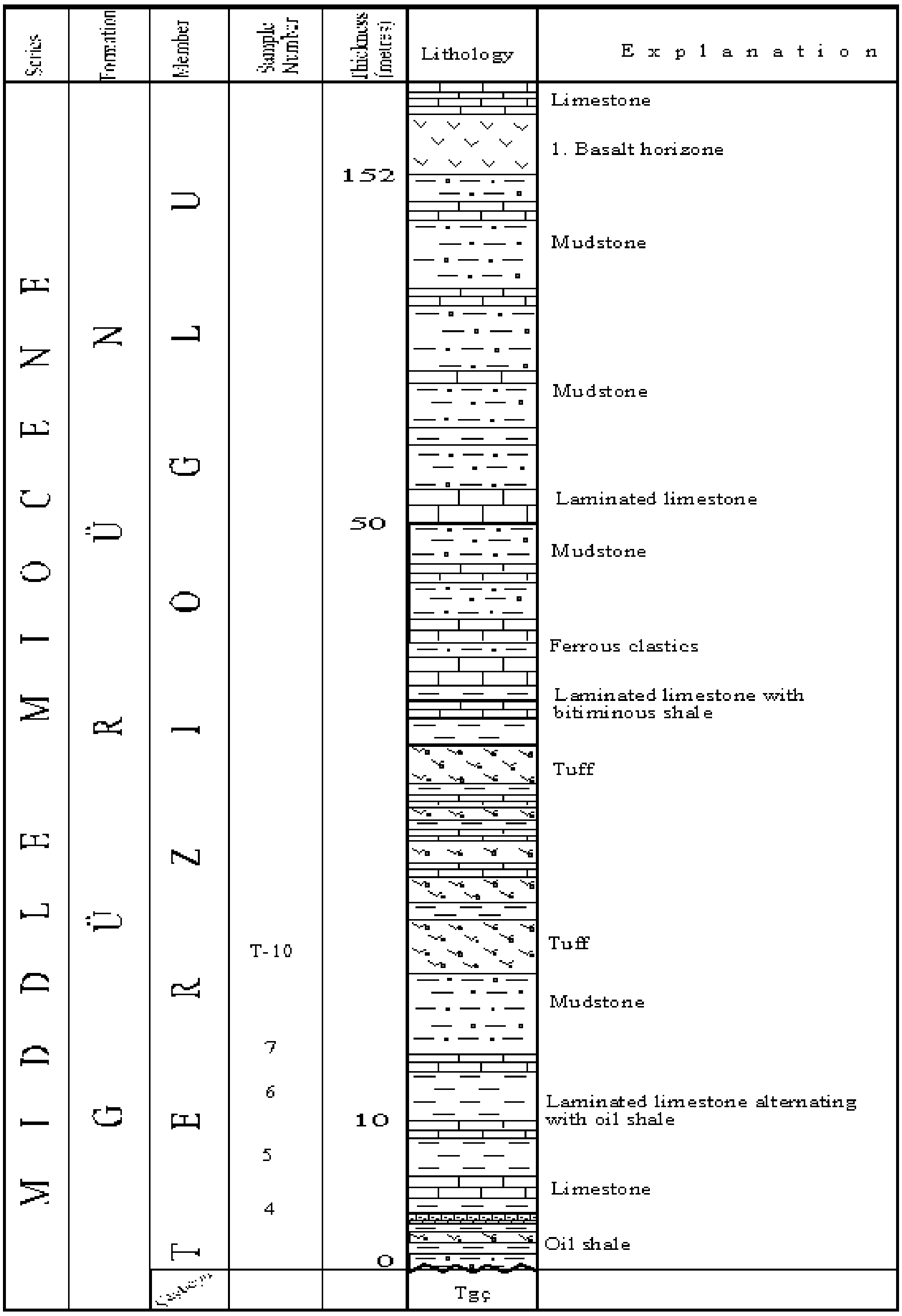

Fig. 2b. Measured section of the upper oil shale unit in the Terzioğlu Member

Results and discussion

Chemical composition of the eight oil shale samples is given in Table 1. Basing on the data, correlation coefficients between major and trace elements were calculated (Tables 2 and 3). These data show that all trace elements have a negative correlation with total organic carbon content.

Table 1. **Whole-rock analysis of major oxide and selected trace elements of the lower and upper oil shale units in the Gürün Basin and of Hatıldağ oil shale**

Sample number	T-3	T-6	14	17	18	19	22	24	Hatıldağ [4]
Major elements, wt%									
SiO_2	3.8	29.5	3.5	5.1	21.0	2.5	31.5	16.5	16.13
Al_2O_3	0.7	3.9	0.6	0.6	22	0.4	5.5	2.5	4.67
Fe_2O_3	0.2	2.6	0.3	0.3	1.4	0.2	3.5	1.0	3.18
MnO	< 0.1	0.1	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1	0.01
	0.9	1.0	1.1	0.9	0.9	0.9	1.3	3.5	4.3
CaO	51.5	33.6	52.5	50.2	4.0	52.7	29.8	42.0	29.42
Na_2O	< 0.1	0.2	< 0.1	< 0.1	0.1	< 0.1	0.1	0.1	1.58
K_2O	0.7	0.6	0.1	0.1	0.5	< 0.1	0.8	0.5	2.1
TiO_2	< 0.1	0.2	< 0.1	< 0.1	01	< 0.1	0.2	0.1	0.24
P_2O_5	0.1	< 0.1	< 0.1	< 0.1	0.1	< 0.1	< 0.1	< 0.1	0.16
L.I.	12.0	28.2	41.5	42.4	29.0	42.8	27.1	23.6	36.7
Total	99.8	100	100	100	99.4	100	100	100	98.47
Minor elements, ppm									
Sc	< 20	< 20	23	< 20	< 20	< 20	< 20	24	6
V	18	59	19	17	29	< 15	73	30	65
Cr	< 60	148	< 60	< 60	81	< 60	185	69	81
Co	< 50	< 50	< 50	< 50	< 50	< 50	< 50	< 50	nd
Ni	< 30	110	< 30	< 30	56	< 30	126	47	103
Cu	616	612	< 30	< 30	< 30	< 30	309	< 30	37.7
Zn	273	335	18	< 15	37	< 15	203	26	15
Rb	< 15	39	< 15	< 15	19	< 15	48	39	29.5
Sr	846	1533	446	946	1572	472	1511	1896	430
Y	< 15	< 15	< 15	< 15	< 15	< 15	< 15	< 15	5
Zr	47	114	< 30	103	53	< 30	121	120	14
Nb	< 20	< 20	< 20	< 20	< 20	< 20	< 20	< 20	3.95
Ba	< 100	< 100	< 100	< 100	212	< 100	< 100	310	235
La	< 40	< 40	< 40	< 40	< 40	< 40	< 40	< 40	nd
Ce	< 40	< 40	< 40	< 40	< 40	< 40	< 40	< 40	nd
Pb	< 20	< 20	< 20	< 20	< 20	< 20	< 20	< 20	12
Nd	< 20	< 20	< 20	< 20	< 20	< 20	< 20	< 20	nd
Yb	< 15	< 15	< 15	< 15	< 15	< 15	< 15	< 15	nd
Th	< 15	< 15	< 15	< 15	< 15	< 15	< 15	< 15	nd
U	< 15	< 15	< 15	< 15	< 15	< 15	< 15	< 15	nd

nd: not determined

Table 2. **Correlation coefficient between main and trace elements (XRF data)**

	SiO_2	Al_2O_3	Fe_2O_3	MgO	CaO	Na_2O	K_2O	TiO_2	L.I
Sc	1.00	1.00	1.00	1.00	–1.00	nd	1.00	nd	–1.00
V	0.93	0.98	0.99	0.01	–0.94	0.34	0.66	0.96	–0.16
Cr	0.97	0.97	1.00	–0.52	–1.00	0.33	0.94	0.96	0.34
Ni	0.98	0.35	0.99	–0.55	–0.99	0.43	0.90	0.98	0.38
Cu	–0.56	–0.76	–0.72	–0.97	0.64	1.00	–0.86	nd	–0.46
Zn	0.34	0.36	0.42	–0.43	–0.34	1.00	0.66	0.93	–0.50
Rb	0.52	0.80	0.64	0.27	–0.64	0.15	0.73	0.68	–0.45
Sr	0.80	0.73	0.65	0.57	–0.80	–0.35	0.56	–0.68	–0.50
Zr	0.49	0.59	0.53	0.48	–0.54	0.24	–0.07	0.55	0.42
Ba	–1.00	1.00	–1.00	1.00	1.00	nd	nd	nd	–1.00
TOC	0.43	0.35	0.28	0.65	–0.40	0.49	–0.12	–0.15	–0.07

nd: not determined

Table 3. **Correlation coefficient between trace elements (XRF data)**

	Sc	V	Cr	Ni	Cu	Zn	Rb	Sr	Zr	Ba	TOC
Sc	1.00	–0.96	–1.00	–1.00	nd	0.74	nd	0.55	1.00	1.00	1.00
Cr			1.00	0.82	–1.00	0.82	0.68	0.14	0.54	–0.97	0.19
Ni				1.00	–1.00	0.65	0.66	–0.43	0.08	–0.43	–0.40
Cu					1.00	0.88	–1.00	–0.49	–0.58	nd	–0.33
Zn						1.00	0.51	0.22	0.35	–0.22	0.28
Rb							1.00	0.01	0.95	1.00	–0.18
Sr								1.00	0.74	0.49	0.14
Zr									1.00	0.33	0.59
Ba										1.00	0.61
TOC											1.00

nd: not determined

Except Sc, the amounts of trace elements do not depend on deposition of organic matter. The arithmetic mean for SiO_2 is 14.75 wt%. Content of Na_2O and SiO_2 may reach 0.2% and 31.5 wt%, respectively. CaO content ranges from 29.8 to 52.7 wt%, and content of Sr, Cu, Zn, Ba and Zr (ppm) exceeds that of the other trace elements.

Low Na content in oil shale samples may be due to the formation of Na-carbonate minerals (trona) [7]. On the other hand, Sr content of samples is high. High Sr values could originate from limestones of marine basement such as those in the Munzur and Yüceyurt Formations. Also, these results were compared with the data on Hatıldağ (Miocene) oil shale. As for major elements, their contents in Gürün and Hatıldağ oil shales are similar; however, there are some differences such as the abundances of CaO and MgO. In addition, the ratio K_2O/Na_2O in the Gürün samples is 2–6 times higher than that in the Hatıldağ sample, and the ratio Al_2O_3/Na_2O is 6–18 times higher. The ratio SiO_2/Al_2O_3 in the Gürün samples is higher than that in the Hatıldağ sample. As for the content of the minor elements, the oil shales from these two fields contain different amounts of lead, copper, strontium, barium and niobium.

XRD data of the samples are shown in Fig. 3. All samples contain aragonite and calcite, and elemental analysis supports this data. Samples 14, 18, 17 and 19 contain significant amounts of calcite, and it dominates in sample 22. In addition, a clay mineral smectite was determined in this sample (peak at 16.23 and 4.446 Å). However, spectra of some samples (e.g., sample 17) show little or no clay minerals. High calcite content of samples is accompanied by minor amounts of clay. Comparison between TOC results and clay content shows that samples 18 and 22 that contain detectable amounts of clay contain also much TOC by LECO, 4.05 wt% and 4.55 wt%, respectively. The two samples containing clay minerals are rich in organic matter that may have resulted from absorption of organic matter between clay mineral sheets. Leachates of oil shale samples of the Terzioğlu Member contain starkeyite, hexahydrite, dolomite and calcite [7].

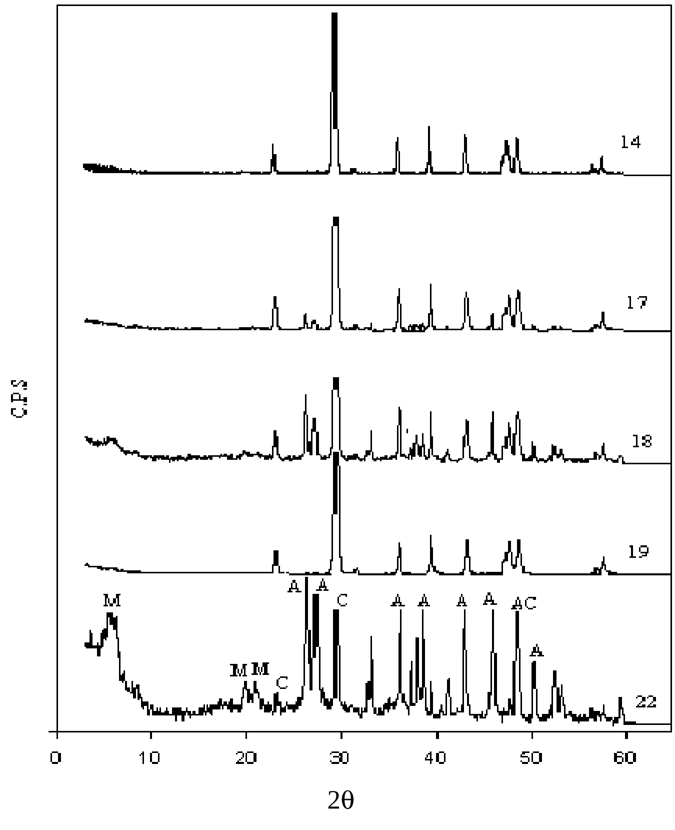

Fig. 3. XRD patterns of investigated samples (A: aragonite, C: calcite, M: montmorillonite)

DTA spectra of all analyzed samples in Fig. 4 show generally the same pattern. Different exothermic peaks between 200 and 500 °C related to TOC (Table 4) indicate that the samples contain organic materials of different molecular masses. The maximum exothermic peak of sample 22 indicates that the sample is rich in aromatic carbon, and that the result is compatible with its high TOC contest. The maximums of exothermic peaks of samples 22, 14, 17 and 19 were recorded at 460 °C, 345 °C, 356 °C and 354 °C, respectively. Endothermic peaks of all samples observed at 787–825 °C result from decomposition of aragonite and calcite [10]. As samples 17, 18 and 22 contain aragonite, their endothermic peak maxima appear at a lower temperature. The endothermic doublet of sample 22 shows decomposition of aragonite and calcite at temperatures 788 °C and 820 °C, respectively.

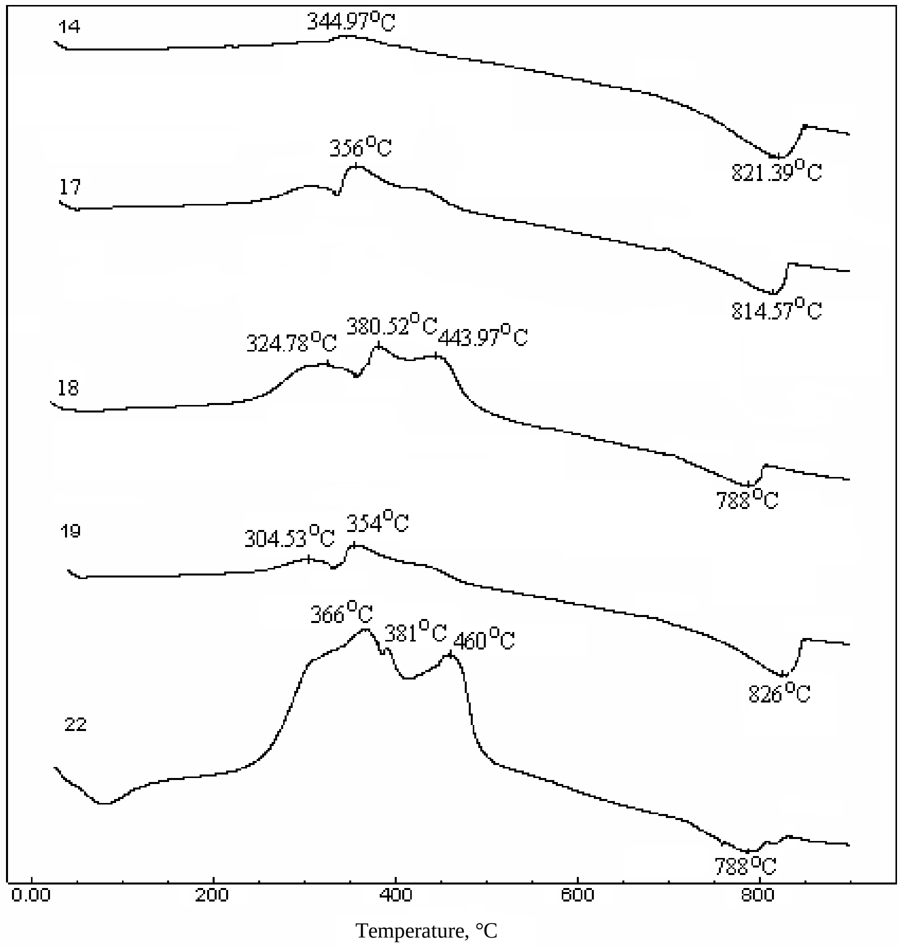

Fig. 4. DTA curves of five oil shale samples

TGA curves of the same five samples are presented in Fig. 5. Weight losses of the samples are compatible with their TOC content indicating decomposition of organic matter. The highest weight losses were determined in samples 17 and 22. Weight losses in these samples above 700 °C result from decomposition of aragonite and calcite. The other weight losses are probably related to decomposition of different organic compounds occurring in different samples. Considering DTA data, these samples are interpreted to contain high-molecular aromatic compounds. Weight loss data of samples differ, because the samples contain different amounts of organic matter, although temperature interval of alteration of the samples is the same. TGA curves of samples 14 and 19 show one case of weight loss (37% and 36%), those of samples 17 and 18 – two cases (4 and 35% and 10 and 27%), and the curve of sample 22 – three cases (8, 16 and 26%).

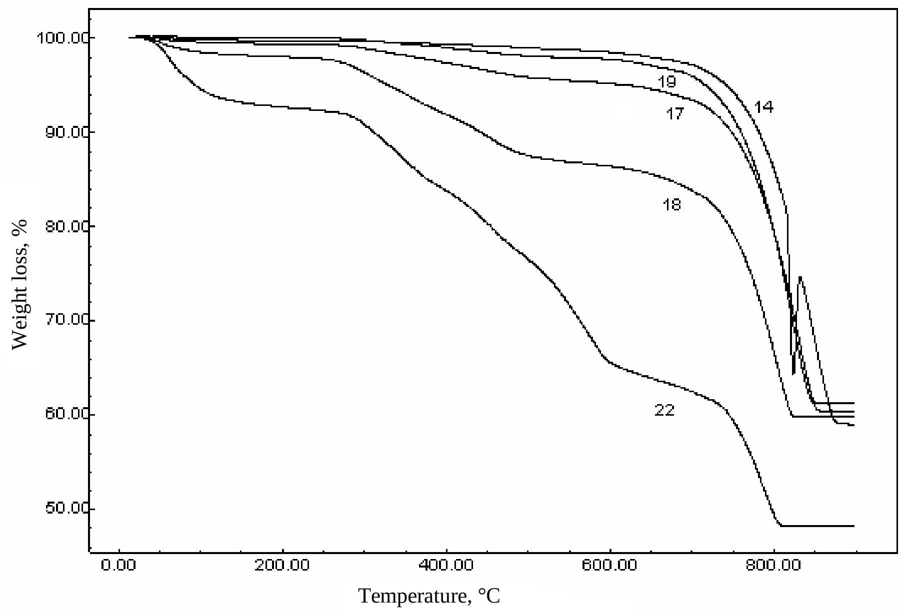

Fig. 5. TGA curves of five oil shale samples

Characterization of organic matter

The oil yield upon pyrolysis depends on the abundance, type and thermal maturity of oil shale organic matter. The quantity of the organic matter was determined basing on TOC determined by Rock-Eval 2 Analyzer and values of genetic potential (GP) (Table 4). Oil shale samples of the lower unit are characterized by TOC values that exceed the minimal one (0.5% TOC); 2.0 mg/g GP are required for a rock to be a potential oil source [11]. TOC values of these oil shale samples are between 0.05 and 9.03 wt%, and contents of S_1 and S_2 reach 5.66 mg/g and 47.7 mg/g, respectively. T_{max} values range from 398 to 427 °C, and HI values are as high as 810. TOC and GP results characterizing lower oil shales prove the potential of those shales to be used for oil generation. However, in the upper oil shales, TOC values range from 0.05 to 1.54 wt%, GP (S_1+S_2) values are very low, except one sample, T_{max} values range from 411 to 436 °C, HI values are generally low and OI values very high.

Table 4. **Total organic carbon (TOC %) and results of Rock-Eval pyrolysis of the samples taken from the lower and the upper oil shale units in the Gürün Basin**

Upper Oil Shale Unit								
Sample No	TOC, %	S_1	S_2	S_3	T_{max}	PI	HI	OI
1	0.07	0.04	0.15	0.35	436	0.22	214	500
3	0.05	0.04	0.08	0.36	429	0.33	160	720
9	0.07	0.03	0.16	0.81	424	0.17	228	1157
24	1.54	0.82	6.31	1.80	419	0.12	409	116
26	0.18	0.04	0.19	0.42	411	0.18	105	233
Lower Oil Shale Unit								
T-1	0.58	0.27	1.99	0.60	427	0.12	343	103
T-3	1.34	1.02	6.33	1.01	408	0.14	472	75
T-6	4.41	5.12	25.02	2.15	394	0.17	394	567
T-9	0.05	0.04	0.11	0.36	422	0.29	422	220
14	0.51	0.38	2.85	0.56	418	0.12	558	109
17	4.05	4.51	28.48	2.06	398	0.15	696	53
18	2.63	3.11	18.32	1.40	399	0.14	703	50
19	1.78	1.89	14.35	0.90	399	0.12	810	50
22	4.55	3.90	23.83	2.59	395	0.14	522	56
28	3.58	1.62	21.97	nd	398	0.07	613	nd
29	5.91	0.68	16.13	nd	424	0.04	272	nd
30	9.03	5.66	47.7	nd	406	0.11	528	nd

nd: not determined

The quality or type of organic matter present in oil shales was inferred from the plots of hydrogen index (HI) versus oxygen index (OI), as presented in a modified van Krevelen diagram in Fig. 6. The samples are mainly plotted between types II and III pathways but closer to type II, and some to type I. This type of organic matter can yield both oil and gas upon pyrolysis [11]. In terms of thermal maturity, both the temperature at S_2 peak maximum (T_{max}) and production index (PI) are generally lower than 435 °C and 0.1 expected for a mature shale. Figure 7 shows that most samples are plotted within the immature, but two of them in the mature zone. The samples from the lower unit with high GP are immature but have the potential to yield both oil and gas under pyrolysis.

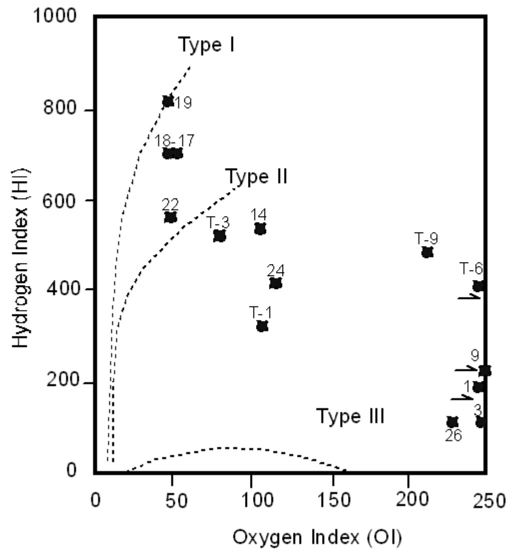

Fig. 6. Modified van Krevelen diagram of Gürün oil shales (After Espitalié et al. [12])

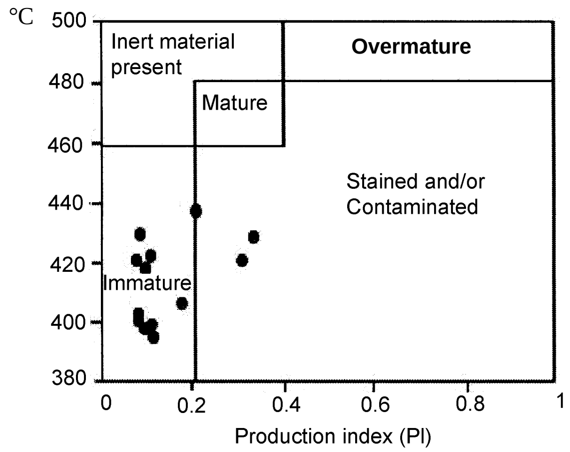

Fig. 7. Plots of T_{max} (°C) against production index (PI) for Gürün oil shales

Conclusions

In this paper, oil shales are regarded as two different units: the lower oil shale unit in the Gökpınar Member, and the upper oil shale unit in the Terzioğlu Member. The results show that

1. Their CaO content ranges from 29.8% to 52.7%, and content of Sr, Cu, Zn, Ba (ppm) exceeds that of other trace elements.

2. The data on weight loss measured by TGA are compatible with DTA data and elemental composition of the samples. Generally**,** weight losses of all samples are about the same.
3. DTA curves demonstrate different exothermic peaks, which are also compatible with TOC values of the samples. In addition, specific endothermic peaks of aragonite and calcite were observed.
4. The lower oil shale unit is characterized by different content of organic matter (from 0.05 to 9.03 wt %), high genetic potential (up to 53.36 mg HC/g) and mostly type II kerogen. According to data, this oil shale yields oil and gas upon pyrolysis. Organic part of some samples (17 and 22) was considered to contain more aromatics than the others.

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