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Study of the component composition of organic matter of the East Pre-Caucasian basin rocks based on the results of lithological, petrophysical and geochemical studies

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Abstract. This study introduces results of lithological, petrophysical and geochemical investigation of Lower Cretaceous (K_1) and Middle Jurassic (J_2a-b) rocks of East Pre-Caucasian basin.

According to pyrolytic and bituminological studies method of separate determination of kerogen and bitumen concentration been developed. In accordance with this method differentiation of organic matter components in different lithotypes of rocks been described. Also relationship between bitumen and kerogen concentrations been revealed.

The majority of samples have poor to fair organic richness and poor source potential. Kerogen type is commonly presented by type III and stages of maturity characterized by stages PC_3 to MC_3. Bitumen compounds have low concentrations of asphaltenes and aromatic hydrocarbons and mainly contains light and heavy resins.

Based on petrophysical and geochemical studies a close relationship between the concentration of organic carbon and the weight concentration of potassium nuclides was obtained. This relationship indicates that kerogen in the sediments under consideration is associated with clay minerals, which is also confirmed by the mineral composition of the rocks.

Keywords: kerogen, bitumen, source rocks, pyrolysis, extraction

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An analysis of the literature data showed that most of geochemical characteristics of rock investigations of North Caucasus and the East Pre-Caucasian sediments deal with Miocene and Oligocene deposits, in particular the Maikop series rocks (Kerimov et al., 2015; Kerimov et al., 2017; Lukanova, 2011; Kholodov, Nedumov, 1981; Yandarbiev et al., 2017; Vincent, Kaye, 2018). However, the industrial oil and gas potential of the North Caucasus and East Pre-Caucasian basin is also associated with oil and gas complexes of the Jurassic and Cretaceous periods (Sokolov et al., 1990; Orel et al., 2001; Kerimov et al., 2014). The aim of this work is to study conversion of organic matter regularities of the Lower Cretaceous (K_1) and Middle Jurassic (Aalenian and Bajocian stages, J_2a-b) terrigenous rocks of the East Pre-Caucasian basin.

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The object of investigation is one of the studied areas (area П2) of the East Pre-Caucasian oil and gas region. Oil and gas content of the Lower Cretaceous and Middle Jurassic sediments are confirmed by drilling and well tests, as well as the oil and gas content of the same sediments at nearby areas. The overview map of the research area is shown in Fig. 1 (according to Khisamov et al., 2020). Characteristics of studied sediments presented in table 1.

The study of the regularities of the interconnected processes of catagenetic transformation of organic matter (OM) in the terrigenous and terrigenous-carbonate oil and gas mater matrix requires closer attention due to the need to develop difficult-to-recover hydrocarbon reserves confined to the oil and gas source rocks of the West Siberian, Caspian, North Caucasus and Volga-Ural provinces.

In this paper, we studied the Lower Cretaceous and Middle Jurassic rocks of the East Pre-Caucasian basin, which in the core selection zone are represented by different-grained polymictic sandstones with clay-

Fig. 1. Overview map of studied area П2 of East Pre-Caucasian oil and gas region (according to Khisamov, et al., 2020)

Location	Lithology	Age	Tectonic association
East Pre-Caucasian basin	Terrigenous rocks	K_1, $J_{2a\text{-}b}$	Chograyskiy trough of Manychskiy troughs area

Table 1. Characteristics of studied rocks

carbonate cement and clay-siliceous-carbonate rocks. The mineral composition of the clastic part is mainly represented by quartz, fragments of igneous acidic rocks and feldspar. According to the mineral composition of the studied rocks, two types of cement predominate. The first one is characterized by a mixed clay mineral composition: illite, kaolinite, and chlorite. The type of cementation is mainly basal, sometimes film-pore. The second type of cement is carbonate, with a mineral composition of dolomite-siderite. The reservoir rock porosity reaches 25 %.

Standard pyrolytic studies were performed at a Rock-Eval 6 Turbo type installation (Behar et al., 2001) under sequential temperature conditions: 0–180–650–800 °C (sample weight for analysis was 70 mg with fraction 0.25 mm). Based on the results of pyrolytic studies (Table 2) for non-extracted samples based on HI = f(T_{max}) diagram (HI – hydrogen index according to pyrolysis studies; T_{max} – temperature of maximum hydrocarbon output at S_2 peak; S_2 – pyrolysis peak fixed by flame ionization detector in the temperature range 300–650 °C), it can be concluded that the kerogen is mainly represented by mixed type (Fig. 2a). In accordance with S_2 = f(TOC) (Total organic carbon) cross-plot (Fig. 2b) organic carbon content varies from fair to poor, the kerogen generation potential is poor (Peters, Cassa, 1994), and level of maturity is characterized by grades from PC_3 to MC_3 (Vorob'eva, 2014). The Lower Cretaceous deposits are characterized by grades PC_3-MC_1, and the Middle Jurassic MC_1-MC_2. Due to the fact that OM concentrations are low and pyrolytic studies were performed on non-extracted samples, the use of the Tmax parameter to estimate the degree of kerogen conversion may not be correct enough and may lead to incorrect results (Chen et al., 2016; Dembicki, 2009).

For effective extraction of high – molecular bituminous components (bitumen), powders (grain diameter 0.25 mm) of selected samples were prepared, which were successively extracted with the following solvents: chloroform (CF1) – alcohol-benzene (AB1) – chloroform (CF2) – alcohol-benzene (AB2).

Quantitative determination of the content of bitumen in the isolated chloroform extracts was carried out by weight method. The chloroform extract filtered on a de-salted filter was placed in an open container to remove the solvent by evaporation. The concentration of bitumen was determined by the weight difference between the pre-weighted container and the container with bitumen dried to a constant weight. Thus obtained quantitative values of the content of bitumen for 4 gram of rock were converted into weight percentages. The component composition of the bitumen was determined by capillary extracts method.

Results of complex processing of pyrolysis and extraction data

Separate determination of organic matter components is important, both in the analysis of the regularities of the transformation of OM, the degree of maturity of OM, and in reservoir properties determination of rocks.

Fig. 3 shows the dependence between extracted bitumen concentrations to total organic carbon (TOC). The diagram also indicates low concentrations of organic carbon and sufficiently high concentrations of bitumen for some samples, indicating a sufficiently high degree of organic matter conversion and the possible presence of light oil.

The component composition of bitumen is an important criterion for assessing the maturity of the OM and its ability to generate petroleum hydrocarbons. At the early stages of generation, the component composition of bitumen is mainly represented by asphaltenes and heavy (alcohol-benzene) resins, the proportion of which decreases significantly during maturation and passes into tarred components (light resins), oils and liquid petroleum hydrocarbons (Bogorodskaya et al., 2005; Vassoevich, 1982; Jarvie et al., 2015).

Sample	Age	S1 (mg/g)	S2 (mg/g)	PI Production Index	Tmax (°C)	TOC (%)	HI Hydrogen Index
1VB/3	K1	0.02	0.1	0.13	610	0.07	143
1VB/4	K1	0.19	0.39	0.33	360	0.16	244
1VB/5	K1	0.06	0.3	0.17	427	0.12	250
1VB/6	K1	0.04	0.48	0.07	415	0.09	533
1VB/7a	K1	0.08	0.15	0.35	428	0.22	68
1VB/7б	K1	0.09	0.19	0.31	608	0.23	83
1VB/8	K1	0.08	0.27	0.22	611	0.21	129
1VB/9	K1	0.09	0.33	0.22	427	0.21	157
1VB/10	K1	0.05	0.38	0.13	428	0.25	152
1VB/15	K1	0.12	0.26	0.32	429	0.33	79
1VB/16	K1	0.13	0.52	0.2	425	0.38	137
1VB/17	K1	0.08	0.25	0.24	428	0.38	66
1VB/18	K1	0.13	0.62	0.18	611	0.17	365
1VB/20	K1	0.06	0.58	0.1	610	0.29	200
1VB/22	J2a-b	0.11	0.24	0.32	609	0.12	200
1VB/24	J2a-b	0.14	0.21	0.4	611	0.05	420
1VB/25	J2a-b	0.1	0.29	0.26	610	0.07	414
1VB/26	J2a-b	0.09	0.27	0.25	611	0.09	300
1VB/28	J2a-b	0.09	0.27	0.25	441	0.2	135
1VB/29	J2a-b	0.1	0.32	0.24	598	0.17	188
1VB/32	J2a-b	0.07	0.31	0.18	437	0.4	78
1VB/33	J2a-b	0.18	0.5	0.26	436	0.4	125
1VB/35	J2a-b	0.05	0.51	0.09	438	0.4	128
1VB/38	J2a-b	0.07	0.57	0.12	438	0.52	110
1VB/39	J2a-b	0.13	0.63	0.17	439	0.62	102
1VB/40	J2a-b	0.35	0.75	0.32	439	0.7	107
1VB/42			0.6	0.25	440	0.67	90
1VB/43	J2a-b	0.16	0.9	0.15	441	0.82	110
1VB/45	J2a-b	0.2	1.3	0.14	442	0.76	171
1VB/47	J2a-b	0.09	0.67	0.12	442	1	67
1VB/54	J2a-b	0.16	0.98	0.14	443	1.08	91
1VB/57	J2a-b	0.2	1.18	0.15	441	0.69	171

Table 2. The results of pyrolitic studies

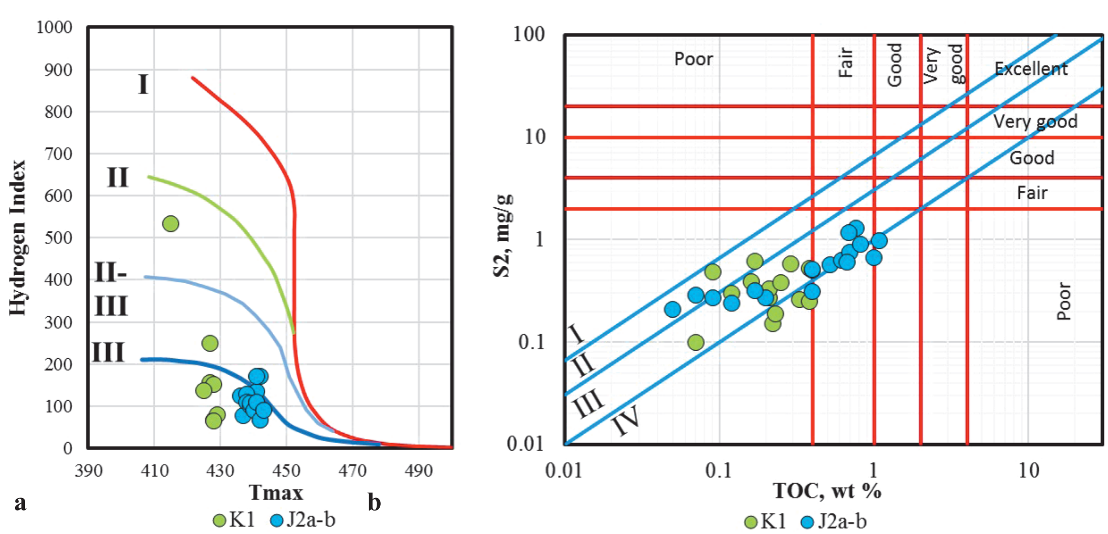

Fig. 2. HI = f(T_{max}) diagram (a) and S_2 = f(TOC) cross-plot (b) for Lower Cretaceous and Middle Jurassic rocks of East Pre-Caucasian basin

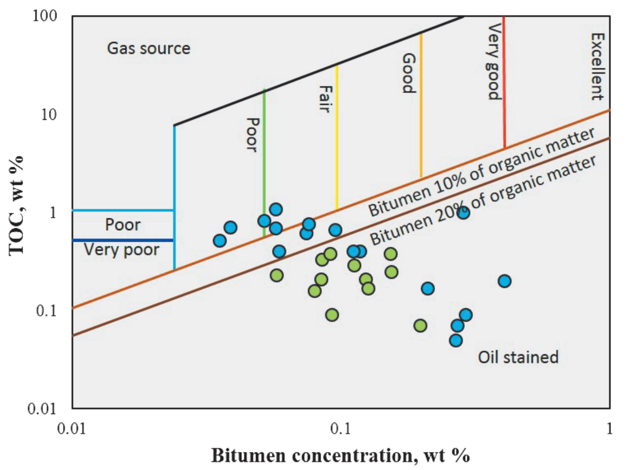

Fig. 3 Plot of bitumen extractions versus TOC content for Lower Cretaceous and Middle Jurassic rocks of East Pre-Caucasian basin

The studied samples of bitumen from the rocks of the Lower Cretaceous and Middle Jurassic deposits of one of the areas of the East Pre-Caucasian basin are characterized by a low content of asphaltenes (except for some samples of J_2a-b age). Concentrations of aromatic hydrocarbons are relatively low. There is a relatively high content of light and heavy resins. This allows us to conclude that the degree of maturity of the OM corresponds mainly to the grades of MC_2 – the beginning of MC_3 (Jarvie et al., 2015). The average component composition of bitumen is shown in Table 3.

A method for the separate determination of concentrations of kerogen and bitumen

Without consideration of sulfur and nitrogen proportion in the elemental composition of organic matter, the sum of weight pyrolytic parameters can be considered as the weight concentration of organic matter, soluble (bitumen) and insoluble (kerogen) in organic solvents (total weight concentration of organic matter or TOM – Total Organic Matter). The TOM parameter defined on non-extracted samples is the total concentration of kerogen and bitumen (in weight %) (Samokhvalov et al., 2020). If extracted samples are used, the TOMex parameter characterizes the concentration of kerogen (in weight %).

In the presence of pyrolytic studies on non-extracted powders and data on powder extraction for the same core sample, it is possible to estimate the weight concentration of kerogen in the sample (Samokhvalov et al., 2019). To do this, the weight concentration of bitumen obtained by extraction must be subtracted from the total concentration of OM in the non-extracted powder (TOM):

$$\begin{cases} TOM = RC + PC + PH + PO = C_{BMK} + C_{KEP} \\ TOM_{ex} = C_{KEP} \approx TOM - C_{BMK} \end{cases} \quad (1)$$

where RC – (Residual organic carbon) weight concentration of carbon formed in the oxidation phase; PC (Pyrolysable organic carbon) weight concentration of carbon formed in the pyrolysis phase; PH (Pyrolysable organic hydrogen) weight concentration of hydrogen formed in the pyrolysis phase; PO (Pyrolysable organic oxygen) weight concentration of oxygen formed in the pyrolysis phase, C_{KER} – weight concentration kerogen, C_{BIT} – weight content of bitumen (according to the results of extraction (all values in weight %).

Fig. 4 shows the change in the total concentration of OM and the concentration of bitumen with a change in porosity for the lithotypes of rocks represented in the core selection interval.

Sandstones with carbonate-clay cement are characterized by a low content of kerogen and the highest concentrations of bitumen and are characterized by an open porosity of 14 % to 25 %. Clay sandstones are characterized by average concentrations of kerogen and bitumen and an open porosity in the range from 7 to

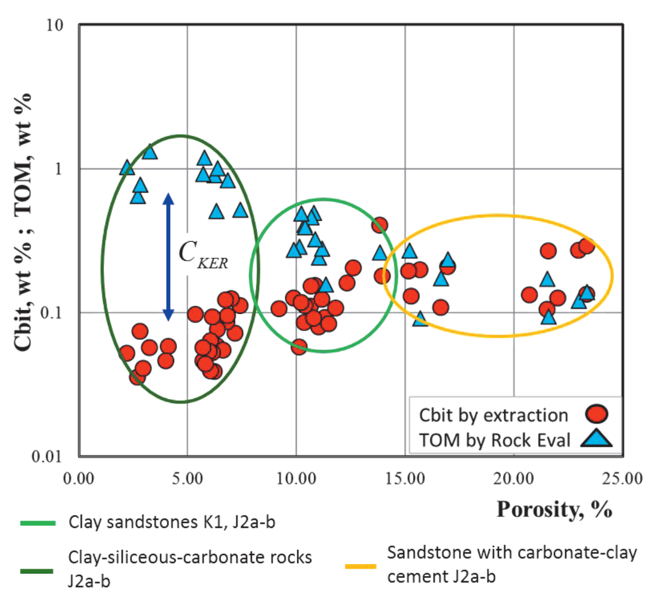

Fig. 4. Regularities of changes of TOM and bitumen concentration with changes in porosity of different rock lithotype of East Pre-Caucasian basin

	Age	Oils, %			Resins, %		Asphaltenes, %
		Aromatic HC	Methane-naphthene HC	∑(oils)	Light	Heavy	
East Pre-Caucasian Basin	K_1	11.6	25.6	37.2	47.3	12.6	2.8
	J_2a-b	1.4	12.3	13.7	44.7	33.9	7.7

Table 3. Average component composition of bitumen of the Lower Cretaceous and Middle Jurassic deposits of the East Pre-Caucasian Basin

14 %. Clay-siliceous-carbonate rocks are characterized by relatively high kerogen content and low bitumen content, and the open porosity varies between 3 % and 7 %.

Fig. 5 shows the relationship between the kerogen content and the ratio of bitumen content to kerogen content for the Middle Jurassic and Lower Cretaceous rocks of the East Pre-Caucasian basin. For type III kerogen, this dependence is described by a single linear function.

The current resource potential of the kerogen of the presented deposits is poor: the values of the pyrolysis parameter S_2 for this collection of samples range from 0.05 to 1.3 mg/g.

The component composition of bitumen in the lithotypes under consideration has approximately the same composition and mainly consists of resins, with sufficiently high concentrations of methane-naphthene oils. Fig. 6 shows a diagram of the average relative component composition of the rocks OM Mesozoic sediments of the East Pre-Caucasian basin.

Results of interpretation of data from geochemical and well logging data

Traditionally, the concentration of kerogen is correlated with uranium, but at low concentrations of uranium there are dependencies with thorium and potassium, which may be explained by association of kerogen with clay or other minerals (Kozhevnikov, 1997; Kozhevnikov, 2000; Fertl, 1979; Schmoker, 1981). For the Lower Cretaceous and Middle Jurassic sediments of the East Pre-Caucasian basin, the relationship of TOC with uranium is widely dispersed, while for thorium, potassium and the integral uranium equivalent, closer relationships can be noted. Fig. 7 shows the obtained close relationship of TOC with the weight content of potassium (R = 0.847).

Fig. 8 shows the results of pyrolytic and bituminological studies and the results of well logging data interpretation for the interval under consideration. Track 10 shows a volumetric rock model calculated using well logging methods. Based on the data presented in track 8 and 9, it can be concluded that clay sandstones

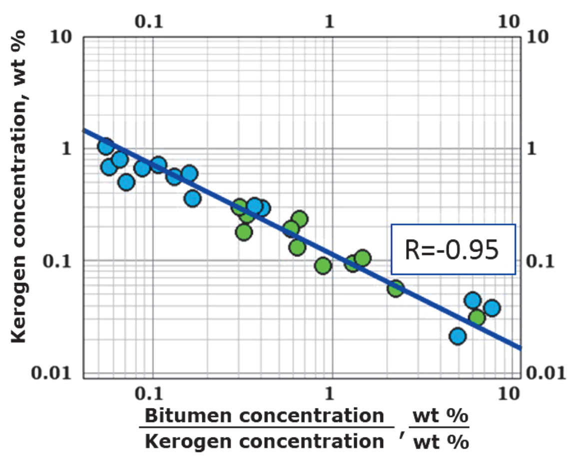

Fig. 5. Relationship the kerogen content and the ratio of bitumen content to kerogen content for Middle Jurassic and Lower Cretaceous rocks of the East Pre-Caucasian basin

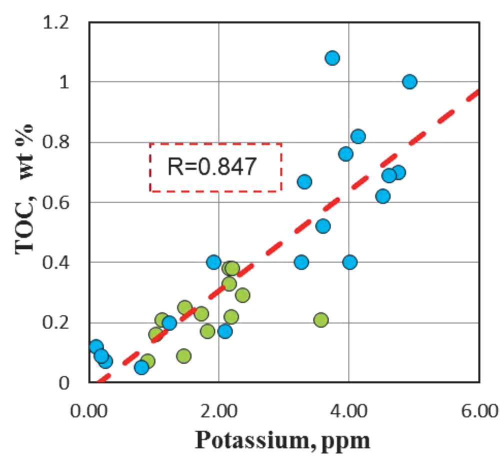

Fig. 7. TOC = f(Potassium) cross-plot for Middle Jurassic (green points) and Lower Cretaceous (blue points) rocks of the East Pre-Caucasian basin

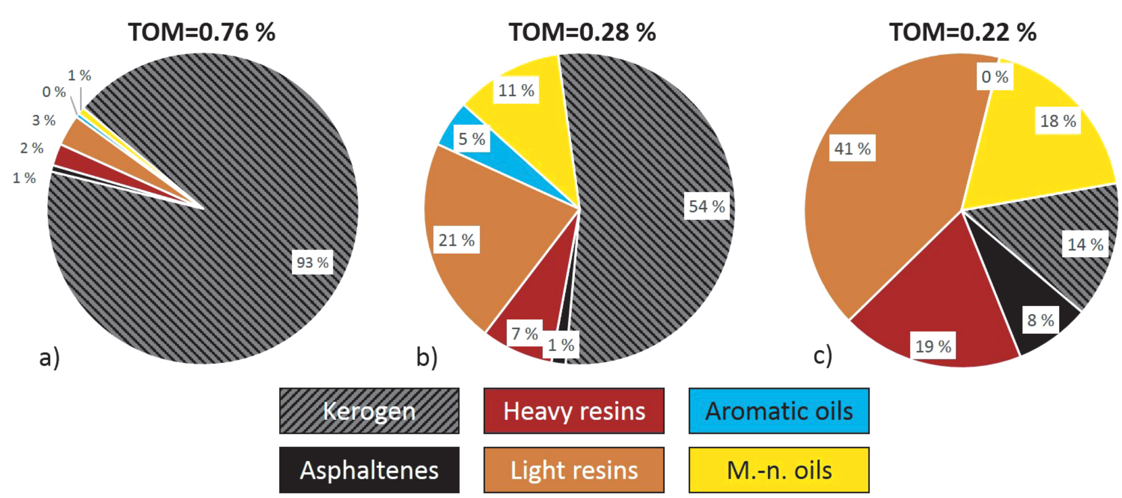

Fig. 6. Average organic matter component of a) clay-siliceous-carbonate rocks, b) clay sandstones and c) sandstones with carbonate-clay cement of Mesozoic sediments of the East Pre-Caucasian basin

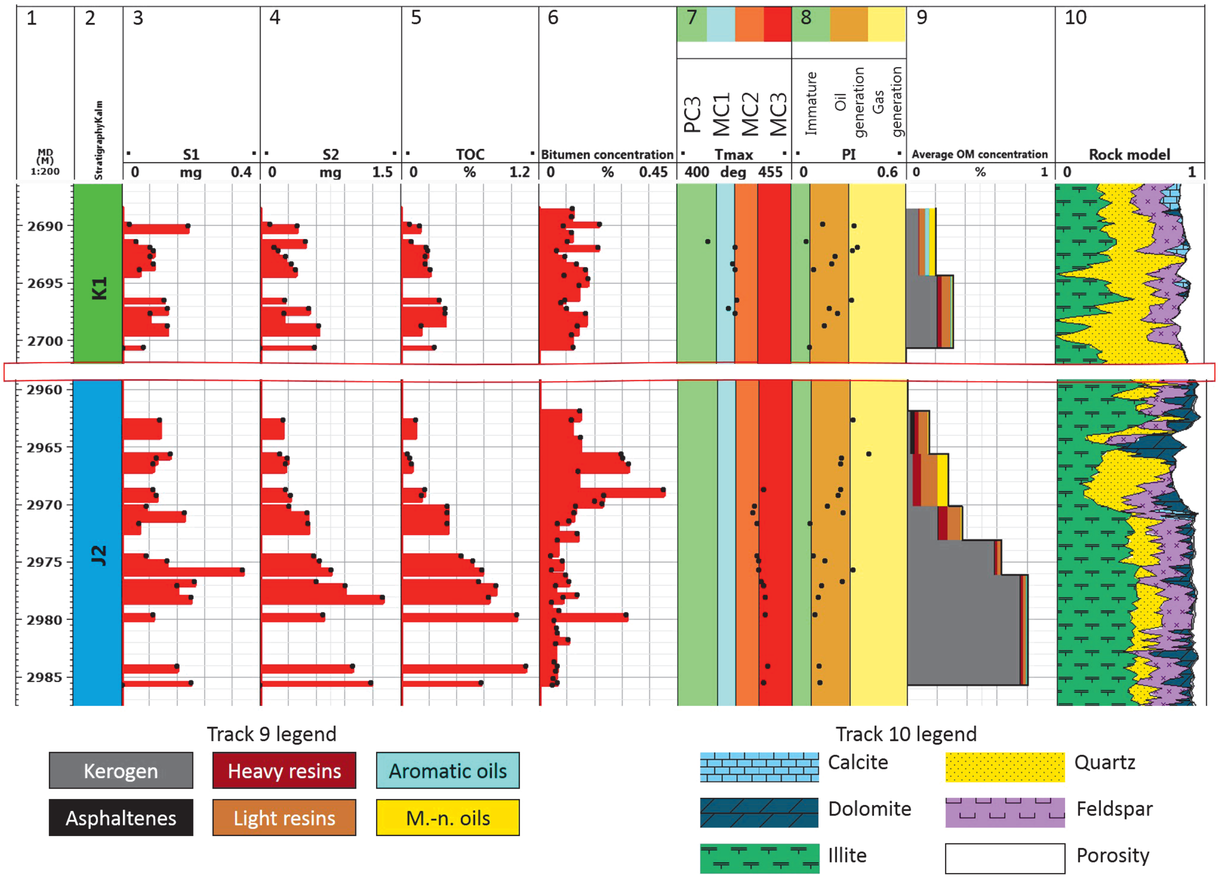

Fig. 8. Layout with results of pyrolytic and bituminological studies and the results of well logging data interpretation for Middle Jurassic and Lower Cretaceous rocks of the East Pre-Caucasian basin

and sandstones with carbonate-clay cement have fairly high values of the pyrolytic production index (PI), which means that the most converted OM is in the rocks with the best reservoir properties compared with clay-siliceous-carbonate rocks. Also, such a high production index indicates that the OM in these rocks is mainly in the mature state (PI > 0.1) (Schmoker, 1981).

Conclusions

The results of pyrolytic and bituminological studies of the deposits indicate that kerogen in the Mesozoic sediments of the East Pre-Caucasian basin is mainly represented by mixed type. The current content of organic carbon varies from fair to poor, the current generation potential of kerogen is poor and level of maturity of OM is characterized by grades from PC_3 to MC_3.

The component composition of bitumen also indicates the degree of maturity of the organic matter. The studied samples of bitumen from the rocks of the East Pre-Caucasian basin are characterized by a relatively low content of asphaltenes (except for some samples of J_2a-b age) and aromatic hydrocarbons. There is a fairly high relative content of light and heavy resins. This allows us to conclude that the degree of maturity of the OM corresponds mainly to the grades MC_2-MC_3.

From the assumption that organic matter is primarily composed of carbon, hydrogen, and oxygen its weight concentration can be calculated as the weight concentration of organic matter (TOM). The TOM parameter for non-extracted samples includes the total concentration of bitumen and kerogen, while the TOM parameter for extracted samples is approximately equal to the concentration of kerogen.

Rocks of Mesozoic sediments in the core selection interval of the East Pre-Caucasian basin are represented by three lithotypes: clay-siliceous-carbonate rocks, clay sandstones and sandstones with carbonate-clay cement. The three lithotypes under consideration are characterized by different relative composition of the components of organic matter and reservoir properties.

Based on the results of gamma-spectrometric and petrophysical studies, it can be assumed that organic matter is associated with clay minerals.

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