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HEAVY CARBON ISOTOPE COMPOSITION OF THE MIOCENE DIATOMITES AND OILS IN THE SOUTH CASPIAN BASIN AS A POSSIBLE GLOBAL PHENOMENON

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Summary. The paper provides an analysis of the results of isotope-geochemical studies of rocks and oils of the Cenozoic-Mesozoic section of the South Caspian Basin (SCB) over the past 30 years. As a result, the previously established fact of the heavy carbon isotope composition (ICC) of organic matter (OM) of Miocene rocks (Diatom Formation), as well as its derivative oils accumulated in the main (Lower Pliocene) reservoir of the SCB, were confirmed. To verify the global nature of this phenomenon, a comparative analysis of isotope-geochemical data on sedimentary basins of 18 countries was carried out. A generalized diagram showing the limits of change in the ICC of kerogen and oils and their average values for various stratigraphic complexes for all considered basins has been constructed. It has been established that this phenomenon is also characteristic of other basins of the world. Based on this, a conclusion was made about the global nature of this phenomenon. The reasons that led to the isotopic shift in the global carbon budget that occurred in the Miocene are not completely understood. The existing ideas about its possible nature are considered. The results once again confirmed the effectiveness of using $\delta^{13}C$ for oil-oil and oil-source rock correlation in combination with other geochemical criteria.

Introduction

The South Caspian basin (SCB) is a large tectonic element of the Earth's crust in the central segment of the Alpine-Himalayan mobile belt, which includes the deepest depression on Earth – the South Caspian Depression (SCD). The SCB is characterized by very rapid Pliocene- Quaternary subsidence and sedimentation (1.2-3.0 km/Ma). The sedimentary fill of the SCB reaches a thickness up to 25 km and is formed by deposits of Middle Jurassic to Quaternary age.

The SCB is among the oldest oil and gas bearing provinces in the world. Oil and gas is produced from reservoirs over a wide stratigraphic range: from the Upper Cretaceous to the Quaternary. The majority of commercial oil reserves, however, are located in the Lower Pliocene – Productive Series (>75%), with the remainder in the Miocene-Paleogene (15%) and the Upper Cretaceous (10%) (Feyzullayev et al., 2001).

Over the past 30 years, a large amount of research has been carried out to study the generation potential of the Mesozoic-Cenozoic rocks from outcrops and wells, as well as the carbon isotope composition (ICC) of reservoir oils. Oil-to-oil and source rocks-to-oil correlations based on carbon isotopic ratios suggest that the Miocene interval (Diatom, Chokrak and Upper Maykop strata) has played the principal role in the formation of commercial oil accumulations in Lower Pliocene – Productive Series (PS) reservoirs (Guliyev et al., 1997; Feyzullayev et al., 2001; Guliyev et al., 2001; Feyzullayev, 2019).

Based on the ICC of organic matter (OM), two groups of rocks were clearly identified in the sedimentary section of the SCB: diatoms and pre-diatoms. OM of diatom rocks, as well as its derivative oils, is characterized by relatively heavier ICC (Fig. 1 and 2).

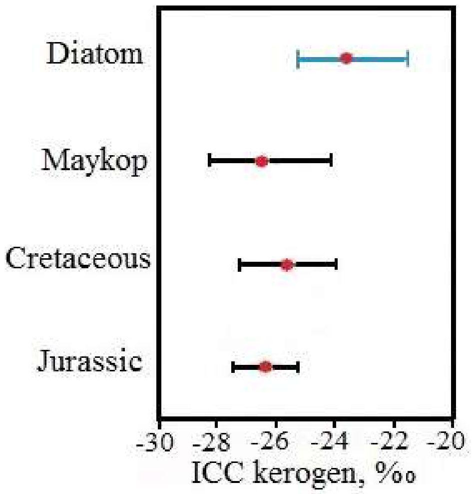

Fig. 1. SCB. Limits of change and average values of $\delta^{13}C$ in kerogen of rocks of different ages (Feyzullayev et al., 2001)

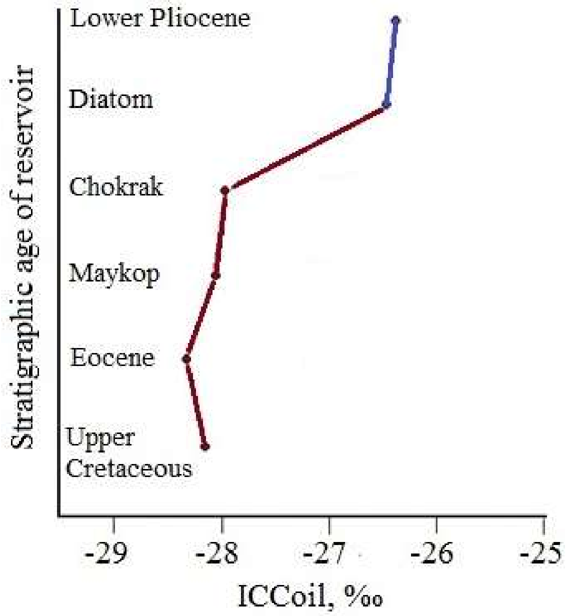

Fig. 2. Average values of carbon isotope ratios in the alkane fraction of oils from different age reservoirs in the South Caspian Basin (Guliyev et al., 2001)

The source of oils of the main (Lower Pliocene) SCB reservoir is mainly the diatomaceous formation (Feyzullayev, Aliyeva, 2003) and therefore they are also characterized by relatively heavy ICC.

The Diatom suite belongs to the Middle-Upper Miocene stratigraphic interval and includes 4 horizons namely: Konk, Karagan, Sarmat and Meotis. This formation is distinguished by a high Total Organic Carbon (TOC) content (on average 4.35 wt.%) with type II-I kerogen (HI up to 770 mg HC/g TOC) and a high generation potential (~3 t. HC/m^2).

As the name of the formation suggests, it consists of more than 50% diatom shells and is represented by loose or cemented siliceous deposits of light gray or yellowish color (Diatomite, 1978; Zhuze, 1973).

The diatomite is a porous rock, light-weight, siliceous, being a result of accumulation and compactions of diatom remains, clay and silt. The diatoms accumulate in areas where the rate of deposition of diatoms frustule is higher than the deposition of other sediments (e.g., Berger, 1970; Barron 1987). Diatomites are of marine, less often freshwater (lake) origin and contain 70-98% silica (Jordan, Stickley, 2010).

Diatomites are prolific hydrocarbon source rocks in many basins worldwide (Schwartz, 1987; Aoyagi, Iijima, 1987; Aoyagi, Omokawa, 1992; Bailey et al., 1996; Bazhenova, 2002; Mayer et al., 2018; Sachsenhofer et al., 2018; Tulan et al., 2020).

Examples include the Miocene Monterey Formation in California (Isaacs and Rullkötter, 2001). Several Miocene diatomaceous deposits are documented in Central Europe. In the Pannonian Basin the Middle Miocene diatom-rich rocks are associated with volcanic activity (Dill et al., 2008).

They are distributed mainly in Paleogene-Neogene and Quaternary geological deposits (Tomkeev, 1986). Diatomites acquire a particularly important significance in the Tertiary period; in Paleogene diatomites, silica is often replaced by pyrite (Maslov, 1974).

The starting discovery of a similar phenomenon (heavy ICC of the Miocene OM and oils) in some other sedimentary basins motivated this study (Kennett, 1986; Chung et al., 1992; Magoon et al., 1995; Lillis et al., 2001; Younes, 2012; Philip, Jarde, 2015).

In this regard, the main objective of this study was to collect, summarize and compare the ICC of kerogen and oils of the Miocene and older rocks/reservoirs of various sedimentary basins of the world. The main purpose is to test the assumption about the global nature of the phenomenon of heavy ICC of OM and oils of the Miocene identified in the SCB.

Analytical database

The data on the carbon isotope composition of OM (kerogen/extract of rocks) and SCB oils used in the paper represent the most complete summary of research results over the past 30 years. For a comparative analysis with other basins of the world, data on Miocene (Diatom Formation) and relatively older Oligocene-Lower Miocene (Maykop Series), Eocene and Cretaceous sediments were used. The amount of this data is reported in Table 1.

Results from other basins in the world are divided into two groups in Table 2 based on their age (Miocene and Pre-Miocene).

Table 1

Number of analyzes ICC of OM and oils in the SCB

Age of oil source and reservoir rocks	Diatom		Maykop		Eocene		Cretaceous	
Analyzed objects	OM	Oil	OM	Oil	OM	Oil	OM	Oil
Number of analyses	31	64	52	29	12	28	14	10

Table 2

Number of analyzes ICC of OM and oils for various basins of the world

Country	**Basins / Province**	**Number of ICC**				**Source**
		Miocene		**Pre-Miocene**		
		OM	**Oil**	**OM**	**Oil**	
Azerbaijan	South Caspian – Kura basin	31	64	78	57	Guliyev et. al., 1997; Guliyev et al., 2001
USA	San Joaquin and Santa Clara basins, California; Pismo, Santa Maria and Hartford basins; Denver and Powder River basins; Gulf of Mexico.	29	152	57	88	Clayton et al., 1992; Magoon et al., 1995; Lillis et al., 2001; Lillis and Magoon, 2007; Peters et al., 2018; Andrusevich et al., 1998; Schouten et al., 1997; Johnson, 1998; Spiker et al., 1988; Komada et al., 2005; Froelich, Robinson, 1988;
Russia	Eastern and Western Siberia basins, Yenisei-Khatanga OGR, Sakhalin, Kamchatka, Crimea-Caucasus region, Caspian depression.		28	77	206	Kontorovich et al., 2011; Leushina et al., 2021; Afanasenkov et al., 2019; Oblasova et al., 2020; Andrusevich et al., 1998; Timoshina, 2005
Canada	Saanich Inlet, British Columbia	30				Johnson, 1998
Norway	Wring Plateau, Nothern Sea.	8			11	Morris, 1976; Andrusevich et al., 1998
Hungary	Pannonian basin	20		4		Körmös et al., 2021; Veto et al., 2016
Poland / Ukraine	Outer and CisCarpathia (Polish-Ukrainian segment)			14	20	Kotarba, Koltun, 2006; Kotarba et al., 2021; Wieclaw et al., 2012
Austria	Alpine foreland basin				41	Gratzer et al., 2011
Lithuania	Baltic Syneclise				12	Zdanaviciute, Bojesen–Koefoed, 1997
Kazakhstan	Aryskum trough				14	Golyshev et al., 2020; Madisheva, 2020
Oman	Salt basin			40		Grosjean et al., 2009
Libya	East Sirte Basin				16	Aboglila, 2010
Guinea	Niger Delta Basin			12	39	Ogbesejana et al., 2020
Egypt	Gulf of Suez		4			Younes, 2012
Turkiye	Gulf of Edremit, northwestern Anatolia	3				Bozcu, 2015
India	Indus Fan (Laxmi basin in the eastern Arabian Sea)	7				Khim et al., 2020
Malaysia	North Sabah Basin	3				Anuar, 1994
TOTAL		**131**	**248**	**282**	**504**	

The Pre-Miocene data presented in Table 2 covers a wide stratigraphic interval spanning from the Oligocene to the Cambrian. These data include the results of the determination of the ICC of both kerogen/extract and oil as a whole, as well as their saturated and aromatic fractions.

Data processing and graphical constructions were performed using standard computer programs.

Results

The South Caspian Basin

The fact of isotope-heavy carbon of OM of Neogene rocks and oils of the SCB in comparison with underlying rocks and reservoirs was first noted in previous studies (Guliyev, Feyzullayev, 1996; Guliyev et al., 1997), and later in the works (Feyzullayev et al., 2001 and Guliyev et al., 2001).

In order to consider this problem from a global scale, the results of a profound analysis of ICC of OM and oils in SCB based on the most complete summary of data are given below.

Figure 3 shows histograms of the distribution of ICC values of OM and oils of Miocene and pre-Miocene rocks and reservoirs, according to which an isotopic difference between these two age groups is obvious with a boundary value of -27.0‰.

Isotopic differences in OM and oils of the two age groups under consideration are clearly reflected in the graphs of the relationship between their saturated and aromatic fractions (Fig. 4).

There is an interesting feature in the change of ICC values with depth: the values of oils from Miocene reservoirs shift upward (mainly above a depth of 3 km) compared to the ICC of OM of Miocene rocks, which is apparently associated with subvertical migration processes (Fig. 5). Due to these processes, the oils of the main reservoir of the SCB (Productive Series), which have an epigenetic nature and are derivatives of predominantly Miocene source rocks, have also a heavy carbon isotope (Guliyev et al., 2001).

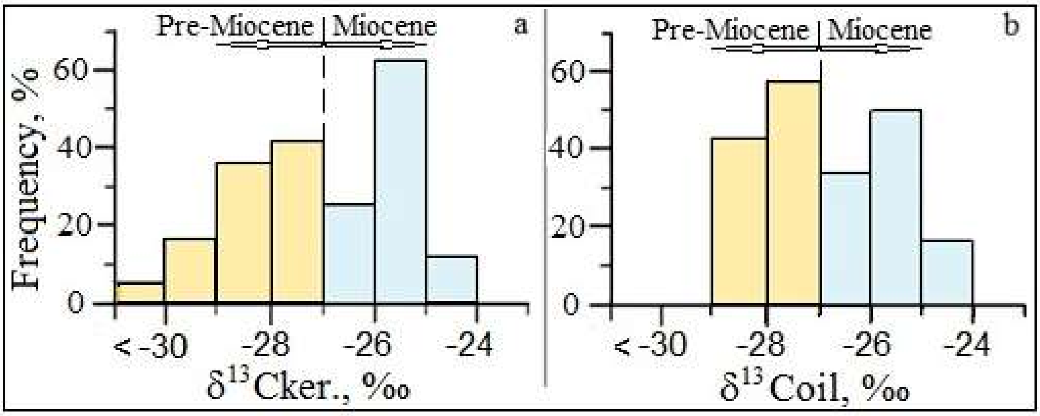

Fig. 3. Distribution of ICC values of Miocene and Pre-Miocene OM (a) and oils (b) in the SCB

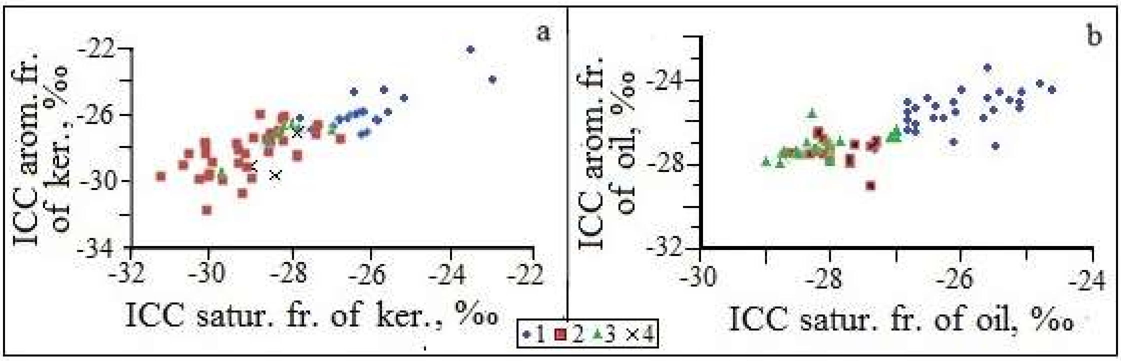

Fig. 4. Graphs showing the relationship between ICC of saturated and aromatic fractions of OM and oils of rocks and reservoirs of different stratigraphic age: 1-Miocene; 2-Maykop; 3-Eocene; 3- Cretaceous

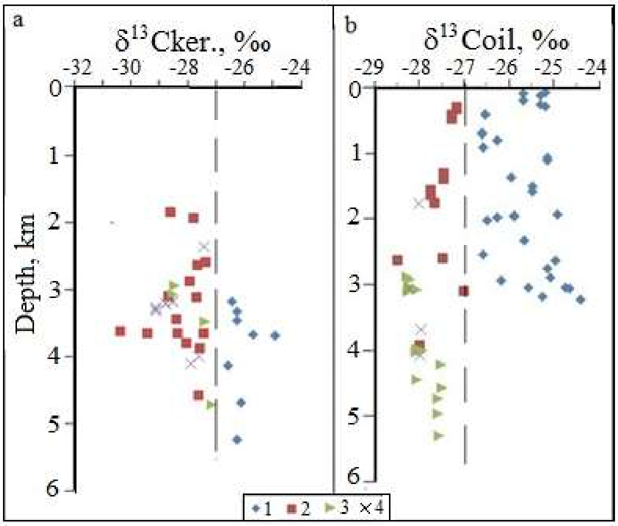

Fig. 5. SCB. ICC values of OM and oils of different stratigraphic units vs. depth: 1-Miocene; 2-Maykop; 3-Eocene; 4-Cretaceouse.

Results for other basins around the world

Before conducting a comparative analysis for all basins of the world as a whole, we will first consider the example of basins in America and Russia, for which (see Table 2), as well as for the SCB, there is a more complete and statistically significant amount of information. All charts below were compiled by the author of this paper based on the sources listed in Table 2.

San Joaquin basin (USA)

Histograms of ICC values of the saturated and aromatic fractions of kerogen in the OM of Miocene and pre-Miocene rocks of the San Joaquin basin show clear isotopic differences between these two age groups (Fig.6). A similar pattern is also observed in the histograms of the ICC values distribution of the saturated and aromatic fractions of oils from the San Joaquin reservoirs (fig. 7). The ICC boundary values between Miocene and Pre-Miocene reservoirs are: for OM – within -26.0/-25.0‰ and for oils – -25.0‰.

Obvious differentiation in the plots of the relationship between carbon isotopes of the saturated and aromatic fractions of kerogen and oils can also be observed in the San Joaquin basin (Fig. 8).

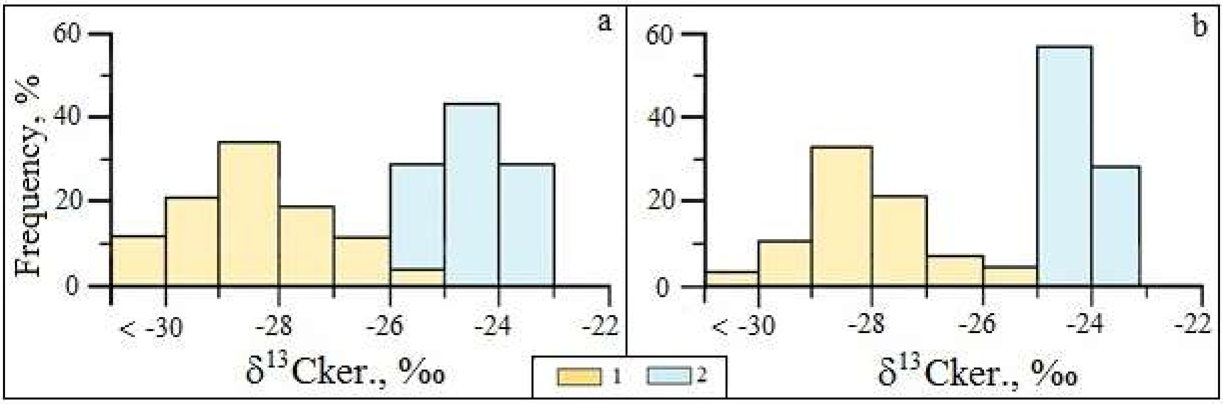

Fig. 6. Histograms of distribution of ICC values of the saturated (a) and aromatic (b) fractions of kerogen in OM of Pre-Miocene (1) and Miocene (2) rocks in San Joaquin basin (USA)

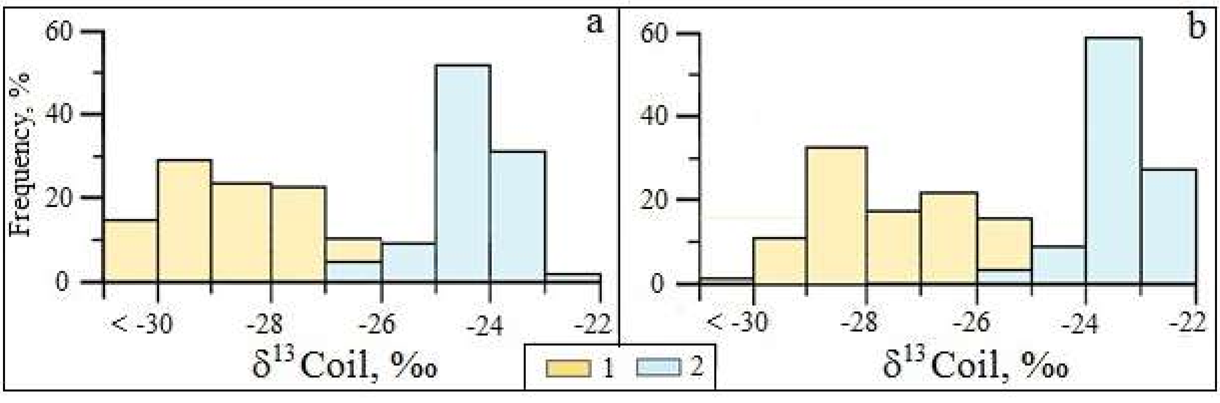

Fig. 7. Histograms of distribution of ICC values of the saturated (a) and aromatic (b) fractions of oils of Pre-Miocene (1) and Miocene (2) reservoirs in San Joaquin basin (USA)

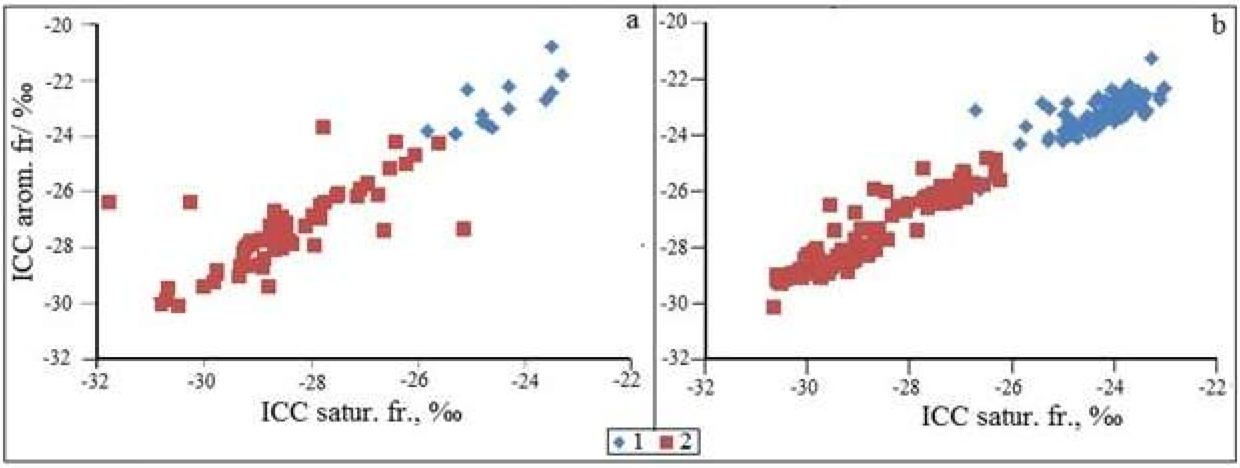

Fig. 8. Graphs of the relationship between ICC of saturated (a) and aromatic (b) fractions of OM and oils of Miocene (1) and Pre-Miocene (2) rocks and reservoirs in San Joaquin basin (USA)

East Siberian basins

For this region, a comparative analysis was performed using ICC for both saturated and aromatic fractions of oils. According to the histograms, although the differences in ICC in fractions of Miocene and Pre-Miocene oils is obvious, the boundary values between these two age groups are less clear, the saturated fraction is situated in the range -29.0/ – -27.0‰, and in the aromatic fraction it lies in the range - 28.0/ – -26.0‰ (Fig. 9). An obvious difference between the two age groups can also be found in the graph of the saturated fraction versus the aromatic fraction of the oil (Fig. 10).

Gulf of Suez province

According to bibliographic sources (Younes, 2012) a differentiation between oils of Miocene and Pre-Miocene reservoirs was also identified in the southern *Gulf of Suez province* (Fig. 11).

Generalization for all considered basins of the world

Based on the data reported in Table 2, a general diagram of the limits of change and average values for ICC of OM and crude oils along the stratigraphic section was compiled (Fig. 12).

Discussion

The problem of identifying of the source of oil is one of the most current in organic geochemistry. The informative value of stable carbon isotopes in solving this problem was indicated in the earliest works of geochemists (for ex., Silverman, Epstein, 1958), which was confirmed by the results of numerous subsequent studies.

The vast majority of geochemists admit that $\delta^{13}C$ values of oils are useful for determining oil-oil and oil-source rock correlation and, in conjunction with other geochemical properties, can indicate the possible age and depositional environment of source rocks.

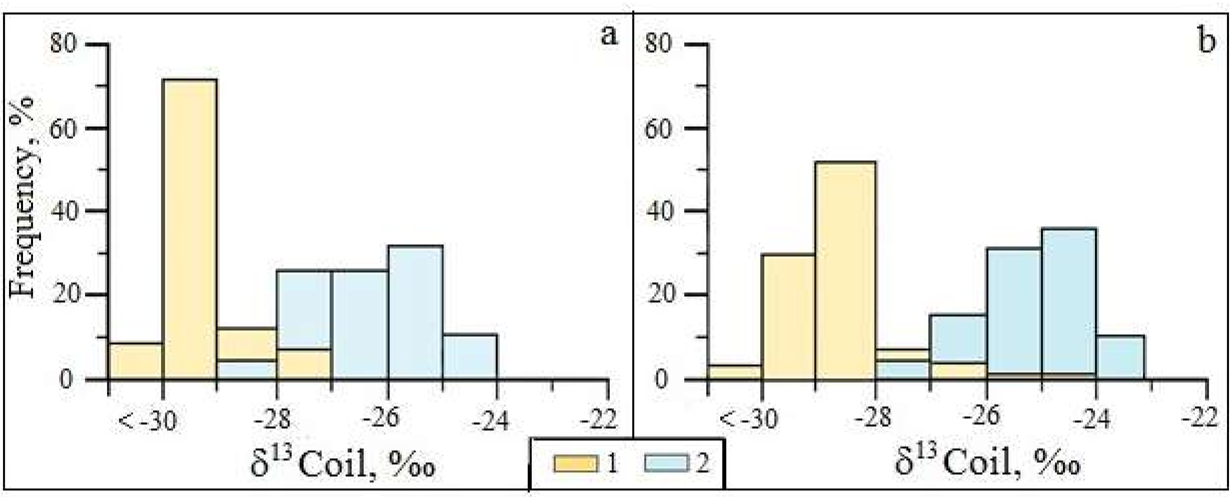

Fig. 9. Histograms of distribution of ICC values of the saturated (a) and aromatic (b) fractions of Pre-Miocene and (1) Miocene (2) reservoir oils in the East Siberian basins

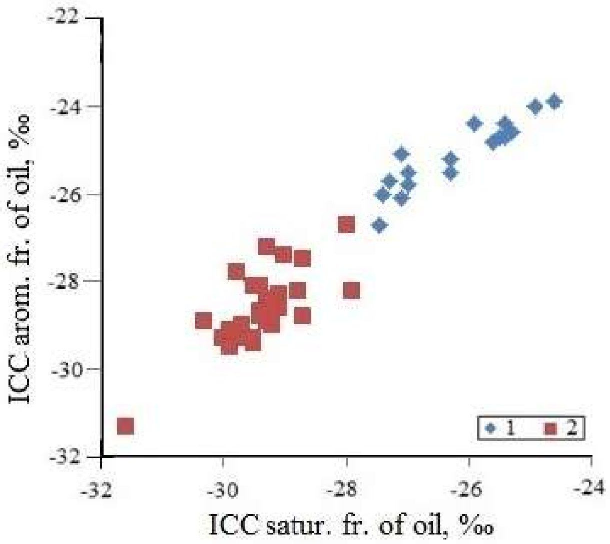

Fig. 10. ICC of saturated vs. aromatic fractions of oils from Miocene (1) and Pre-Miocene (2) resevoirs in the East Siberian basins

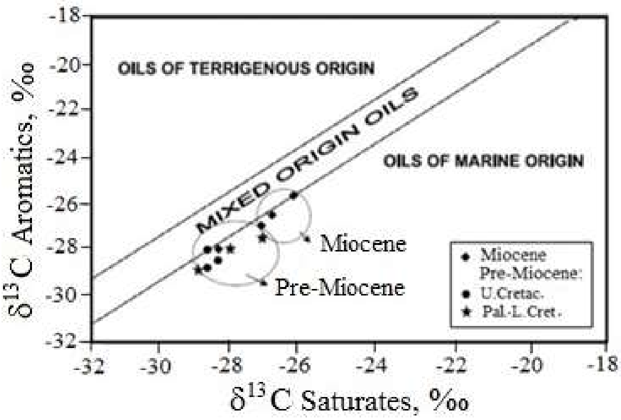

Fig. 11. Relation between ICC of the satureted and aromatic fractions for crude oils from Miocene and Pre-Miocene reservoirs in the southern Gulf of Suez province (Younes, 2012)

The similarities or differences in δ^{13}C values of OM and their derivative oils observed on a global scale are primarily due to the conditions of their formation. During certain geological periods, general conditions for oil and gas formation existed in many regions of the Earth, such as the widespread formation of a heavy carbon isotope composition in the Miocene.

Based on the diagram presented in Figure 12, as well as the results of numerous independent studies of various basins of the world addressing this problem, we conclude supporting the global nature of the phenomenon of heavier carbon in the Miocene.

The reasons that led to the isotopic shift in the global carbon budget that occurred in the Miocene are not completely understood. Numerous hypotheses have been proposed for the cause of such carbon isotope shift.

According to one hypothesis, the nature of this phenomenon is glacioeustatic lowering of sea level (Loutit, Kennett, 1979; Mercer, Sutter, 1982) in the latest Miocene, caused by global regression (Kennett, 1986; Adams et al., 1977; Vail, Hardenbol, 1979).

Chung et al. (1992) believe that Miocene oils are isotopically heavier because of decreased atmospheric CO_2 concentration, which has resulted in decreased isotope fractionation by marine plankton during photosynthesis. However, Mejia et al. (2017) suppose that the role of atmospheric CO_2 in forcing Late Miocene climate remains unclear, with some proxy records suggesting a sharp decrease in CO_2 associated with decreasing extra-tropical Sea surface temperature. Other authors (Pagani et al. 1999; LaRivere et al., 2012) instead suggest a decoupling of CO_2 and global climate during this time interval.

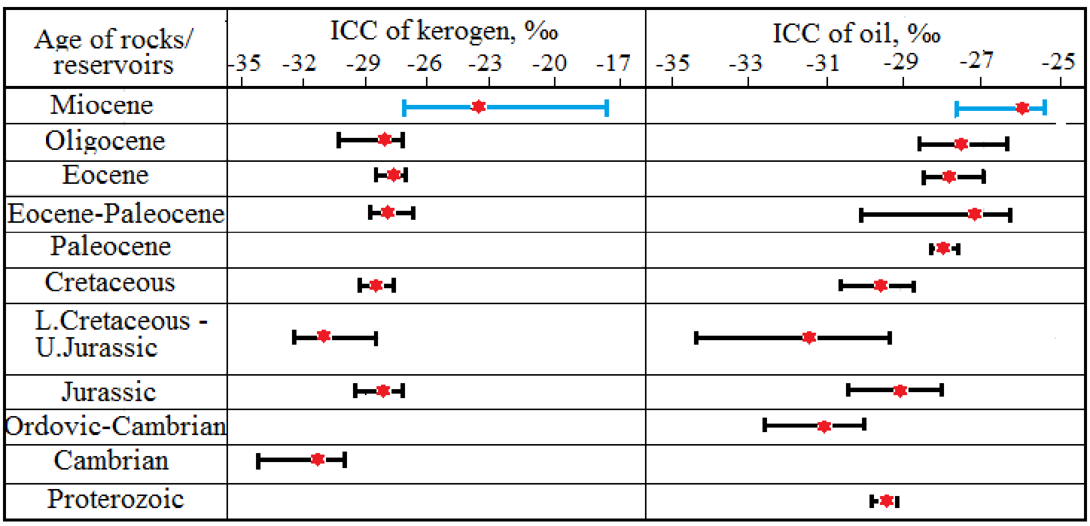

Fig. 12. Generalized diagram showing the limits of change in ICC of kerogen and oils and their average values for various stratigraphic complexes for all the considered basins (The diagram was compiled by the author of the paper based on bibliographic sources given in Table 2)

According to another, most followed model, the Miocene was a period of progressive climatic change, during which the Earth's climate system underwent a remarkable cooling associated with the expansion of the East Antarctic Ice Sheet. This resulted in a marked increase in $\delta^{18}O$ of benthic foraminiferal calcite (Zachos et al., 2001; Miller et al., 1987; Shackleton, Kennett, 1975). Such a climate change may have reduced the amount of water vapor held in the atmosphere, thereby leading to further restrain of moisture source (Li et al., 2014). Middle Miocene cooling was clearly a profound period of climatic change worldwide (Mitchison, 2019).

Conclusion

Based on the collection, generalization and comparative analysis of data gathered from literary sources on the carbon isotope of OM and oils from various basins of the world, as well as the results of own and co-authors researches on the SCB, the following main conclusions can be made:

– the occurrence of isotope-heavy carbon in Miocene OM and oils revealed in the SCB is also characteristic for other basins of the world;

– the global nature of the identified phenomenon is substantiated;

– existing hypotheses about the reasons of this natural appearance are considered;

– the results once again confirm that $\delta^{13}C$ values are useful for oil-oil and oil-source rock correlation in conjunction with other geochemical properties;

– in petroleum exploration, the diatom-bearing sediments can be used as an indicator of the depositional setting.

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ТЯЖЕЛЫЙ ИЗОТОПНЫЙ СОСТАВ УГЛЕРОДА ДИАТОМИТОВ И НЕФТЕЙ МИОЦЕНА В ЮЖНО-КАСПИЙСКОМ БАССЕЙНЕ КАК ВОЗМОЖНОЕ ГЛОБАЛЬНОЕ ЯВЛЕНИЕ

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Резюме. В статье представлен анализ результатов изотопно-геохимических исследований пород и нефтей кайнозойско-мезозойского разреза Южно-Каспийского бассейна (ЮКБ) за последние 30 лет. Подтвержден ранее установленный факт накопления тяжелого изотопного состава углерода (ИСУ) органического вещества (ОВ) миоценовых пород (Диатомовая свита), а также его производных нефтей в главном (нижнеплиоценовом) резервуаре ЮКБ. Для проверки глобального характера этого явления был проведен сравнительный анализ изотопно-геохимических данных по осадочным бассейнам 18 стран. Построена обобщенная диаграмма, показывающая пределы изменения ИСУ керогена и нефтей и их средние значения для различных стратиграфических комплексов всех рассмотренных бассейнов. Установлено, что это явление свойственно и другим бассейнам мира, на основании чего сделан вывод о его глобальном характере. Причины, которые привели к изотопному сдвигу в глобальном балансе углерода, произошедшему в миоцене, до конца не выяснены. Рассмотрены существующие представления о его возможной природе. Полученные результаты еще раз подтвердили эффективность использования геохимического параметра $\delta^{13}C$ для корреляции нефть-порода и нефть-нефть в сочетании с другими геохимическими критериями.

Ключевые слова: *изотопный состав углерода, органическое вещество, нефть, осадочные бассейны, миоцен*

CƏNUBİ XƏZƏR HÖVZƏSİNDƏ MİOSEN DİATOMİTLƏRİNİN VƏ NEFTİNİN AĞIR KARBON İZOTOP TƏRKİBİ MÜMKÜN QLOBAL HADİSƏ KİMİ

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Xülasə. Məqalədə Cənubi Xəzər hövzəsinin (CXH) kaynozoy-mezozoy kəsilişinin süxur və neftlərinin son 30 ildə izotop-geokimyəvi tədqiqatlarının nəticələrinin təhlili təqdim olunur. Miosen süxurlarının (Diatom Formasiyası) üzvi maddələrinin (ÜM) və onun törəməsi olan CXH -nin əsas (Aşağı Pliosen) rezervuarında toplanan neftlərinin əvvəllər müəyyən edilmiş karbon izotop tərkibinin (CİT) ağır olduğu faktı təsdiq edilmişdir. Bu hadisənin qlobal xarakterini yoxlamaq üçün 18 ölkənin çöküntü hövzələrinin izotop-geokimyəvi məlumatlarının müqayisəli təhlili aparılmışdır. Bütün baxılan hövzələrin müxtəlif stratiqrafik komplekslərə aid kerogen və neftin CİT-nin dəyişmə hüdudlarını və onların orta qiymətlərini göstərən ümumiləşdirilmiş diaqram tərtib edilmişdir. Müəyyən edilmişdir ki, bu hadisə dünyanın digər hövzələri üçün də xarakterikdir. Bunun əsasında onun qlobal mahiyyəti haqqında nəticə çıxarılmışdır. Miosendə baş verən qlobal karbon balansında izotop dəyişikliyinə səbəb hələ tam müəyyən edilməməşdir. Onun mümkün mənşəyi haqqında mövcud fikirlər nəzərdən keçirilir. Alınan nəticələr neft-süxur və neft-neft korrelyasiyası üçün $\delta^{13}C$ parametrin, digər geokimyəvi meyarlarla birlikdə, istifadə səmərəliliyi bir daha sübuta gətirilmişdir.

Açar sözlər: *karbonun izotop tərkibi, üzvi maddə, neft, çöküntü hövzələri, Miosen*