REPUBLIC OF AZERBAIJAN

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ABSTRACT

of the dissertation for the degree of Doctor of Science

OIL SHALE OF AZERBAIJAN: FORMATION CONDITION, DISTRIBUTION PATTERNS, GEOCHEMICAL CHARACTERISTICS AND ASSESSMENT OF PROGNOSTIC RESOURCES

Speciality: 2521.01 – "Geology, prospecting and exploration of oil and gas fields"

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INTRODUCTION

Relevance of the topic and degree of elaboration: Azerbaijan has been exporting oil and gas to the world for many years. In addition to the constant demand for hydrocarbon resources on a global scale, indicators related to the dynamics of oil and gas production in the country and the current situation associated with the discovery of new fields require the study of unconventional sources of fuel and energy in the republic and assessing the prospects for their use. From this point of view, oil shale is of particular importance, the existence of which in our country was known back in the 30s of the twentieth century. Oil shale, widely used in the world (e.g. USA, China, Germany, etc.) as a raw material for the production of synthetic oil and gas, heat and electricity, medical goods, construction materials, organic fertilizers and other products, has not yet been used in any industry in Azerbaijan.

The analysis of works published on the study of oil shale in Azerbaijan shows that, apart from the oil shale fields previously discovered, the issue of identifying new promising fields is completely ignored. The study of the conditions for the formation of oil shale is based not on the methodological principles of modern mineralogical and geochemical studies carried out throughout the world, but, at best, on hypotheses arising from the analysis of lithostratigraphic variations. The issue of studying the oil and gas potential of oil shale objects is also not reflected in published literature. However, a number of works published by foreign researchers jointly with local scientists have adequately studied a limited number of oil shale sections. In these researches, the oil shale rocks identified in the sections were studied either under the name shale (paper shale) or argillite.

Object and subject of research: Due to its specific geodynamic-tectonic, geological, mineralogical and geochemical features, in addition to mud volcanoes, oil seeps (including oil-bearing rock outcrops) and rich oil-gas deposits, large-scale evolution of oil shales has also occurred in eastern Azerbaijan. Spatial distribution of oil shales in tectonic zones where mud volcanoes and oil rocks are manifested requires the study of geological and structural-tectonic factors that control their joint formation. In addition, issues such as the study of the mineralogical diversity of such rocks belonging to different geological periods, the factors that lead to their richness in organic matter, and the ability to generate oil or gas are also the subject of current research. The work also reflects issues of practical importance, such as identifying new directions for using oil shale in Azerbaijan and assessing their prognostic resources.

Goals and tasks of the study: The patterns of spatio-temporal distribution of previously known and newly discovered oil shale areas in tectonic zones, the study of mineralogical, geochemical and petrographic features of shale rocks associated with surface outcrops and ejecta of mud volcanoes, the conditions of their formation and oil and gas potential, as well as the calculation of prognostic resources, is the general goal of the work. In this regard, the tasks set for this work are:

1. Analysis of research on the study of oil shale in Azerbaijan and assessment of the current situation;
2. Discovery of new oil shale fields and mapping of the distribution of shale facies in the country;
3. Study of geodynamic, structural-tectonic and geological factors that control the spreading of oil shale in tectonic zones where mud volcanoes and oil manifestations are recorded, and the identification of patterns associated with spatio-temporal distribution;
4. Determining the place of the oil shale of Azerbaijan in the mineralogical and geochemical classification by conducting mineralogical and chemical comparisons with the oil shale of other countries;
5. Study of sedimentological maturation, palaoweathering, protolithic, paleogeodynamic-paleotectonic, paleobasinal, diagenetic and paleoclimatic features of oil shales based on mineralogical and geochemical proxies;
6. Assessment of the role of primary bioproductivity of the paleobasin and organic matter-mineral associations that control the richness of organic matter in oil shales;
7. Determination of the type and thermal maturity of kerogen based on extraction, spectroscopic analyses, thermogravimetric, pyrolytic and petrographic studies, as well as assessment of the ability of Eocene-Miocene oil shale to generate oil or gas hydrocarbons;
8. Studying the prospects for obtaining synthetic hydrocarbons (shale gas and oil) from oil shale in Azerbaijan based on the results of

geological, mineralogical and organo-geochemical studies;

9. In addition to the technological features of oil shales of Azerbaijan, identification of some new areas of use and assessment of prognostic resources.

Research methods. The conducted research includes the analysis of numerous literature materials, methods of solving problems based on modern approaches and methodologies, and comparative analysis. Mineralogical, geochemical, petrographic, bituminological, spectroscopic, thermogravimetric and pyrolytic studies performed on modern analytical instruments such as MiniFlex 600 XRD, S8 TIGER Series 2 WDXRF, Agilent 7700 Series ICP-MS, Carl Zeiss "Microscope GmbH", "Luminoscope Filin", "Bruker TOP SPIN", ALPHA FT-IR", "Jenway 6850 UV/visible", "Cary Eclipse", FTIR "Lumos", "STA449F3 Jupiter", "Rock-Eval 6" and others. To interpret the results obtained, the most common modern approaches to the study of oil shale in the geological literature were widely used, including statistical correlations, indices, formulas, discriminant function diagrams, etc.

Main defended provisions:

1. Patterns of spatio-temporal distribution of oil shale of the Eocene-Miocene period of Eastern Azerbaijan;

2. Formation conditions of oil shale based on complex mineralogical and geochemical studies;

3. Prospects for the production of shale gas and oil by tectonic zones and prognostic resources of oil shales.

Scientific novelty of the research:

1. As a result of long-term field research and exploration, numerous new oil shale areas were discovered and the distribution areas of shale facies in Azerbaijan were mapped;

2. The features of the distribution of oil shales in tectonic zones where mud volcanoes and oil-bearing rocks are recorded were studied, and the pattern of their spread across areas, structures and sections was assessed from a stratigraphic point of view;

3. Based on the study of numerous samples, the mineralogy and geochemistry of oil shales belonging to different oil-gas regions and Eocene-Miocene units were investigated, and their mineralogical and geochemical classification was given. In addition, the mineralogical

maturity, chemical weathering and sources of parent materials, including the role of igneous rocks, volcanic ash and sediments of hydrothermal origin in the formation of the oil shales were studied;

4. Based on geochemical and mineralogical indicators, paleo-geodynamic, paleotectonic, paleobasin and paleoclimatic conditions for the formation of oil shale were reconstructed and, according to the results, spatiotemporal patterns associated with genesis were established;

5. In addition to the original parent compositions, considered typical for the evolution of oil shale, innovations associated with the reconstruction of paleogeodynamic and paleotectonic conditions corresponded to the geodynamic and geotectonic characteristics of the studied region, relating to certain geological periods, as well as volcanisms established in the published literature and the associative relationship between them was assessed;

6. Along with the possible influence of mineralogical diversity, the rates of chemical weathering, the role of other features (the trophic type, depth, salinity, volcanic ash and nutrient availability of paleo-basin, as well as the location of the sediment accumulation area in it and its redox conditions) in the enrichment of oil shale with organic matter and the formation of kerogen types were studied, and specific characteristics for each stratigraphic unit were defined;

7. The study of the bituminous and kerogen composition of organic matter using modern research methods made it possible to study the potential and thermal maturity of oil shale as a source rock, as well as to identify some patterns associated with tectonic zones and geological epochs;

8. Along with the geological and mineralogical research results of the Eocene-Miocene oil shales, taking into account their generation capacity for oil or gas hydrocarbons, the prospects of obtaining shale gas and oil in tectonic zones were evaluated;

9. Some technological features of oil shales by stratigraphic units were studied, proposals were made for their possible use in several new directions of industry and prognostic resources were estimated.

Theoretical and practical significance of the research: The unconventional approach and methods used in the work, as well as the goals and tasks defined for the implementation of the research topic, can be used to study the mineralogical and geochemical characteristics, genesis and oil and gas content of other sedimentary assemblages in Azerbaijan. The results of the study of oil shales belonging to different geological regions and stratigraphic units can be used for comparison and citation purposes in the study of other Eocene-Miocene sedimentary lithotypes in Azerbaijan. The discovery of new areas of oil shale, mapping the distribution zones of shale facies and scientific substantiation of the prospects for using their rich organic and mineral composition in addition to oil and gas, especially for the obtaining of organic luminophores and organic fertilizer will contribute to the development of the oil-gas and other industries of the republic.

Approbation and application: The dissertation used 53 works of the author, including a study of the geology, mineralogy and geochemistry of oil shale sampled from outcrops and ejecta of mud volcanoes, features of their spatio-temporal distribution, genesis, oil and gas potential and prospects for use, 33 of which, including 1 patent, 1 map, 19 articles (6 of which are indexed in WOS and 5 in Scopus) and 12 thesis (7 of them published abroad) directly reflect the scientific results obtained in the work. One important scientific result (For the first time, based on complex mineralogical and geochemical studies of the Eocene oil shales of Azerbaijan, the conditions of their formation, the high potential of shale oil and promising areas (in Central Gobustan) were determined) of the author were discussed at the Expert Council (Colloquium) and the Academic Council of the Institute of Geology and Geophysics of the Ministry of Science and Education of the Azerbaijan Republic and approved in 2023. The results of the work were also presented at a number of prestigious international scientific platforms, as well as discussed at the "6th International Conference of Young Scientists and Students – 2015" in Azerbaijan, the "XIV International Conference "Resource-producing, low-waste and environmental technologies for subsoil development – 2015" in Kyrgyzstan, "Ideas and Innovations in Geosciences" – 2017" in Ukraine, "36th National and 3rd International Congress on Geosciences – 2018" in Tehran, "General Assembly of the European Geosciences Union – 2019" in Austria, etc. The results obtained for the production of an organo-mineral complex based on breccia and oil shale ejecta from mud volcano and its long-term use in growing cotton in laboratory and field conditions have been patented.

Name of the institution where the dissertation work was performed: The work was performed at the Institute of Geology and Geophysics of the Ministry of Science and Education of the Republic of Azerbaijan.

The total volume of the dissertation: The total volume of the work is 429492 characters, including its introduction – 12298, chapter I – 8700, chapter II – 86938, chapter III – 12571, chapter IV – 7919, chapter V – 143801, chapter VI – 131680, chapter VII – 20283 and conclusion - 5302 characters. In addition, 402 references, 93 figures and 25 tables were used in the dissertation.

The author is deeply grateful to his scientific supervisor, Prof. Ad.A.Aliyev who gave him valuable advice in preparing the work, to General director of the Institute of Geology and Geophysics, Academician Ak.A.Alizadeh and Executive Director of the same Institute, Corresponding member of ANAS D.A.Huseynov for the for the conditions they created during many years of field research, conducting relevant analyzes and preparing the dissertation, to employees of the “Department of Mud Volcanism” E.E.Baloglanov and R.V.Akhundov, with whom he conducts joint research in field work, to all employees of the "Center for Collective Use of Analytical Devices and Equipment" and personally to the Head of the Center N.M.Sadigov, to employees of the “Department of Mud Volcanism”, Candidate of Chemical Sciences A.J.Ibadzade and E.S.Samedov, at the same time the employees of the Institute of Petroleum Chemical Processes named after Academician Y.H.Mammadaliyev, in particular, Head of the "Physical and Physical-Chemical Research Department, Prof. R.A.Jafarova and Head of the "Spectroscopic Analysis Laboratory, Associate prof. U.J.Yolchuyeva who provide the necessary support in conducting mineralogical, geochemical and other analyses, as well as "Azerbaijan Science Foundation" and "SOCAR Science Foundation", which provide financial support for research within the framework of the grant projects.

CHAPTER I. OIL SHALES

This chapter presents the main characteristics of oil shale as a valuable fuel, energy and raw material, as well as its geographical distribution areas and resources.

Oil shale was included in the list of new promising energy sources in Resolution No. 33/148 of the UN General Assembly. Rich organo-mineral associations make it possible to use them as raw materials for the production of more than 50 types of products (ichthyol, phenol, electrode coke, mineral wool, U, V, Mo, Ni, Zn, etc.), including synthetic oil and gas and thermal-electric energy [8, s. 44]. As of 2000, about 69% of the world's oil shale was used to produce heat and electricity, 25% for shale oil and gas, and 6% for other products.

A number of authoritative sources, such as the World Energy Resources report[1] published in 2016 by the World Energy Council, mention the discovery of oil shale on all continents, including 33 countries and 600 deposits. Since such information does not reflect the truth, prevailing ideas about shale reserves are contradictory. Because according to our research, oil shale deposits have been identified in at least more than 50 countries of the world. In particular, although there was information about the presence of oil shale in our country at the beginning of the last century, there is no information about the presence of oil shale in Azerbaijan in any of the works dedicated to oil shale and widely cited.

CHAPTER II. OIL SHALES OF AZERBAIJAN

Issues such as a brief historical review and the current state of the study of shale in Azerbaijan, features of their distribution in stratigraphic sedimentary complexes, oil shale in the ejecta of mud volcanoes, as well as patterns of spread of shale facies across tectonic zones and areas, structures and sections are discussed extensively in this chapter.

Until now, the analysis of published literary materials related to

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[1] World Energy Council. World energy resources: [Electronic resource] / London, UK. 2016. URL: https://www.worldenergy.org/assets/images/imported/2016/10/World-Energy-Resources-Full-report-2016.10.03.pdf.

the study of the oil shale of Azerbaijan in various directions allows us to emphasize the importance of work carried out in several stages.

The first stage includes the research carried out (mainly by I.M.Gubkin, V.V.Weber and others) until the 1930s, the second stage - until the beginning of the 1960s (especially by A.J.Sultanov, R.H.Sultanov and A.A.Ali-zadeh), and if we exclude the book written by S.H.Salayev and co-authors in 1989, the next third stage covers the study of oil shales in Azerbaijan with more modern approaches and methods, starting from the beginning of 1960, after a long break, precisely since 2000. In this regard, we can note the research work of some employees of the "Department of Mud Volcanism" of the Institute of Geology of ANAS (now the Institute of Geology and Geophysics of the Ministry of Science and Education of the Republic of Azerbaijan), carried out under the guidance of Prof. Ad.A.Aliyev. Such studies have formed clearer ideas about the significance of oil shale in Azerbaijan and determined the need to study them from the point of view of wider territories and directions in later periods.

According to our analysis, most of the research is aimed at studying the lithostratigraphic characteristics of previously known oil shale sections, assessing some physical and geochemical parameters (density, moisture and organic matter content, etc.) of shale and preliminary calculations of prognostic resources. With the exception of the oil shale formations identified by the above-mentioned researchers, the issue of searching for new perspective areas has not been considered. General opinions realted to the genesis of oil shale are based on hypotheses about the lithostratigraphy of rocks, rather than on the methodological principles of studies conducted on a global scale. The role of geotectonic and stratigraphic control in the distribution of oil shales on the territory of Azerbaijan, as well as the role of paleobioproductivity, mineralogical and other factors in the enrichment of organic matter, has not been studied. The potential of oil shale as source rocks capable of generating oil or gas, especially their prospects in the production of synthetic hydrocarbons, has not been assessed. In evaluating the deep distribution of oil shale and the oil and gas potential of the region, no special attention has been paid to the pieces of oil shale rocks found in the ejecta of mud volcanoes.

According to the results of many years of field research, the number of previously known (70-80 [9, p. 33]) shale outcrops has increased significantly (Fig. 1). The oil shale facies identified only in East Azerbaijan, including the Pre-Caspian-Guba, Shamakhy-Gobustan, Absheron and Pre-Talysh (Jelilabad) oil and gas regions, are associated with the Cretaceous, Middle Eocene, Maikop and Diatom periods [9, p. 33; 25, p. 43].

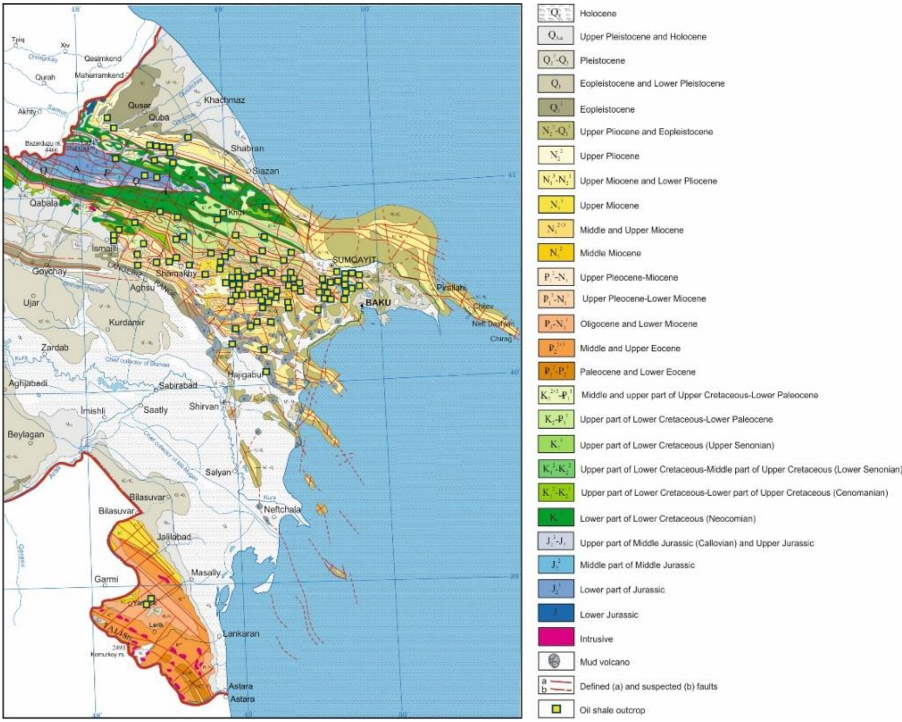

Figure 1. Location map[2] of oil shale outcrops recorded in Azerbaijan [9, p. 33].

Oil shales of the Cretaceous period are of no economic importance since they often occur in the form of small thick layers in

[2] Kengerli, T.N., Ahmedbeyli, F.S., Ismayilzade, A.J. [et al.] Geological map of the Republic of Azerbaijan / 1:750000 / – 2013.

sections recorded in a few local outcrops, and most of the organic part is associated with allochthonous bitumens [22, p. 31; 25, p. 53].

Significant sequences of Eocene oil shale are revealed in outcrops covering a 120 km long southeast-northwest strip starting from the Goytepe area [25, p. 43] of the Absheron peninsula to the Diyalli village of Ismayilli. During field studies, along with some new Eocene oil shale areas (for example, Boyuk Siyeki and Chayli), significant outcrops of oil shale facies belonging to the Lower Maikop sequence were discovered for the first time in Azerbaijan. Although the Lower Maikopian shales are not considered typical of Azerbaijan in the geological literature, the results of our field studies indicate that the oil shales associated with this stratigraphic unit are composed mainly of shales, with some sandiness and clayey-conglomerate rocks, found in the Pre-Caspian-Guba (on the outskirts of Babachay and Agchay rivers), Shamakhy-Gobustan (in the areas of the Lahij streams of Girdimanchay Rivers), Central Gobustan (around Mount Gaibler) and Yardimly (at the confluence of the Vilashchay River streams).

A strip about 128 km long, stretching from Agchala to the villages of Ismailly, located in the upper streams of the Girdimanchay, is characteristic of the distribution of shale facies belonging to the Upper Maykop. In the south and southeast of this strip, stretching from northwest to southeast, there are established oil shale-bearing structures (Shikhzerli, Gaibler, Shaibler and others), which we consider very important. During field work in several mud volcanic areas of Central and Southern Gobustan, as well as in the south of the Uchtepe mud volcano in Western Absheron and other locations, new outcrops of Upper Maykop oil shale were discovered.

Spatial analysis of the Diatom oil shale facies requires special attention to the Upper Sarmatian deposits of the Pre-Caspian-Guba region [19, p. 33; 25, p. 43]. Here, a strip of oil shale facies associated with the Rostov horizon, which starts from the Gilgilchay River and continues to the northwest, to the Gudiyalchay River (even to the Gusarchay-Tahirjalchay watersheds (Fig. 1)), is followed for a long distance. But in comparison, relatively important outcrops are located at a comparatively smaller diapason of that strip, reaching about 30 km, recorded between the Velvelechay and Gudiyalchay rivers [7, p. 21; 11, p. 24; 25, p. 43]. Although the Rostov analogue (Akhudag formation) is established in the central parts of Gobustan, the thickness of the oil shale layers, which is not considered satisfactory, has reduced attention to them.

Oil shales associated with the Konkian (Baygushgaya Formation) and Meotian (Birgut Formation) deposits can be traced (mainly in the same section) in a strip of about 82 km, starting from the northwest of Gobustan and continuing to Western Absheron. In the northwest, they are recorded in a small number of local structures (for example, Jeyirli and Nabur). Except for outcrops in the Yashma region, their occurrences in the north do not attract much attention [11, p. 24; 8, p. 45; 25, p. 43]. Paper shales of Diatom age are also noted in the south of Gobustan.

In Azerbaijan, oil shales are found not only in surface outcrops but also in ejecta of mud volcanoes [10, p. 342; 16, p. 48]. For the first time, Prof. Ad.A. Aliyev paid attention to them and considered it important to study them as oil and gas source rocks. In addition, the study of the deep presence of analogues of oil shale formations observed on the earth's surface and the issue of oil and gas perspectivity require special attention to oil shale found in eject materials of mud volcanoes [10, p. 342; 16, p. 48; 17, p. 17; 18, p. 28; 21, p. 4; 23, p. 32; 26, p. 310].

The results of structural analysis, drone and other complex studies, covering many years after paroxysms and between paroxysms of the mud volcanoes of Azerbaijan, made it possible to establish three zones in the crater zone (central, median and distal) of mud volcano. The zone that we consider characteristic of shale ejecta during eruptions is the median ring, which has a logarithmic spiral morphology [25, p. 45; 33, p. 10]. We consider depth intervals corresponding to the range of 2-6 km to be typical for volcanic eruptions that cause the release of Eocene-Diatom oil shale to the surface [2, p. 53].

Of particular importance for the distribution of oil shale in Eastern Azerbaijan is the Central Gobustan, which is established within the boundaries of the accretionary prism of the Greater Caucasus and the Jeyrankechmez-South Caspian Megabasin [1, p. 13;

14, p. 16; 25, p. 53]. Particularly favourable for the distribution of surface oil shales of the Eocene age were the areas corresponding to the accretionary prism north of the Zengi-Garajuzli fault [25, p. 44]. The anticlines (for example, Sarıdashchay, Altyaghaj, Kurkechidagh, etc.) defined on the southern slope of the Greater Caucasus are characteristic of the distribution of the Cretaceous bituminous shale facies.

With some minor exceptions [7, p. 21], the zones extending to the Great Caucasus Thrust can be considered typical for the distribution of the Eocene oil shale in the north of Eastern Azerbaijan [25, p. 42]. The cores of the troughs (Shikhandag, Chargyshlag, Kichik Siyeki, etc.) are characteristic of the Koun oil shales found in the northern parts of Gobustan [11, p. 28; 14, p. 16; 25, p. 45]. It is characteristic of Eocene oil shales, both in area and in structure, that the lower horizon of the shale section contains thicker and more whole shale layers than the upper. In particular, in the northern troughs, the dominant tendency for an increase in the number of oil shale layers of the upper horizons is manifested in the lower horizon of the south with thicker layers. Compared to numerous troughs, only a few anticlinal structures (Jangichay, Diyalli, etc.) are of greater practical importance in relation to Middle Eocene oil shales [7, p. 19; 25, p. 45]. Signs of bituminous content, identified in the Eocene facies of the Gylynj and Rehim areas of Southern Gobustan, are due to the absorption of allochthonous oil by shale rocks. In addition to central and southern Gobustan (especially Shikhzerli, Dashmerdan, etc.), another indicator of the presence of thick layers of oil shales in the deeper Eocene section is pieces of Eocene oil shale ejecta rocks identified in the eruption zone of the Absheron mud volcanoes.

In our studies, the boundary of the Upper Maikop-aged oil shale facies is limited by the areas along the Gurjuvan and Yukhary Julyan intermediate streams of the Girdimanchay River from the west. To the south of Gobustan, outcrops belonging to Upper Maikop appear up to the zones of distribution of the Cheyildagh and Gylynj structures. In addition to the Kichik Siyeki and Boyuk Siyaki troughs, shale lithofacies found in the south and east of Gibledagh reflect the importance of partial synclines as well as anticlines in Central Gobustan [25, p. 47].

As for Diatom shales, they can be traced in a western direction in the Lengebiz ridge. In East Azerbaijan, the continuation of such shale facies in the southern zones is limited to the Dashmardan-Goturdagh anticline. With the exception of Rostov, the southern Aladash-Ilkhidagh structures can be considered typical for the distribution of shale facies associated with other Diatom formations in the northern zones of Gobustan. The lack of data on oil shale exposures in Central Absheron suggests that only areas within Western Absheron are typical of the eastern limit of the Diatom oil shale facies [11, p. 24]. Compared to the anticlines identified in the north-west of Gobustan (Goredil, Nabur, Bekle and Jeyirli), the most important Diatom shales for this region are associated with troughs located in its center (Boyuk Siyeki, Kichik Siyeki, etc.) [25, p. 47]. In general, although the Diatom section of Gobustan and its eastern regions is dominated by oil shales of Meotian age, the troughs contain oil shales of Konkian age, the thickness of which sometimes also reaches several tens of meters. Diatom shales are recorded in the structures of large anticlines, more typical of the Western Absheron. Although almost no surface outcrops of oil shale have been identified in the structures of mud volcanoes located in its south and southeast, oil shale of Eocene-Miocene age is quite typical for the volcanoes that erupt here. Shale outcrops in Saray, Binegedi, Fatmayi and other areas in the center and north of Western Absheron are located in the intersection zone of tectonic lines. These areas are associated with the structures of mud volcanoes located mainly in the periclinal and some arc parts of anticlinal folds [11, p. 24] and is characterized by a lens-shaped form. There are also numerous oil seeps and oil-bearing outcrops [6, p. 101; 31, p. 1] in these folds. The results of our studies show that the high content of organic matter in Diatom shales located in this zone is enriched with allochthonous hydrocarbons [25, p. 47]. A similar picture is observed for some outcrops of Diatom shales identified in anticlines containing numerous oil-bearing occurrences of the Lengebiz-Alat tectonic zone in Southern Gobustan. On the other hand, since the bituminous shale areas identified in the center and north of Western Absheron are currently used as objects for various purposes, the prospects for their further use are not considered favorable [25, p. 48].

Taking into account the results of many years of field research, as well as our information regarding new oil shale areas, the distribution areas of oil shale facies throughout the country have been identified and mapped [25, p. 44].

CHAPTER III. MINERALOGY OF OIL SHALES OF AZERBAIJAN

In this chapter, for the first time, the mineral nature of oil shale samples taken from previously known and recently discovered outcrops and from mud volcano ejecta has been studied in detail, and their mineralogical classification is given.

In the studied oil shale samples, 21 minerals were identified, representing 6 classes. Most minerals are silicates. Clay minerals found in the samples included montmorillonite, illite, chlorite, kaolinite and clinochlore. Carbonates, sulfides, sulfates, halogens and oxides were also found in the oil shale of Azerbaijan.

According to quantitative distribution, the samples of the Upper Maikop contain more quartz and pyrite than the Eocene ones. Samples from volcanic areas are better saturated with chlorite. Miocene volcanic oil shales of the Absheron contain montmorillonite (~27%), and Eocene samples contain high concentrations of chlorite (>12%). Compared to Maikop, Eocene and Diatom oil shales are clearly enriched in calcite (>10%). Oil shales sampled from outcrops and volcanoes of Absheron contain more jarosite.

The results of the mineralogical classification showed that in the oil shales of Azerbaijan, compared with other countries, brittle minerals constitute a minority, and clay minerals constitute the majority; in general, they correspond to the clayey-siliceous classification. Analysis of Fig. 2 allows us to emphasize the tendency of Eocene samples to the carbonate category, which distinguishes Gobustan as a whole. In particular, some contain more brittle minerals, such as Barnett shale, from which shale oil is extracted.

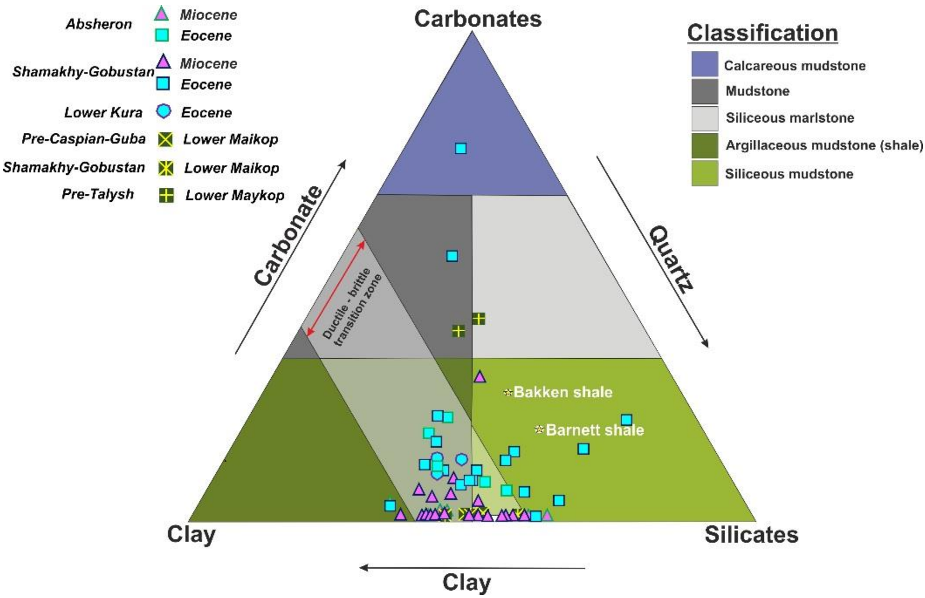

Figure 2. Distribution of samples belonging to different petroleum regions and geological ages in a three-dimensional mineralogical classification diagram.

CHAPTER IV. MAJOR AND TRACE ELEMENTS OF OIL SHALE OF AZERBAİJAN

In this chapter, the macro- and microelement composition of oil shale of different tectonic zones and geological ages is comparatively studied. In addition, classifications related to chemical composition are carried out.

More Si and Al were recorded in the Shamakhy-Gobustan oil shales belonging to the Miocene sequence. This preference for Eocene oil shales is evident in Absheron samples. Along with the Eocene oil shales of Shamakhy-Gobustan, especially the Lower Maikop shales of Talysh, rich in Ca. The distribution trend of Si, Al and Fe in the black oil shales of Lower Maykop along the Pre-Caspian-Guba, Shamakhy-Gobustan and Talysh directions shows an increasing trend from north to south. The richest S in shales was found in Maikop samples. Their relative enrichment in pyrite is also recorded in mineralogical studies.

Analyzes show that with increasing geological age, the concentrations of trace elements such as Zr, Sr, Ni and As in the composition of samples increase significantly. Compared to the Eocene, Miocene samples are characterized predominantly by Ba, V, Mo and to some extent Br, Se, Ga, U. Lower Maikop shales contain more V, Cu, Zn, Mo and Nb, but less Ba, Rb, Cr, Ga and Pb.

Comparison of the macroelements of Azerbaijan and 11 well-studied oil shale countries of the world shows the abundance of Al, Fe, Na, Mg and Ti, and low concentrations of Si, Ca and P in the studied samples.

The results of geochemical classification reveal that the studied samples unambiguously belong to shales [15, p. 32].

V CHAPTER. STUDY OF THE EVOLUTIONARY CHARACTERISTICS OF AZERBAIJAN OIL SHALES BASED ON MINERALOGICAL AND GEOCHEMICAL PROXIES

This chapter examines issues such as mineralogical maturity, paleo-weathering features, sediment sources, as well as paleogeodynamic, paleotectonic, paleobasinal, paleoclimatic conditions and diagenetic mineral characteristics associated with the evolution of Azerbaijan oil shale.

In SEM images, detrital mineral grains have angular edges, indicating poor sorting.

Fe_2O_3/K_2O values indicate an unstable mineralogical nature of oil shales, but comparative analysis shows that the evolution of the Upper Maikop samples is characterized by more distant transport of terrigenous material.

Lower values of the $Al_2O_3/(CaO + MgO + Na_2O + K_2O)$ index calculated for the samples indicate that the major elements of oil shales have stable mobile properties. The indicators of the Upper Maikop samples for this indicator significantly exceed the average value. This means the presence of more immobile elements in the jarosite-rich Maikop shales formed after primary mineralization. The relatively low Mg retention and high Zr content also support this idea.

Due to the presence of terrigenous quartz, a group of Miocene samples belonging to Absheron shows a very close association with graywacke, and samples of Upper Maikop age, mainly belonging to

Shamakhy-Gobustan, show an association with arkose. Only the Vileshchay samples from Lower Maikop are closer to wackes. This indicates that they are associated with parent sediments brought from a short distance. The Eocene samples are mostly grouped in the zone between wacke and arkose. Even a few samples (Chayli, Uchtepe and Pirekeshkul) show a tendency towards litharenites. It is also noteworthy that some Eocene samples exhibit an arkose-related protolith. The Eocene-age Boyuk Siyeki sample is isolated from all samples and shows an association with subarkose, while the Khilmilli sample demonstrates a relationship with K-rich arkose. Lithotypes belonging to all three terrigenous quartz compositions are generally immature. However, since subarkosis is considered closer to maturity, the sharply differentiated genetic character of Boyuk Siyeki samples is of interest.

The ICV values of the studied samples are significantly higher than PAAS, but slightly higher than UCC. The result obtained for this indicator confirms that the oil shale of Azerbaijan is associated with typical rock-forming minerals and has not reached satisfactory mineralogical maturity. However, jarosite and relatively rich quartz-bearing samples of the Upper Maikop, which demonstrate an association with arkose, are distinguished by the presence of ICV values around 1. In general, together with the group of samples of the Upper Maikop, oil shales of the Maikop series (except for samples from Talysh) tend to have lower ratios of this index, indicating that they are more susceptible to mineralogical maturity. Diatom and Eocene samples (Khilmilli, Nardaranakhtarma, Boyuk Siyeki, etc.) are characterized by high ICV values due to the increased content of carbonate minerals.

For the analyzed oil shales, the assessment of chemical weathering due to large-ion lithophile elements probably indicates moderate weathering [29, p. 20]. A decreasing trend in the Rb/Sr ratio was recorded in samples from the Miocene to the Eocene. This means that the Eocene exposed less chemical weathering than the Miocene. Calculations of the PIA and CIA indices also support this idea. The correlation of the quartz-rich Upper Maikop shales with the highest Rb/Sr values also suggests that they were relatively intensively weathered and formed from metamorphic source rocks consisting predominantly of quartz and chlorite (as confirmed by mineralogical composition). In addition, one group of Eocene shales is particularly notable for its relatively low concentrations of CIA, PIA, and Rb/Sr. In the genesis of Eocene and some Miocene (Diatom) shales with this feature, a decisive role is played by loess formations (volcanic ash is not excluded), which are considered to be associated with wind activity and are often lightly compressed from carbonate cement. Thus, mineralogically, the Eocene shales of Absheron, Shamakhy-Gobustan and Southeast Shirvan contain on average >10% calcite (Fig. 2), which provides preliminary information about the role of wind in the transport of parent sediments into the basin.

Analyzes of normalization curves related to PAAS, NASC, Average shale, and UCC indicate that the studied shales are better correlated with Average shale. This means the dominance of clay minerals in the rock, as indicated by the proximity to PAAS. A sharp difference from UCC, but somewhat different from NASC, which is characterized by initial weathering according to CIA, PIA and K/Rb index, confirms that the local shales low-moderately weathered.

In modern studies, three-dimensional diagrams consisting of systems such as A – CN – K are used in order to evaluate the erosion trends of oil shale, and this approach was applied in the present study. In the A – CN – K study (Fig. 3), most Eocene oil shales exhibit binary grouping. Thus, some of them show relatively greater chemical weathering compared to others. In this diagram, only the sample belonging to the Boyuk Siyeki area is grouped into a zone close to the UCC. This characterizes poor chemical weathering. Some Upper Maikop samples taken from the Shamakhy-Gobustan region demonstrate more intense weathering compared to PAAS and NASC. Such samples, more oriented towards peak "A" of the diagram, are distinguished by the predominance of the clay mineral (kaolinite), which characterizes this peak and is subject to the influence of sorting. The oil shale protolith in A – CN – K is characterized by basalt-granite intermediate zones. Secondary transformations of the samples allow to evaluate illitization as a stability stage for mineralization [24, p. 1; 29, p. 20]. The fact that some Miocene and especially Paleogene samples are more shifted towards the A – K junction than comparable average shales indicates the contribution of K-rich ions to their formation. The addition of potassium to the system at the diagenesis stage leads to the transformation of kaolinite into illite or plagioclase into potassium feldspar (Fig. 3). This process, characterized as K-metasomatism, sometimes causes artificially low CIA values. The sharp difference between PIA and CIA values calculated for oil shale indicates the presence of diagenetic potassium enrichment. On the other hand, the results of calculations by $CIA_{corr.}$ show relatively high indicators, in contrast to the CIA values. In general, the A – CN – K results (Fig. 3) and the $CIA_{corr.}$ calculation indicate that authigenic potassium feldspar is relatively progressive for Eocene shales [15, p. 30; 29, p. 20]. However, the process of metasomatism accompanied by illitization of kaolinite is more characteristic for Miocene samples. This is evidenced by a stronger positive correlation of K_2O with Al_2O_3 than with Na_2O [15, p. 31]. This association suggests control of the distribution of Al_2O_3 by potassium-containing minerals, and also indicates a higher role of K-feldspar in shale formation.

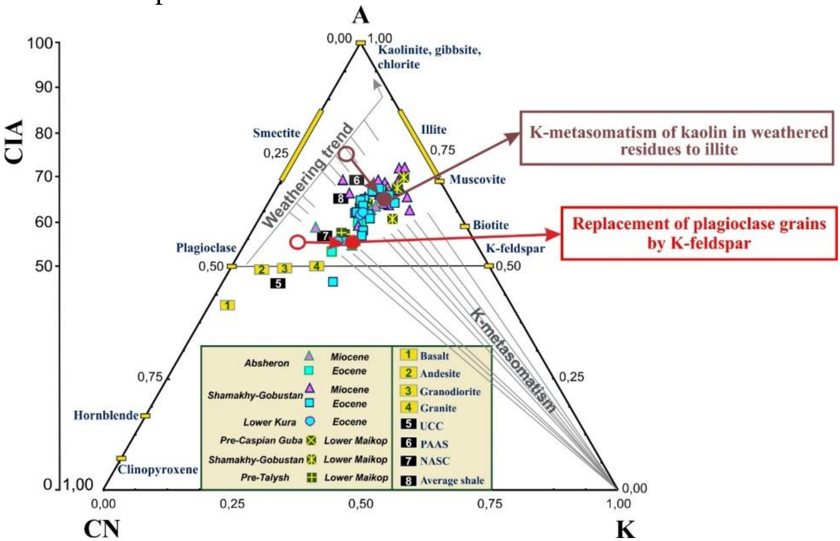

Figure 3. A – CN – K diagram showing the paleoweathering tendency of the studied samples[3].

[3] Fedo, Christopher M. Unraveling the effects of potassium metasomatism in sedimentary rocks and paleosols, with implications for paleoweathering conditions and provenance / Christopher M. Fedo, H. Wayne Nesbitt, Grant M. Young // Geology, – 1995. 23 (10), – p. 921-924.

The result shown in Fig. 4A indicates the contribution of, in particular, ultramafic minerals, basalts and, to some extent, andesites in the formation of Azerbaijan oil shale. Fig. 4B demonstrates the special role of mafic igneous rocks in the evolution of Talysh and Eocene samples. Fig. 4C reflects the influence of mixed complexes with more basalt and less andesite on the formation of the studied shale rocks. The last illustration also reflects the relationship between the jarosite-rich Upper Maikop oil shales and plutonic sources. The sources of these shales may be related to deeply weathered granite gneisses and pre-existing sedimentary rocks. The chemical composition of such rocks follows the pattern $K_2O>Na_2O$, $CaO<1.5\%$ and $Rb\geq Sr$. Our analysis shows that this pattern is typical for the mentioned samples of Upper Maykop. Referring to the mineralogical and geochemical characteristics of some samples belonging to the Upper Maykop, we undoubtedly accept the role of the earliest compositions, probably subjected to repeated sedimentation and derived from granite or quartzose sources belonging to high relief areas. On the other hand, comparing the results of Fig. 3 (displacement of samples towards angle K) with the location of the samples in Fig. 4A, B and C, it is once again clear that although the parent sediments of oil shales were brought from basaltic sources, the plagioclase was subsequently transformed into K-feldspar as a result of mineralogical transformation under the influence of K-ion activity in the diagenetic environment.

Igneous series identification studies have shown a stronger association with high-iron tholeiitic basalts and a weaker association with andesitic tholeiitic sources. At the same time, similar results for some Eocene samples (Otmanbozdagh, Goturdagh, Boyuk Siyeki, Shekikhan, etc.), demonstrating a connection with andesites in the A – FT – M diagram, were confirmed with the basicity index (Fig. 4B). The same feature in A – FT – M is demonstrated by the Upper Maikop shales, which in chemical composition are associated with more acidic igneous rocks compared to other samples (Fig. 4B). In addition to Talysh, the Lower Maikop samples (mainly Guba oil shales) are grouped in the zone of transition from basalt to andesite.

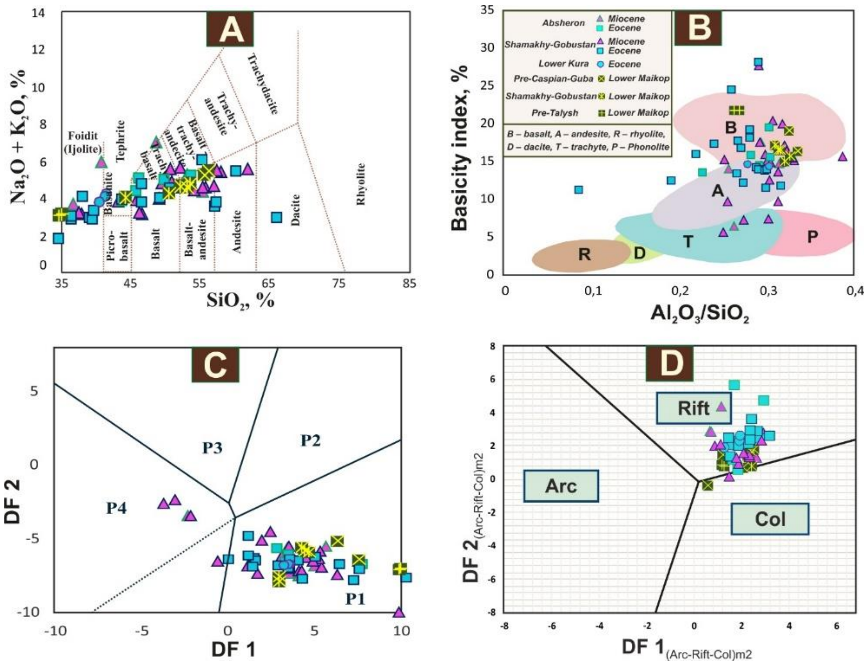

Figure 4. Diagrams showing protoliths of oil shale samples.

According to the Zr/TiO_2 index, most samples contain protoliths associated with intraplate setting. Along with some Eocene samples such as Boyuk Siyeki, Shikhzerli and Nardaranakhtarma, several Miocene (Diatoma) samples show affinity to arc volcanism, including alkaline magmatism.

The estimated (based on Cr and $Cr_{\text{"PAAS"}}$ in the samples) amount of terrigenous materials in oil shales was slightly lower in some Diatom and especially Eocene samples (Chapylmysh, Otmanbozdagh, Shekikhan and Aghtirme, etc.). Along with transition metals such as Cr, V and Ni, the Zr/TiO_2 index, high values of which are considered indicators of felsic magmatic sources, and analyzes based on other approaches also separately support the reliability of volumetric calculations of terrigenous materials. On the other hand, the Boyuk Siyeki sample in Fig. 4A, which in terms of Zr/TiO_2 is associated with acidic igneous rocks and arc volcanism, but also retains zeolitic minerals in its mineralogical composition and shows unsatisfactory paleoweathering (Fig. 3), does not correlate with pyroclastics in any diagram. This sample is one of two samples (the other is Suleyman-akhtarma) with the highest K_2O/Al_2O_3 value among other samples. Such a high concentration of K_2O/Al_2O_3 indicates not only clinoptilolite-rich sediments due to the supply of K during diagenesis but is also an indicator of diagenetically altered epivolcanoclastic (re-sedimentation of previously deposited volcaniclastic) turbidites..

Since sediments associated with hydrothermal sources are characterized by high Fe and Mn (as well as Cr, B, V, Ca, etc.) and low amounts of Al, Ti, and Si, such a regularity unequivocally denies the association of studied oil shales with hydrothermally altered sediments.

In general, protolithic and paleotectonic reconstructions for oil shales show a genetic relationship with tholeiitic basalts, rift-to-collisional transition (Fig. 4D), active continental margin, and pull-apart basin deposition of clasts derived from arc volcanism of the continental margin [28, s. 53]. This is in good agreement with the geodynamic processes that occurred in the tectonic zone (Central Gobustan), corresponding to the accretionary prism, where the studied oil shales demonstrate a spatial distribution. The mentioned processes are related to the rift located between the North Caucasus and South Caucasus-Transcaspian microplates belonging to the front active parts of the Scythian-Turanian plate of the Eurasian continent. This graben, active during periods J1-P2, is called a suboceanic basin consisting of the North Crimea-Greater Caucasus-Kopeh Dagh system[4]. The subduction of the Tufan basin under the Scythian plate in the Jurassic and the movement of the Dubrar trough under the Kakheti-Vendam zone in the Cretaceous caused the formation of island and Andean rift volcanisms in the region in the Jurassic and Cretaceous periods. The closure of this basin in the Upper Eocene triggered the onset of collision in the region[4], as reflected in our paleotectonic reconstruction (Fig. 4D) [28, s. 43]. The characteristics of tholeiitic basalts and the transport features associated with high relief for most of the studied

[4] Rustamov, M.I. Geodynamics and magmatism of the Caspian-Caucasian segment of the Mediterranean belt in the Phanerozoic / M.I.Rustamov. – Baku: “Nafta-Press”, – 2019. – 544 p.

samples reveal the special importance of tholeiitic volcanism that occurred in Tufan in their formation. The fact that the oil shale protoliths are also associated with an andesitic composition (Fig. 4A) indicates the contribution of the Vendam-related Cretaceous volcanism, which was accompanied by activation of the continental margin and intraplate meridional faults on the southern flank of the flysch basin (it was aspid-schist basin in the Lias-Aalean periods of the Jurassic period). After the Cretaceous period, no eruptive volcanism was observed in the accretionary prism zone, where oil shale is more common in Azerbaijan. However, it is considered possible that pyroclastics belonging to the known strong explosive volcanism that occurred in Talysh, Nakhchivan and the Lesser Caucasus in the Lutetian and Bartonian stages, coinciding with the period of the Eocene oil shale formation, were transported by wind into the paleobasin existing in the Middle Eocene shale-bearing regions of East Azerbaijan. In our studies, this idea is not assumed, but is based on an integrative interpretation of the results of mineralogical and geochemical analysis. However, in the formation of the initial mineralogical composition of the rich oil shales of Sarmatian age recorded in Guba, the role of uplifted zones of the Megazone of the Lateral Range of the Greater Caucasus cannot be excluded. On the other hand, beyond the Eocene, the association of some Diatom samples with volcanic ash draws attention to recent collisional volcanisms that occurred during the Upper Sarmatian and Meotian stages in the Lesser Caucasus regions. Recording of high Fe_2O/K_2O and ICV values for oil shales of the Vilashchay area proves the transport of sediments containing rich lithic fragments brought in the Lower Maikop period to the Lerik-Yardimly tectonic bend, where no volcanic sources are recorded, from very close areas. From this point of view, an important fact is that in the axial zone of the Paleocene-Eocene rift (Erdebil-Salavat), along with the calc-alkaline and alkaline magmatic series, tholeiitic volcanism is also widespread.

Our mineralogical studies related to the reconstruction of the sedimentary basin draw attention to the predominance of quartz, which correlates well with the shoreface in many Upper Maikop samples. The occurrence of calcite with relatively high concentrations in

Eocene (as well as some Diatom) oil shales (Fig. 2) gives grounds to characterize the paleobasin conditions in which they formed with a certain degree of salinity. As in the "A – K – F" diagram (Fig. 5A), samples located in the continental-marine transition zone [14, p. 16; 30, p. 157], the evolution of Eocene oil shales shows a closer connection with the influence of the marine environment [30, p. 163]. In particular, the Paleogene oil shales of the Nardaranakhtarma, Khilmilli and Vileshchay areas show a tendency towards the marine zone. Such patterns are very consistent with shale found along the Scandinavian coast. They underwent a very short period of paleo-weathering due to the rapid transport process. The mineral composition of such shales is either absent or contains less than 10% kaolinite, and the fact that kaolinite is not recorded at all in the Naradaranakhtarma and Khilmilli samples strengthens our conclusion about the similarity.

In general, the Mn/Al and Fe/Mn indices show significant differences between the depths of the Paleogene and Miocene basins. Mineralogical and geochemical indicators reflect the association of the studied oil shales with oligotrophic basins, characterized by lower biological productivity than other trophic analogues. In contrast to the Eocene, the evolution of Upper Maikop samples in more peripheral zones of the basin is supported by both mineralogical and geochemical proxies (Fig. 5B).

The results of Fig. 5C show that the Eocene oil shale basin is medium-deep (>20 m), and the Miocene is shallow (<20 m), characterized by weak hydrodynamic activity, belonging to the Central Gobustan, where oil shales are more widespread in Azerbaijan. Diatoma oil shales belong to the Ayazakhtarma, Bayanata and Siyeki areas, showing differences between Miocene samples, indicating that they formed in a deeper environment. We believe that the Paleogene basin deepened from the Lower Kura to the Talysh. Shale areas near the Caspian Sea are associated with deeper paleobasin conditions [12, p. 254]. Oil shales formed in the deepest environments in Azerbaijan belong to the Lower Maikop, in this respect the Agchay and Vilashchay samples show (<60 m) a clear difference (Fig. 5C).

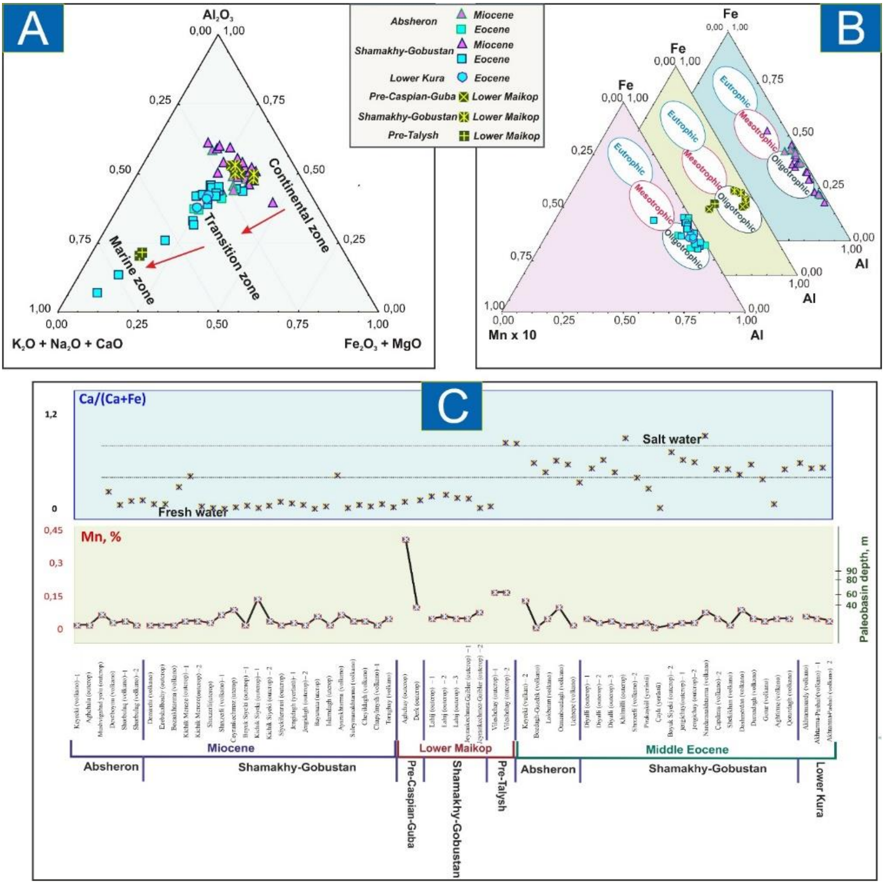

Figure 5. Diagrams showing the evolutionary features of oil shale associated with the paleobasin.

The presence of approximately >5% concentration of calcite from carbonate minerals is an indicator of a brackish basin environment, which means a mixture of fresh and seawater, and a high concentration is a sign of a strong influence of seawater. In addition to the mineralogical indicators (Fig. 2), the results of the diagrams associated with "A – K – F" and Ca/(Ca + Fe) (Fig. 5A and B) revealed Eocene (also Diatom) sediments were deposited in the slightly saline conditions of the paleobasin, which also originates from seawater. The association of such volcanic ash-associated samples with low-salinity paleobasin conditions is further evidence of relatively low transport of volcanic ash into the basin, indicating that the Eocene basin retained some degree of saline water as a result of seawater influence.

The fact that the oligotrophic basin (Fig. 5B), which is known to play a role in the evolution of the studied oil shales, is a relatively open type in terms of the hydrological system, necessitates the formation of a relatively oxygen-rich environment due to new flows. Our paleo-redox studies support this idea. In general, we consider oxygen mineralization, reoxidation, reduction of Mn and Fe oxides, as well as denitrification to be characteristic of the genesis of the studied samples. However, several samples correlate with the depths where sulfate reduction occurred in the anoxic environments belonging to the Upper Maikop [25, p. 50].

The positive results obtained regarding the activity of volcanic ash prove that pyroclastic alteration plays a certain role in the formation of montmorillonite composition of oil shales. This is evidenced by their small size (<1 μm), as well as the filmy to veil-like particles appearance makes them easy to distinguish. The genesis of the Eocene-age Boyuk-Siyeki sample was also characterized by the highest Si content, poor involvement in the chemical weathering process occurring in the source area (Fig. 3), genetic correlation with high alkali content (with dacite) (Fig. 4A) and island-arc volcanism, association with sediments of epivolcanoclastic origin, a trend of K^+ enrichment in diagenetic processes (Fig. 3) and an intense influence of biogenic sedimentation, which differs from other samples by the discovery of high quantities of montmorillonite, calcite, especially tridymite and orthoclase. The formation of montmorillonite as the dominant clay mineral, as well as Si-rich clinoptilolite and tridymite from water-deposited rhyolite or dacite ash, in addition to the alkaline content of ash at low temperature and pressure, also depends on the ability to provide abundant SiO_2 in solution. The fact that we obtained the listed regularities for the Boyuk Siyeki sample demonstrates the correctness of our interpretations based on mineralogical and geochemical proxies. On the other hand, the appearance of authigenic clinoptilolite in the form of automorphic crystals and the presence of organic remains (foraminifera, etc.) in the cavities of this crystal, especially the fact that biogenic opal forms the dominant composition of the rock containing clinoptilolite, confirms the exposure of Azerbaijan oil shales to biogenic sedimentation in the example of Boyuk Siyeki. This is also supported by the assessment of higher Si in the sediment than the normal detrital background and other approaches, in which the Eocene and Diatom samples were more intensively exposed to this process.

All progressive approaches indicate that in addition to the predominance of arid climatic conditions [30, p. 170; 24, p. 1] during the evolution of oil shale in Azerbaijan, there are also significant paleoclimatic differences between the Miocene and Eocene periods. According to our paleoclimatic reconstruction, temperature fluctuations, especially in the Eocene and partly in the Diatom, were higher than in Maikop. Such regularity is in high agreement with the paleotemperature trend that reflects global climate variations determined from isotopic studies[5] of Cenozoic-related seafloor foraminifera.

CHAPTER VI. ORGANIC-GEOCHEMICAL CHARACTERISTICS AND PETROLEUM POTENTIAL OF OIL SHALE SEDIMENTS

Issues such as the role of the primary bioproductivity of the paleobasin and the role of the organomineral association in providing organic matter, as well as the type of kerogen, thermal maturity, hydrocarbon potential according to extraction, spectroscopic, thermogravimetric and pyrolytic analyses, and prospects for obtaining shale gas and oil from Azerbaijan oil shale are reflected in this chapter.

Compared to the Upper Maikop, the evolution of Eocene oil shale under oxygen-rich conditions is likely explained by primary productivity, including oxygenation of the paleobasin through photosynthesis of living organisms. On the other hand, the fact that the Eocene sediments are deposited in the relatively deep middle parts of the basin, and the Upper Maikop sediments in the coastal parts (Fig. 5B and C), characterizes the deposition of the former at a higher rate

[5] Zachos, J. Trends, rhythms, and aberrations in global climate 65 Ma to present / J.Zachos, M.Pagani, L.Sloan [et al.] // Science, – 2001. 292 (5517), – p. 686-693.

and the latter at a slower rate. Also, the greater exposure of samples of Eocene and Diatom age to biogenic sedimentation indicates the bio-productivity of the oil shale paleobasin belonging to these geological periods.

Our petrographic studies revealed that the organic matter in the pore spaces of the Miocene-aged sample is distributed as a discrete assemblage, but in the Eocene shale, it is distributed in a structureless form along the joints of phyllosilicate mineral.

Eocene oil shales contain much more (on average = 19%) organic matter compared to the Maikop sediments, which are considered to have the highest potential as a source rock in Azerbaijan [12, p. 254]. In general, the lowest (average = 5%) content of organic matter was found in solid oil shale samples from Lower Maykop. Although the average amount of organic matter in Miocene oil shales is 11%, in contrast to the Upper Maikop samples, Diatom samples contain much more (about 20%) organic matter.

Providing an Al concentration of about 6-8% in the studied oil shales led to the over-enrichment of the rock with organic matter [25, p. 50]. An increase in Al content to approximately 8-10% due to an increase in the input of terrigenous materials into the paleobasin led to poor concentrations of organic matter in sediments, which is typical for the Upper Maikop oil shale in general. This means that primary paleobioproductivity played a decisive role in providing sediments with organic matter to a high degree in the Eocene and Diatom oil shale formation basin, which was exposed to less terrigenous inputs than Maykop. On the other hand, the recording of tuffaceous sediments in outcrop sections (for example, Diatom age) containing organic matter-rich oil shales [25, p. 50], shows a certain positive relationship between volcanic ash and organic matter enrichment. This can be explained by the positive effect of those acidic magmatic associations rich in nutrients on the growth of algae in water. A comparison of organic matter enrichment with CIA values shows that there is no high correlation between intense chemical weathering and increases in organic matter. This can be explained by the lack of weathering of ash in the source area, which is carried into the basin in the form of pyroclastic deposits.

Our research shows the decisive importance of the role of salinity of paleobasin water, the type of mineral, as well as the specific characteristics of their surface areas and the component composition of organic matter in the relationship between minerals and organic molecules. Using the example of montmorillonite, which is most often found in the oil shale of Azerbaijan, we try to explain the reason for the positive correlation of this mineral with organic matter for the Eocene sample and the negative correlation for the Upper Maykop sample. Due to the increase in salinity in the basin, depending on the electrostatic properties of the mineral surface, the degree of aggregation of montmorillonite increases due to the effect of van der Waals forces. When this mineral associates with larger molecules such as lignin, humic acid and chitin derived from allochthonous organic matter, its aggregation is reduced due to the dominant feature of steric repulsion. Montmorillonite can sorb organic substances of terrigenous origin together with organic substances of aliphatic structure, consisting of smaller molecules, only when this mineral must be Na-based and first absorb fulvic acid, formed from organic matter of allochthonous origin, and then can more strongly absorb organic substances formed from plankton. This is an indicator of the role of planktonic organisms associated with autochthonous organic matter, as well as allochthonous sources brought to the basin by terrigenous flows in the formation of Eocene oil shales. The general result of the issues listed is that the Eocene shales, which are more supplied with organic matter due to their closer connection with the sea (Fig. 5A) and greater exposure to biogenic sedimentation than the Upper Maikop shales, have good sorption of organic matter in terms of montmorillonite, is caused by the fact their evolution in aquatic environments with relatively high salinity (Fig. 5C) and autochthonous organic matter, as well as the composition of montmorillonite, consisted of Na (such montmorillonite simultaneously sorbs organic compounds of both allochthonous and autochthonous origin) [25, p. 50].

According to the extraction results, an average of 1.77% chloroform and 1.36% alcohol-benzene bitumen were found in the samples. This figure is similar to the extraction bitumen found in the famous Kukersite oil shale of Estonia. Our analyses show that in Maikop shales, alcohol-benzene bitumens are superior to chloroform bitumens. However, the opposite trend is revealed for Eocene and Diatom shales. Maykop's distinction is because they formed under dissimilar paleobasin conditions and probably contain different organic molecules and functional groups.

Analysis of the parameters of the ^{1}H NMR spectra of benzene and alcohol-benzene bitumens (Table 1) shows that the degree of aromaticity is significantly lower in bitumens belonging to Diatom and Eocene samples. On the contrary, high values were recorded for paraffin compounds in both fractions.

Table 1

Comparison of average parameters of structural groups calculated from ^{1}H NMR spectra of bitumen extracted from Diatom and Eocene samples.

Samples	Bitumen	H_{ar}	H_{α}	H_{naften}	$H_{paraffin}$	H_{γ}	Aromaticity (fa)
Bayanata (outcrop)	Benzene	Trace	10	9.9	61.4	17.6	Weak
	Alcohol-benzene	3.0	8.4	15.4	45.2	28.0	0.115
Keyreki (volcano)	Benzene	2.0	7.9	13.1	13.1	23.9	0.07
	Alcohol-benzene	2.0	22.7	23	23	16.5	0.07

Note: $\boldsymbol{H_{ar}}$ - hydrogen atoms belonging to aromatic nuclei, $\boldsymbol{H_{\alpha}}$ - CH_3 and CH_2 groups that are in the α position relative to aromatic nuclei, $\boldsymbol{H_{naphthen}}$ - protons belonging to naphthenic structures, $\boldsymbol{H_{paraffin}}$ - CH_2 groups belonging to the alkyl chain, $\boldsymbol{H_{\gamma}}$ – terminal methyl (CH_3) groups.

^{1}H NMR and FTIR results indicate that Eocene and Diatom oil shales contain rich aliphatic compounds of algal and bacterial origin and have relatively long chain bonds. FTIR spectra also show that the compositions of bitumen and kerogen consist of aliphatic, aromatic, alkene compounds and oxygen-containing functional groups. The structure of aliphatic hydrocarbons shows a noticeably high relative content of long-chain methyl and methylene, as well as, to some extent, cycloalkanes. The richness of such compositions is an indicator of a certain periodicity of kerogen in the process of oil formation.

The study of extraction products from Eocene shales with a "PRK 4 lamp" in a UV light filter made it possible to obtain different visual luminescences. Thus, it was found that bitumen extracted from benzene gives a yellow colour, alcohol-benzene - light yellow, and hexane - greenish. Since the composition of the latter is rich in relatively light fractions, the colour of the visual luminescence changed to blue after cleaning them with gamma Al oxide. Obtaining a visual luminescence of various colours from the listed bitumen fractions opens up high prospects for the use of the studied oil shales in terms of producing luminophores, the results of which have no analogues.

Thermogravimetric studies show that shale oil yields from Eocene and Diatom samples are significantly higher than from Upper Maykop. Thus, the oil shales of both samples, containing more extraction bitumen, are subject to maximum losses of organic matter in the temperature range up to 400 °C [5, p. 4]. In the same thermal environment, it was found that Maikop shales exhibit many times less loss, and their significant loss of organic matter is due to an increase in temperature to 600 °C. After the decomposition of the organic part, containing relatively weak C–N, C–S and C–O bonds, corresponding to a temperature range up to 400 °C, a higher heating rate can lead to the decomposition of organic components with strong C–C bonds due to high activation energy requirements.

According to our kinetic calculations on the DTA curve (Fig. 6), recorded depending on the mass loss due to the combustion of organic matter in thermogravimetric studies, the activation energy of the Upper Maikop oil shales is 76.408 kC/mol, the reaction rate constant is 0.049, and the Arrhenius number is 3.21. Our comparisons with oil shales of different types of kerogen from other countries have shown that rocks rich in aromatic compounds and oxygen-containing functional groups have a lower activation energy rate. In this regard, using the example of Eocene and Diatoms oil shales, the kerogens of which are well supplied with compounds containing a highly paraffinic structure (Table 1), require a higher activation energy.

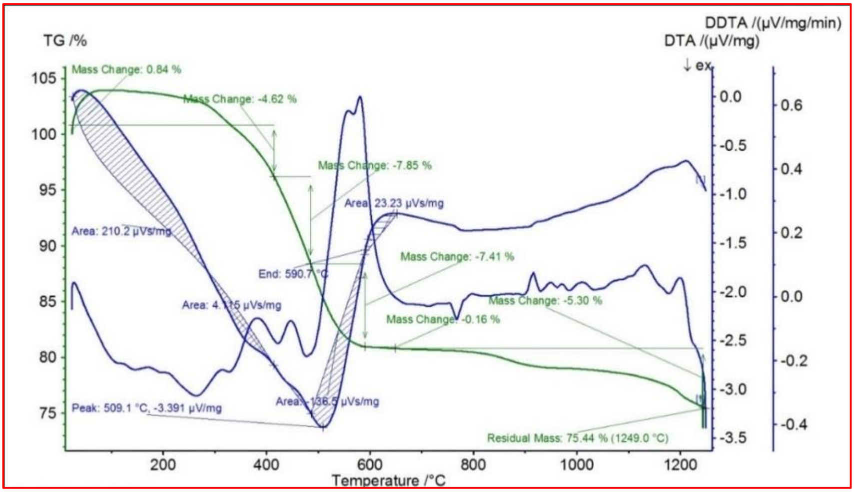

Figure 6. TG/DTA/DDTA spectra of a sample belonging to Upper Maykop.

Based on pyrolytic parameters, our interpretation demonstrates that Eocene and Diatom shales from volcanic ejecta and surface outcrops correspond to type II kerogen. Upper Maikop oil shales correspond to II-III mixed and III types (Fig. 7).

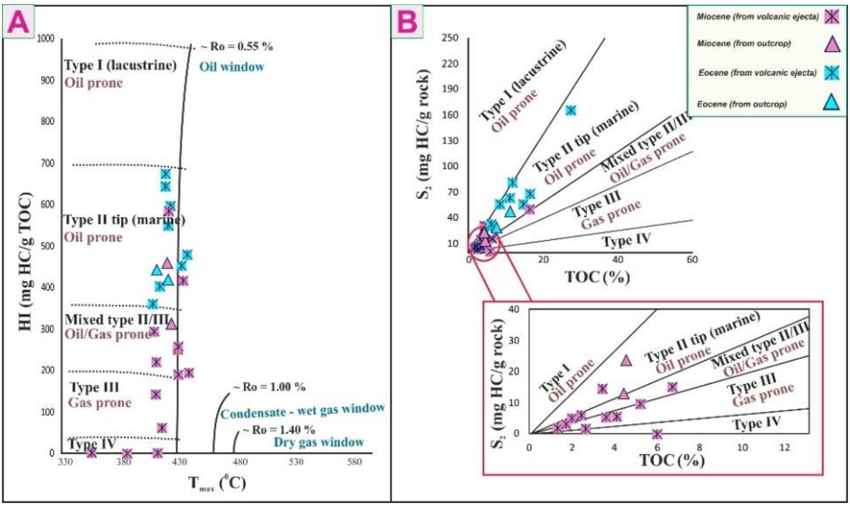

Figure 7. Diagrams showing kerogen types and oil and gas generation characteristics of Eocene-Miocene samples [25, p. 51].

The types of kerogen determined for the organic composition of oil shales are confirmed by the results of petrographic studies. Thus, it has been established that 60-80% of Eocene and Diatom oil shales are composed of alginite, tellalginite and lamalginite, and the rest are macerals of vitrinite and inertinite. Among the main maceral groups in the Upper Maikop oil shales, vitrinite dominates. On the other hand, Maikop samples from Guba region show lower Rb/K values, probably due to an increase in the content of pollen organic matter in the sedimentry basin.

We explain the difference in kerogen types between the Middle Eocene and Upper Maikop oil shales, the role of which in the formation of the rich hydrocarbon resources of Azerbaijan is unconditionally recognized due to different conditions of deposition, based on an integrative analysis of complex research results related to mineralogical, chemical, petrographic and spectral analysis of the studied samples, which are presented in a generalized graphic illustration in Fig. 8.

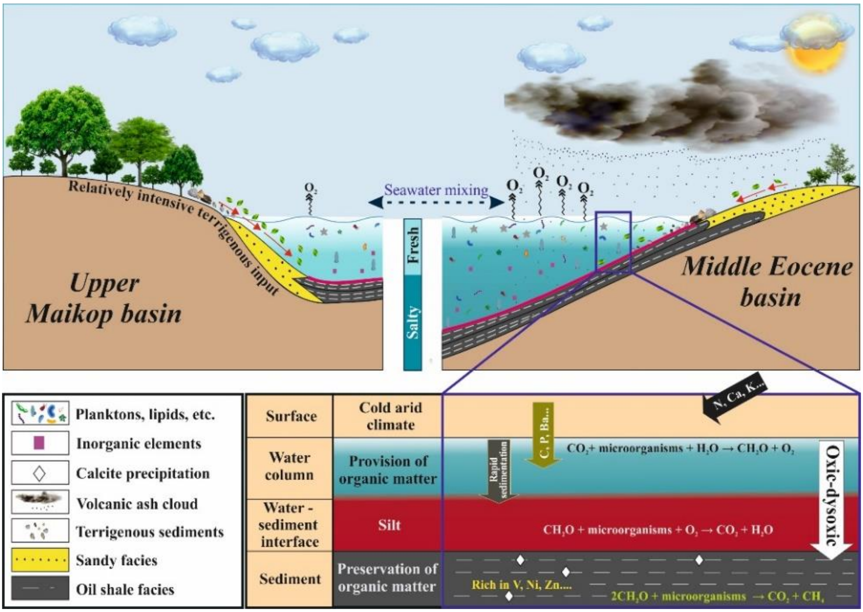

Figure 8. Schematic illustration showing the evolutionary features of Upper Maikop and Eocene oil shales under different paleobasin conditions [25, p. 52].

If we take into account the results obtained in discussions related to the diagenesis of minerals, as well as the temperature intervals required for illitization of smectite (50-200℃) and the value of illite in the mineralogical composition (average = 14.69%), it becomes clear that the studied oil shales have immature thermal nature. This is supported by overlapping ^{1}H NMR and FTIR results, including the fact that the aliphatic hydrocarbon structure of bitumen in oil shale consists of long-chain methyl and methylene (such long chains are characteristic of the early stages of maturity). If we take into account the positive correlation of the immaturity of organic matter with the oxygenated compounds of organic substance, then certain concentrations of functional groups containing C – O and C = O bonds in FTIR studies are also an indicator of insufficient heat supply for the maturity of the oil shales. On the other hand, in comparison, especially in some of the Eocene-aged samples, appreciable retention of chloroform bitumen can be interpreted as closer to the petroleum phase. From this point of view, we consider the degree of maturity of the Toraghay and Otmanbozdagh samples, characterized by medium-high indicators, to be satisfactory. The location of the mentioned volcanoes near the shorelines of the Caspian Sea, as well as the fact that the Eocene sediments in those areas are buried deeper than in the northern zones, can be taken into account as a geological criterion in terms of the correctness of the conclusions made by ensuring the necessary temperature. At the same time, the EPR parameters determined for the Shikhzerli sample, including the density of free radicals, indicate the immaturity of Eocene oil shales belonging to relatively shallow volcanic ejecta in the center of Gobustan. In general, the T_{max} (Fig. 7) and vitrinite values determined for the samples also indicate the immaturity of the Azerbaijan oil shales.

Of particular interest is the fact that some of the samples with the highest thermal maturity belong to the flat-topped volcanic areas around Shamakhy, which do not contain the thick Cenozoic layer characteristic of coastal volcanoes around the Caspian Sea. We believe that the high internal heat source of the mentioned zone, characterized by a relatively small thickness of the sedimentary layer, is related to the intrusives and the presence of large tectonic structures. Our opinion is also confirmed by the identification of a higher temperature gradient for this zone in some published research studies.

Gases in rocks occur in free, adsorbed and dissolved phases. Among them, the formation of synthetic shale gas is characterized mainly by free and adsorbed gases. The total share of such gases in shale rocks can reach about 85%. When the amount of organic matter in the rocks is 3%, it is assumed that the adsorption of shale gas in them has reached a satisfactory level. The presence of adsorbed gas in rock depends on the surface of organic matter and minerals. In this regard, in the present study, in addition to pyrolysis gases [17, vol. II, p. 136], adsorbed gases in Azerbaijan shales were also investigated. Among the gases of adsorption origin for local oil shales, methane dominates. Relatively high concentrations of other analogues of methane are noticeable in Maykop-aged samples taken from Shikhzerli, Boyuk Siyeki and Derk.

In addition to mineralogical diversity and rich organic matter, issues such as the effect of the hydrophilic nature of montmorillonite on methane adsorption were also widely investigated, and in this regard, it was concluded that Azerbaijan oil shales rich in organic matter have high porosity and satisfactory gas sorption capacity.

The fact that type II kerogen has a lower sorption capacity than type III has been attributed to the increased aromaticity of the kerogen.

The mineralogical composition of Eocene and Miocene clay-siliceous shales (Fig. 2) was compared with the shales of the United States and China, whose commercial importance was confirmed from the point of view of shale hydrocarbons. it was found that the quartz + feldspar and clay mineral contents of the local shales are better correlated with the Barnett shales, which are of great importance for the world shale gas system. Especially in terms of brittle minerals, a group of Eocene shales attracts more attention (Fig. 2), which is considered a favourable mineralogical factor for their future exploitation.

If we take into account some parameters (geological-geochemical, etc.) of several well-known shale formations of the USA, whose economic efficiency is confirmed, then, 1) especially due to the quartz mineral and rich organic content, capable of storing free and adsorbed gas, 2) contact with "very good-excellent" source rock, 3) demonstrate high generation potential and 4) predominantly bearing type III kerogen, the Maikop-aged oil shales, and its located intervals by depth, thicknesses of oil shale layers in the shale-containing section [18, p. 28] points to the high prospects of their shale gas potentials [8, p. 47].

The shale oil potential of oil shale is due to their better supply of lipids. Compared to Type I kerogen, Type II kerogen generates more shale oil due to its organic content. This is explained by the active participation of each organic carbon in the removal of hydrocarbons from kerogen and the low residue remains due to the high amount of extracted oil, as well as the possibility of the formation of organic acids in type II kerogens that contain relatively high oxygen. That is why, when thermally affecting the oil shale section (due to the immaturity of oil shale) to obtain oil from Eocene and Diatom deposits, which show some connection with type II kerogen (Fig. 7A) and carbonate minerals (Fig. 2), it is possible to achieve a certain degree of extraction of organic acids and CO_2 along with hydrogen enrichment, which leads to an increase in pressure causing intensified migration. On the other hand, the abundance of transition elements such as Fe and V in the analyzed oil shales is a favourable factor as a catalytic effect in the process of formation of hydrocarbons from organic compounds. Another remarkable fact for storing shale oil and ensuring its movement during operation is that oil shale has a lamellar texture [16, p. 48].

According to the results of thermogravimetric studies, it is possible to use a low-temperature (<600 °C) retort to extract hydrocarbons from Paleogene-Miocene oil shale sections (depth up to 4 km) in-situ conditions. The results of extraction, spectroscopic studies and S1 peak of pyrolysis indicate that Eocene and Diatom shales with a temperature of about 400 °C have significantly better oil generation potential than the Upper Maikop ones.

In terms of producing shale oil, we can mention the oil shale strata of the Eocene period (Diatom can also be considered) of Central Gobustan, located in relatively small depth intervals (up to about 4.5 km), significantly complicated by tectonic faults (a favourable factor for the formation of hydraulic fractures) as primary objects [8, p. 47; 11, p. 28; 12, p. 254, 255; 20, p. 119; 22, p. 40; 32, p. 1]. It is also possible to exploit thick clayey shale layers, containing satisfactory organic matter and alternating in the same section with oil shale layers, which is considered an additional advantage. From the point of view of requiring low costs, due to deep-bed properties, as well as relatively high thermal maturity and hydrocarbon gas storage capabilities, in particular, the northern zones of Gobustan, as well as the Maikop shale regions of Talysh (high calcite reserves are an additional advantage) and Guba, demonstrate satisfactory prospects for shale gas.

CHAPTER VII. TECHNOLOGICAL CHARACTERISTICS AND PROGNOSTIC RESOURCES OF AZERBAIJAN OIL SHALE

In the last chapter, along with the technological characteristics of Azerbaijan oil shales, the prognostic resources were also assessed.

Based on many years of research [1, p. 15; 7, p. 24; 8, p. 46; 11, p. 26] the main technological characteristics of oil shale belonging to different geological regions and ages were comparatively studied. The results show that compared with Maikop, the technological characteristics of Eocene and Diatom shales are better (Table 2).

Table 2

Some technological indicators of Cretaceous-Miocene oil shales of Azerbaijan [8, p. 46; 9, p. 33, 35; 13, p. 15; 19, p. 34]

Area	Geological age	density, g/cm^3	Moisture, %	Ash, %	Heat capacity, MJ/kg
Gilezi	Upper Cretaceous	2.13-2.31	5.7-7.1	74.5-79. 2	5.2-5.7
Diyalli	Middle Eocene	2.0-2.12	2.9-5.1	69.42-76.85	9.8-14.1
Jengichay	Middle Eocene	1.94-2.26	2.4-3.9	66.94-73.8	8.2-10.6
Aghchala	Upper Maikop	1.83-2.01	4.8-5.7	76-83.2	2.82-4.36
Masazyr	Konkian	1.68-1.94	2.4-6.8	70.89-83.06	4.1-6.92
Kamalchay	Sarmatian	1.71-1.84	2.2-3.9	67.21-78.5	4.09-9.89
Bayanata	Meotian	1.69-1.79	3.8-7.0	68.42-82.37	3.88-6.64

Table 2 shows that the ash content (>65%) of local oil shale is similar to Fushun oil shale, which is widely used in cement production. If analyzed from the point of view of stratigraphic age, the Maikop oil shales in Azerbaijan contain more ash. Besides the production of cement and trace elements, the most popular application of oil shale ash is in the fertilizer industry. For the first time, together with chemists, technologists, botanists and agronomists, along with Maikop oil shale (burned), the mineralogical and chemical properties of volcanic mud, cotton stalk and industral waste residues were studied, and new organomineral complexes were obtained from their various ratios. Through several years of repeated laboratory and field testing, an improved fertilizer, the main ingredient of which is oil shale, was applied to cotton plants for three consecutive years, and it was found that in addition to plant growth, high productivity was achieved in every subsequent year [3; 4, p. 63, 64].

In this study, using visual, aerospace, geological, drill hole and sectional data, the parameters of oil shale formations were estimated and the prognostic resources of previously known and newly discovered areas in the P_1 category were calculated. Unlike previous researchers, we did not carry out calculations taking the continuity of shale layers 200 m deep as a standard but taking into account the geological features of the studied region, based on estimates related to the thickness of oil shale facies. As an example of our evaluations related to oil shale storage structures on different stratigraphic units, the calculation of forecast resources was carried out based on the estimated parameters (Fig. 9) of oil shale areas (Uchtepa and Agchala) associated with the limb of the syncline that we identified between Uchtepe and Damlamaca anticlines.

We estimate that only about 60% of the Upper Maikop shale facies contain oil shale. The average density of oil shale is 1920 kg/m^3. Among the listed indicators, the predicted resource, calculated both on the surface and at a depth of 220 m for one limb of the syncline, amounted to 47.4 million tons.

Areas of oil shale that attract attention in terms of large reserves are recorded in Pre-Caspian-Guba. According to our estimates, the participation of oil shale in the Rostov horizon is slightly more than 10%. In the oil and gas region, oil shales of the Lower Maikop are also identified, the outcrops of which attract our attention are located 10-15 km below (to the south) the more productive strip of Sarmatian oil shale located between Velvelechay and Gudiyalchay. The estimated prognostic resource alone of the Sarmatian and Lower Maikop oil shale outcrops (Yerfi area) of that strip is more than 300 million tons.

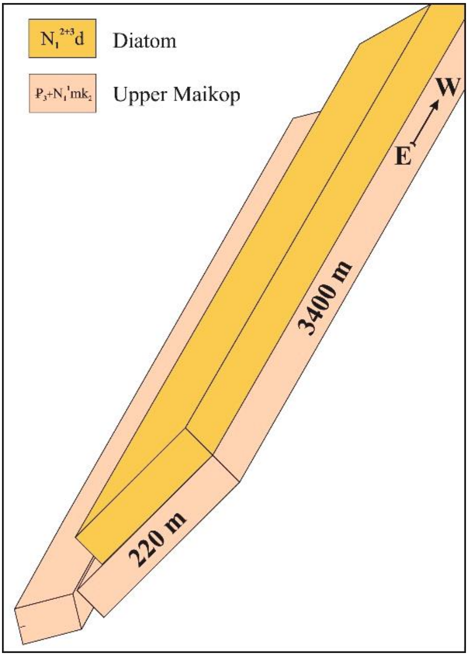

Figure 9. Estimated parameters of the oil shale strata associated with the northern limb of the syncline defined between Uchtepe and Damlamaca anticlines.

As the most important structure in the north-west of Shamakhy-Gobustan, the strip of oil shale-bearing facies in Diyalli has been traced by individual researchers to a distance of 1.5 km, 2.5 and even 6 km, starting from the north-west but our new searches show that there are some oil shale outcrops in the areas between Gurjuvan and Khasydere. This shows that the shale facies strip continues southwestward for at least 20 km. Based on data from outcrops and drilled shallow wells, it has been established that the total participation of shale in the sections averages 8-9%. The total thickness of the shale facies in this zone is 200-250 m. Our calculations, assuming an average oil shale density of 2300 kg/m^3, showed that Diyalli has an inferred resource of approximately 193.2 million tons. However, the proportion of oil shale in the shale facies increases towards the center of Gobustan (on average >25% in Cengichay, but 15% in East

Azerbaijan as a whole).

Our evaluations on Meotian oil shale-bearing sections in the Central Gobustan and Western Absheron show that the participation of oil shale in shale facies is around 20%. The thickness of Meotian reaches 250 m in Northern Gobustan, doubling towards the center. From this point of view, the predicted resources of the km-long shale facies are noteworthy here. The thickness of the Meotian does not reach 200 m in the north-west of the Shamakhy-Gobustan oil and gas region, in the area of Nabur and Jeyirli. The thickness of the shale layers here does not exceed 2 m. From this point of view, the predicted resources of the oil shale areas studied in these territories are significantly less compared to Central and Eastern Gobustan. The important prognostic resource of Konkian, which is involved with Meotian in most sections, is related to synclines, from this point of view, the areas between Boyuk Siyeki-Dostubozu, as well as Pirekesh-kul and Gara-Islam are particularly important.

Estimated resources have only been calculated for about 50% of the more than 100 shale areas identified in Azerbaijan. This is often due to the fact that there are not enough outcrops in the study area. According to our forecasts, the resources concentrated in the areas where we calculate reserves amount to 4-5 billion tons. However, relevant studies to be carried out in the future will lead to a manifold increase in the total reserve.

CONCLUSION

1. The most significant distribution of Middle Eocene-Upper Miocene surface oil shale in Azerbaijan is in the Central Gobustan, established within the boundaries of the accretionary prism of the Greater Caucasus and the Jeyrankechmez-South Caspian Megabasin.
2. The high organic content of the paper shales found in the fault-complicated mud volcano structures of the Western Absheron and Langebiz-Alat tectonic zone was formed due to allochthonous hydrocarbons.

3. A zone of particular importance for the ejection of oil shale rocks during eruption a median ring characterized by strike-slip faults making conjugate logarithmic spirals showing axial symmetry with respect to the emission center.
4. The rich clay, quartz and feldspar content of the samples, which are related to shales in the geochemical classification, led to their correlation with clay-siliceous oil shales in mineralogical classification.
5. A group of Miocene (Upper Maikop) oil shales with a relatively high quartz content, distinguished from other immature samples by their arkosic composition and stable mineralogical nature, demonstrate a more mature sedimentological character compared to the numerous Eocene samples associated with terrigenous quartz of greywacke-litharenite dapozone.
6. Along with weak to moderate chemical weathering, illitization has been established as a stable mineralogical feature of oil shale, the evolution of which also shows some connection with authigenic processes such as K-metasomatism.
7. Volcanic ash brought into the paleobasin by wind has played a certain role in the formation of oil shale samples of Eocene and Diatom samples, showing weak chemical weathering, the lowest Rb/Sr and andesitic protolith features. The protoliths of the oil shales of the accretionary prism zone correlate very well with the Jurassic volcanism of Tufan and the Cretaceous volcanism of Vendam. The role of uplifted zones of the Lateral Ridge Megazone of the Greater Caucasus in the formation of rich oil shale deposits of the Sarmatian in the Guba region cannot be excluded. Protoliths of oil shale sediments known to have been brought from areas very close to the Lerik-Yardimly tectonic bend, where no volcanic sources have been identified, are associated with the tholeiitic magmatic series of the Ardabil-Salavat axis zone. The protoliths of a group of samples from the Upper Maikop that show a genetic relationship with mature passive margins are correlated with sediments brought from granitic sources and subjected to repeated intensive deposition.

8. Compared to Maikop, the paleoclimate genesis conditions of the Eocene and Diatom oil shales are associated with higher temperatures, which is in good agreement with the paleotemperature trend identified for global climate variations associated with the Cenozoic.
9. In contrast to the Upper Maikop, which was subjected to intense terrigenous flows, the remarkable richness in organic matter of the similarly evolved Diatom and Eocene oil shales is associated with the palaeobioproductivity of a relatively deep and saline basin in closer contact with the sea, as well as the irreversible sorption of small-scale organic contents of some minerals.
10. Comprehensive analyzes of extraction, NMR, FTIR, EPR and petrographic analyzes show significantly lower aromaticity but higher paraffinic compounds in bitumens belonging to Diatom and Eocene oil shales compared to Maikop, which is an additional indicator that the latter benefit significantly from organic matter of autochthonous origin.
11. Thermogravimetric studies have shown higher oil yields from Eocene and Diatom oil shales, which lose most of their organic content at lower temperatures (up to 400 °C) than in the Upper Maikop. In such oil shale, high activation energy is required to break strong bonds and increase the effective activity of inactive molecules. However, oil shales, rich in aromatic compounds and oxygen-containing functional groups, have a low degree of activation, which was determined by the activation energy value calculated for the Upper Maykop oil shales.
12. In general, oil shale exhibits immature thermal properties. Eocene and Diatom oil shales have satisfactory oil generation potential, but the Maikop oil shales have higher gas generation potential.
13. Together with the Upper Maikop, predominantly Middle Eocene oil shales belonging to the coastal zones of the Caspian Sea and the northern sides of the Shamakhy-Gobustan region promise certain favourable characteristics for the generation of hydrocarbons according to mineralogical, organo-geochemical criteria, and the depth of their occurrence and the geothermal gradient.
14. The exploitation potential of synthetic shale gas and oil in Azerbaijan is considered quite satisfactory in terms of the burial

depth of shale facies, the effective thickness of shale layers, and their mineralogical and organic-geochemical features. The Maikop oil shales, which primarily contain quartz and Type III kerogen, have shale gas potential, and the Eocene and Diatom oil shales, which preserve more calcite and Type II kerogen, have shale oil potential. The richness of transition elements such as Fe and V is considered to be an additional advantage in terms of catalytic effect in the process of converting organic contents into hydrocarbons.

15. Azerbaijan's oil shale is superior to those containing 70% of the world's oil shale reserves and is considered to be of medium calorific value. Successful results of experiments on crushing oil shale with weak heat-generating ability show that the use of Maikop oil shale, which has a greater prognostic resource than Eocene and Diatom oil shale in Azerbaijan, is also beneficial from a technological point of view.
16. For the first time, a study of the visual luminescent properties of extracted bitumen revealed the importance of using Azerbaijan oil shale in a new direction - obtaining of organic luminophores.
17. For the first time, an organomineral complex prepared on the basis of oil shale and tested in the growing of cotton showed high prospects for the use of Azerbaijan oil shale as an organic fertilizer.

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