

INVESTIGATION OF THE OPTICAL ACTIVITY OF CRUDE OIL FROM THE ABSHERON OIL FIELD

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ABSTRACT

The present study investigates the optical activity of commercial crude oil from the Absheron oil field and its individual distillate fractions. The objective of the study was to establish the relationship between the boiling temperature of petroleum fractions, their hydrocarbon composition, and the magnitude of optical activity. Such information is of practical importance for understanding the chemical nature of crude oil, the distribution of chiral components, and the composition of high-boiling hydrocarbons. Initially, the crude oil was subjected to pretreatment to remove water and mechanical impurities. The purified sample was subsequently fractionated by the displacement method and distilled using a PETRODIST 600 S laboratory distillation unit (Fischer LaboTherm, Germany). Fractionation was carried out over the following boiling ranges: <150 °C, 150–200 °C, 200–260 °C, 260–300 °C, 300–400 °C, 400–450 °C, 450–500 °C, and >500 °C. The resulting narrow fractions were purified from aromatic hydrocarbons and resinous compounds in accordance with GOST 11244–85. Silica gel was used as the adsorbent, and dearomatization was performed by liquid adsorption chromatography using appropriate solvents. The principal physicochemical properties of the purified fractions were subsequently determined. The optical activity of the dearomatized petroleum fractions, including high-boiling fractions with boiling temperatures up to 500 °C, was investigated by polarimetric methods. The results demonstrated a progressive increase in optical rotation with increasing boiling temperature. This behavior indicates an increasing concentration of optically active compounds in the heavier hydrocarbon fractions of crude oil. The established relationship provides additional insight into the distribution of chiral petroleum components and may be useful for the characterization, classification, processing, and geochemical interpretation of crude oils.

Keywords: optical activity; crude oil; Absheron oil field; optical rotation; chiral compounds; enantiomers.

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Introduction

The study of the optical activity of crude oils is of considerable importance from both practical and theoretical perspectives, as it plays a significant role in understanding the origin, geological age, and chemical composition of petroleum [1, 2].

According to recent studies, the optical activity of petroleum is attributed to the unequal formation of R- and S-enantiomers under the influence of various geological and geochemical factors [5]. The optically active compounds present in crude oil generally rotate the plane of polarized light to the right (positive optical rotation, +), with specific rotation values ranging from 1° to 47° [1]. However, petroleum biomarkers such as steranes, hopanes, phytosterols, and sterols, which are considered precursors of petroleum hydrocarbons, are known to rotate the plane of polarized light to the left (negative optical rotation, –). This phenomenon is believed

to result from stereochemical transformations occurring in biomarker molecules during the thermal maturation of crude oil. Nevertheless, cases of negative optical rotation have also been reported for crude oils from equatorial regions, including oils from Java, Borneo, and Argentina [5].

It has been established that the optical activity of crude oil increases with the boiling temperature of its fractions, whereas light fractions exhibit little or no measurable optical activity.

Azerbaijani researchers have also made significant contributions to the investigation of the origin and preservation of the optical activity of crude oils. Their studies demonstrated that Balakhany crude oils contain high concentrations of steranes and hopanes, which play a crucial role in the formation of petroleum optical activity [3].

A. B. Frost and co-workers investigated the optical activity of individual fractions of Balakhany heavy crude oil [1]. Petroleum fractions containing C27 cholestanes, C28 ergostanes, and C29 sitostanes were heated on activated clay and synthetic aluminosilicates. The results showed that com-

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plete racemization did not occur in samples heated either to 20 or to 200 °C, although the magnitude of optical activity decreased significantly.

It has also been demonstrated that the optical activity of crude oil primarily depends on the concentration of steranes and other polycyclic compounds. The optical activity of crude oils from deeper reservoirs generally decreases because of the lower content of polycyclic hydrocarbons in these oils.

In recent years, the investigation of petroleum hydrocarbons has increasingly employed chiroptical spectropolarimetric techniques, including optical rotatory dispersion (ORD) and circular dichroism (CD) spectroscopy, together with ¹H nuclear magnetic resonance (¹H NMR) spectroscopy and gas chromatography–mass spectrometry (GC–MS) [1].

At present, saturated hydrocarbons, as well as alkyl-substituted mono-, bi-, and tricyclic aromatic hydrocarbons in petroleum, have been extensively investigated. In contrast, hybrid aromatic steroids and terpene-containing molecular fragments remain relatively poorly studied. The abundance of these compounds in crude oil is attributed to the oxidative degradation of biological steroids and terpenoids during diagenesis [1, 2].

The investigation of the molecular structures and optical activity of these compounds is of considerable importance for understanding the origin and nature of the optical activity of crude oils [4–14].

It should be emphasized that Azerbaijani scientists, particularly B. A. Suleimanov and his co-workers, have made significant contributions to the investigation of Azerbaijani crude oils, especially in the field of their optical activity and physicochemical properties [15–23].

Experimental part

Crude oil samples obtained from the offshore Absheron oil fields were used as the feedstock for this study. The crude oils were processed according to a common analytical procedure. Initially, they were purified to remove water and mechanical impurities. The purified crude oils were then fractionated by the displacement method, and the resulting fractions were distilled using a PETRODIST 600 S laboratory distillation unit (Fischer LaboTherm, Germany). Fractionation was carried out within the following boiling ranges: below 150 °C, 150–200 °C, 200–260 °C, 260–300 °C, 300–400 °C, 400–450 °C, 450–500 °C, and above 500 °C.

The obtained narrow boiling fractions were purified from aromatic hydrocarbons and resinous compounds using adsorbents and solvents in accordance with GOST 11244–85. Silica gel was employed as the adsorbent. After dearomatization by liquid adsorption chromatography, the physicochemical properties of the purified fractions were determined. The relative density of the individual fractions was measured using a DMA 4500 M digital density meter (Anton Paar, Austria).

Infrared spectra of the narrow fractions were recorded using an ALPHA II FTIR spectrometer (Bruker, Germany) over the spectral range of 4000–650 cm^{−1}. The optical activity of the samples was investigated using both a polarimeter and a spectropolarimeter. Optical rotation was measured with a PerkinElmer Model 140 polarimeter at a wavelength of 1000 nm and with a Polamat A polarimeter at wavelengths of 578, 546, 436, and 406 nm. Circular dichroism (CD) spectra of selected samples were recorded using a SKD-2 spectropolarimeter.

For spectroscopic measurements, the samples were analyzed either in their pure liquid state or as solutions prepared in suitable solvents. Prior to use, all solvents were purified and dried. Liquid alkanes, ethanol, chloroform, and other high-purity solvents were employed. Optical activity measurements were carried out according to the procedure described in Ref. [24].

The specific optical rotation was calculated using the following equation:

$$[\alpha]_{\lambda}^t = \alpha / lc$$

where α is the observed angle of optical rotation (degrees), l is the optical path length of the sample cell (dm), c is the density of the pure liquid sample or the concentration of the solution (g cm^{−3}), t is the measurement temperature, and λ is the wavelength at which the measurement was performed.

The circular dichroism (CD) spectra were obtained in the laboratories of Professor K. A. Kochetkov at the A. N. Nesmeyanov Institute of Organoelement Compounds, Russian Academy of Sciences, and Professor R. G. Kostyanovsky at the N. N. Semenov Institute of Chemical Physics, Russian Academy of Sciences.

Results and discussion

Commercial crude oil from the Absheron oil field was selected as the subject of the present investigation. Prior to the experimental study, the crude oil was characterized by determining its principal physicochemical properties, including density, viscosity, sulfur content, and fractional composition. These parameters were used to evaluate the quality of the crude oil and to provide a basis for interpreting the results of the optical activity measurements.

The physicochemical characteristics of the commercial Absheron crude oil used in this study are presented in table 1.

After the preliminary characterization, the crude oil sample was distilled and separated into narrow fractions with a 50 °C boiling range. The obtained fractions were further separated into aromatic hydrocarbons, naphthenic–paraffinic hydrocarbons, and asphaltene–resinous compounds by liquid adsorption chromatography using various polar and non-polar solvents [25]. Following dearomatization, selected naphthenic–paraffinic fractions were isolated and subjected to further analysis.

Table 1 Physicochemical properties of commercial crude oil from the Absheron oil field	
Parameter	Absheron crude oil
Density, kg/m ³	319.4
Content, wt. %: Resins	15.12
Asphaltenes	0.21
Paraffins	1.09
Sulfur	0.31
Conradson carbon residue (Coking value)	3.64
Pour point, °C	−34
Fraction distilled below 200 °C, wt. %	4
Fraction distilled below 300 °C, wt. %	26

The refractive index parameter of the samples was evaluated using the expression $n_D^{20} - 0.5\rho_4^{20}$, where n is the refractive index and ρ is the density of the sample. The values of the radiation parameter (RP) were calculated according to the equation:

$RP = n_D^{20} - 0.5\rho_4^{20}$ The calculated RP values were analyzed by comparison with the corresponding literature data [23].

The crude oil was distilled into narrow fractions with 50 °C boiling intervals, together with a residue boiling above 500 °C. The yield and density of the obtained fractions of the commercial Absheron crude oil are presented in table 2.

As can be seen from table 2, the density of the petroleum fractions increased with increasing boiling temperature. The density of the 150–200 °C fraction of commercial Absheron crude oil was 803.3 kg/m³, whereas the density of the 450–500 °C fraction reached 944.2 kg/m³. This trend is con-

sistent with the increasing content of high-molecular-weight hydrocarbons in the heavier fractions.

The 50 °C boiling-range fractions obtained from Absheron crude oil were further separated into naphthenic–paraffinic hydrocarbons, aromatic hydrocarbons, and resinous compounds by liquid adsorption chromatography using silica gel as the adsorbent and various polar and non-polar solvents [23]. The hydrocarbon group composition of the obtained fractions is presented in table 3.

The physicochemical properties of the dearomatized 50 °C boiling-range fractions were subsequently determined. The measured parameters included density, refractive index, and other characteristics required for evaluating changes in the composition of the naphthenic–paraffinic fractions after the removal of aromatic and resinous components. The results are summarized in table 4.

As shown in table 4, both the refractive index (n_D^{20}) and the density of the dearomatized petroleum fractions increased with increasing boiling temperature.

The infrared (FTIR) spectra of the 50 °C boiling-range fractions were recorded and analyzed. The FTIR spectra of all fractions distilled within the 150–450 °C boiling range exhibited characteristic absorption bands corresponding to the principal structural groups present in petroleum hydrocarbons. The absorption band at 721–722 cm⁻¹ was assigned to the rocking vibrations of the methylene (CH₂) groups. The deformation vibrations of the methylene C–H bonds (δ CH₂) appeared at 1458–1459 cm⁻¹, whereas the deformation vibrations of the methyl groups (δ CH₃) were observed at 1376 cm⁻¹. The stretching vibrations of the C–H bonds in methyl and methylene groups (ν CH₃ and ν CH₂) produced characteristic absorption bands at 2951, 2921, and 2853 cm⁻¹, respectively. Strong absorption bands in the 3000–2800 cm⁻¹ region correspond to the stretching vibrations of C–H bonds in methyl (CH₃), methylene (CH₂), and methine (CH) groups of aliphatic hydrocarbons.

The stretching vibrations of aromatic C–H bonds (=C–H) in condensed and other aromatic compounds produced an absorption band at approximately 3180 cm⁻¹. In addition, the FTIR spectra contained absorption bands corresponding to heteroatom-containing functional groups, including C–N and C–S bonds. The N–H stretching vibration (ν N–H) of amino groups (NH and NH₂) appeared as a medium-intensity band near 3250 cm⁻¹, while the C–N stretching vibration was observed at approximately 1230 cm⁻¹. The C–S stretching vibration (ν C–S) produced an absorption band near 735 cm⁻¹. The O–H stretching vibration of hydroxyl groups was detected as a medium-intensity band around 3490 cm⁻¹.

Changes in the chemical composition of the petroleum fractions with increasing boiling temperature were also reflected in their FTIR spectra. As the boiling temperature increased, the intensity of absorption bands associated with higher-molecular-weight compounds became more pronounced. Following dearomatization, the absorption bands characteristic of aromatic hydrocarbons disappeared almost completely, whereas the intensity of the bands assigned to naphthenic hydrocarbons increased. The heaviest fractions were characterized by the highest content of naphthenic–paraffinic hydrocarbons, indicating an enrichment of saturated cyclic and aliphatic hydrocarbons after the removal of aromatic constituents.

In the present study, the optical activity of the narrow dearomatized naphthenic–paraffinic hydrocarbon fractions

Table 2
Yield and physicochemical properties of the fractions of commercial Absheron crude oil

№	Boiling range, °C	Yield, wt. %	Density, kg/m ³
I	150-200	5.09	803.3
II	200-260	9.28	849.8
III	260-300	7.26	885
IV	300-400	12.66	904
V	400-450	16.00	932.3
VI	450-500	10.63	944.2
VII	>500	35.86	–

Table 3
Hydrocarbon group composition of the 50 °C boiling-range fractions of commercial Absheron crude oil

№	Boiling range, °C	Hydrocarbon composition, wt. %		
		Paraffinic–naphthenic	Aromatic	Resins
I	150-200	88.5	11.5	–
II	200-260	87.2	12.8	–
III	260-300	86.5	13.5	–
IV	300-400	79.6	18.4	–
V	400-450	64.7	12.0	10.6
VI	450-500	55.3	15.1	19.4
VII	>500	41.0	23.0	23.2

Table 4
Physicochemical properties of the dearomatized 50 °C boiling-range fractions of commercial Absheron crude oil

№	Boiling range, °C	Relative density, d_D^{20} , kg/m ³	Refractive index, n_D^{20}
I	150-200	810.2	1.4497
II	200-260	818.1	1.4506
III	260-300	828.1	1.4615
IV	300-400	849.2	1.4695
V	400-450	871.0	1.4792
VI	450-500	881.3	1.4850
VII	>500	899.2	1.4897

obtained from commercial Absheron crude oil was investigated. Prior to analysis, each sample was dissolved in petroleum ether (a light hydrocarbon fraction consisting predominantly of pentanes and hexanes) to prepare solutions suitable for optical activity measurements. The optical rotation of the prepared solutions was subsequently determined using polarimetric techniques under the experimental conditions described above.

The results of the optical activity measurements for the narrow naphthenic–paraffinic fractions of commercial Absheron crude oil are summarized in table 5.

As shown in table 5, the lightest naphthenic–paraffinic hydrocarbon fraction (Fraction I) exhibited no measurable optical activity. This behavior can be attributed to the absence, or extremely low concentration, of chiral molecules containing asymmetric carbon centers in this fraction. In contrast, the optical activity of the remaining fractions increased progressively with increasing boiling temperature. A corresponding increase in the angle of optical rotation was observed for the higher-boiling fractions.

It should be noted that similar results were previously obtained for the total non-dearomatized petroleum fractions. Comparative analysis demonstrated that the naphthenic–paraffinic fractions exhibited higher optical activity than the corresponding unfractionated samples. This observation can be explained by the fact that the principal carriers of optical activity are polycyclic and steroidal compounds, including petroleum biomarkers such as steranes, terpanes, hopanes, and diterpenoids, which are predominantly concentrated in the naphthenic–paraffinic fraction. During geological maturation, these compounds are gradually transformed into structurally simpler hydrocarbons, such as isoprenoids and other alkanes, resulting in a corresponding decrease in optical activity.

As shown in table 5, all investigated fractions, with the exception of the lightest fraction, rotated the plane of polarized light to the right, exhibiting only positive (+) values of optical rotation. Furthermore, the specific optical rotation increased with increasing boiling temperature of the fractions.

The measurements also demonstrated a pronounced wavelength dependence of optical rotation. For example, the specific optical rotation of Fraction II (boiling range 200–250 °C) was +1.95° at a wavelength of 406 nm, whereas the corresponding value measured at the standard sodium D-line (589.3 nm) was only +0.65°. These results indicate that the magnitude of optical rotation increases as the measurement

Table 5
Optical activity parameters of the narrow naphthenic–paraffinic hydrocarbon fractions of commercial Absheron crude oil

№	Boiling range, °C	Wavelength, nm $[\alpha]_D^{20}$, Optical rotation angle, °				
		$[\alpha]_D^{20}$	578	546	436	406
I	150-200	0	0	0	0	0
II	200-260	+ 0.65	+ 0.79	+ 0.90	+ 1.15	+ 1.95
III	260-300	+ 0.89	+ 0.92	+ 1.03	+ 1.41	+ 2.21
IV	300-400	+ 0.91	+ 1.04	+ 1.33	+ 1.53	+ 2.31
V	400-450	+ 2.02	+ 1.89	+ 2.32	+ 2.64	+ 3.16
VI	450-500	+ 2.10	+ 2.38	+ 2.49	+ 2.96	+ 3.29
VII	>500	–	–	–	–	–

Note: The >500 °C fraction was highly colored, which prevented accurate measurement of its optical activity by polarimetry

wavelength decreases, which is characteristic of optical rotatory dispersion (ORD) in optically active petroleum components.

In addition, circular dichroism (CD) spectra were recorded for selected narrow fractions. The spectra were obtained using an SKD-2 dichrograph (Institute of Molecular Biology and the Institute of Chemical Physics, Russian Academy of Sciences) over the wavelength range of 250–600 nm using a 1 cm quartz cuvette. The instrument was calibrated with D-camphorsulfonic acid as the reference standard. The samples were analyzed as solutions in toluene with a concentration of approximately 1×10^{-2} g mL⁻¹.

According to the available literature, optically active organic compounds present in crude oil have only rarely been investigated by optical rotatory dispersion (ORD) and circular dichroism (CD) spectroscopy. Nevertheless, CD spectroscopy represents a powerful analytical technique for determining the absolute configuration and chiroptical properties of petroleum-derived organic compounds. The experimental results revealed a positive Cotton effect in the wavelength region of 350–355 nm, where an intense CD band coincided with the absorption maximum. The presence of this Cotton effect indicates that the investigated fractions contain chromophoric groups, such as carbonyl (C=O) functionalities, conjugated unsaturated systems, and heteroatom-containing hydrocarbons, which exhibit strong electronic absorption in the 300–350 nm spectral region.

Conclusions

1. The optical activity of individual 50 °C boiling-range fractions of commercial Absheron crude oil was investigated. It was established that all isolated narrow fractions, with the exception of the 150–200 °C fraction, rotated the plane of polarized light exclusively to the right (positive optical rotation). The magnitude of optical rotation increased progressively with increasing boiling temperature of the fractions.
2. The measurements demonstrated that the angle of optical rotation increased as the measurement wavelength decreased relative to the standard sodium D-line (589.3 nm). This result confirms the wavelength dependence of optical rotation characteristic of optical rotatory dispersion (ORD).
3. The chiroptical properties of selected petroleum fractions were investigated by circular dichroism (CD) spectroscopy. A positive Cotton effect was observed at wavelengths of 350–355 nm. The corresponding spectral features were attributed to the presence of chromophoric groups, including carbonyl (C=O) functionalities, conjugated diene systems, and heteroatom-containing molecular fragments, which are responsible for the observed chiroptical behavior of the petroleum fractions.

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