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APPLICATION OF NUCLEAR MAGNETIC RESONANCE TO STUDY BITUMINOUS OIL IN THE SOUTH WESTERN NIGERIA

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ПРИМЕНЕНИЕ ЯДЕРНО-МАГНИТНОГО РЕЗОНАНСА ДЛЯ ИЗУЧЕНИЯ БИТУМИНОЗНОЙ НЕФТИ НА ЮГО-ЗАПАДЕ НИГЕРИИ

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Heavy bituminous oil is the main source of Nigerian unconventional resources. The resources represent oil sand and bitumen oil forming a belt of bitumen oil covering about 120 km extending from Lagos, Ogun, Ondo and Edo. Huge reserves of the resources are located in the state of Ondo. A nuclear magnetic resonance (NMR) method was used in the research to study bitumen oil in South West Nigeria. The NMR spectroscopy on the nuclei of ^{1}H and ^{13}C uses signals from protons and carbon nuclei, respectively, tetramethylsilane molecules $Si(CH_3)_4$. Regions of absorption of aliphatic (7–65 ppm) and aromatic (108–170 ppm) nuclei of carbon atoms are clearly defined in the NMR spectra of 13C of the objects under consideration. Signals of carbon atoms of olefinic fragments make a significant contribution to the last range of spectra of the cores of Nigerian bitumen deposits. An analysis of more known methods of defining the aromatic region of the nuclear magnetic resonance spectrum of 13C showed that for fractions that do not contain condensed cyclic and heteroatomic compounds, the definition of chemical shift subranges (CS) corresponding to aromatic carbon atoms 110–130 ppm not substituted, 130–137 ppm substituted by methyl, another alkyl – and naphthyl substituted – 137–148 ppm is sufficienly reasonable. There are regions of absorption of quaternary carbon atoms bonded to oxygen or nitrogen (148–170 ppm), carbonyl carbon atoms (170–200 ppm), as well as tertiary aromatic carbon atoms, located in the ortho position to the hydroxyl or other oxygen atom (108–118 ppm) in objects containing larger amounts of heteroatoms.

Ключевые слова:
метод ядерно-магнитного резонанса, тетраметилсилан, спектр, поглощение, кислород, водород, керн, ядра, протон, ресурсы, битум, нефть, спектроскопия.

Тяжелая битуминозная нефть является основным источником нигерийских нетрадиционных ресурсов. Эти ресурсы существуют в виде нефтеносного песка и битуминозного масла, образуя пояс битумной нефти, охватывающий около 120 км, простирающийся от Лагоса, Огун, Ондо и Эдо, причём огромные запасы этих ресурсов находятся в штате Ондо. В этом исследовании применен метод ядерно-магнитного резонанса (ЯМР) для изучения битуминозной нефти юго-запада Нигерии. В спектроскопии ЯМР на ядрах ^{1}H и ^{13}C в качестве стандарта используют сигналы протонов и ядер углерода, соответственно, молекулы тетраметилсилана $Si(CH_3)_4$. В спектрах ЯМР ^{13}C рассматриваемых объектов разделяются полностью области поглощения алифатических (7–65 м.д.) и ароматических (108–170 м.д.) ядер атомов углерода. В спектрах керна битуминозных отложений Нигерии в последний диапазон вносят заметный вклад сигналы атомов углерода олефиновых фрагментов. Анализ более известных способов дробного деления ароматической области спектра ядерно-магнитного резонанса ^{13}C показал, что для фракций, не содержащих конденсированных циклических и гетероатомных соединений, достаточно обоснованным можно признать выделение поддиапазонов химического сдвига, соответствующих ароматическим атомам углерода: незамещенным – 110–130 м.д., метилзамещенным – 130–137 м.д., другим алкил- и нафтилзамещенным – 137–148 м.д. В объектах, содержащих более значительные количества гетероатомов, выделяют области поглощения четвертичных атомов углерода, связанных с кислородом или азотом, – 148–170 м.д., карбонильных углеродных атомов – 170–200 м.д., а также третичных ароматических атомов углерода, находящихся в орто-положении к гидроксильному или эфирному атому кислорода, – 108–118 м.д.

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Introduction

According to the definition of the United Nations Institute for Training and Research the word "bitumen" means a hydrocarbon that is essentially immobile in a formation. Heavy oil may differ from bitumen by about 20° API. Within this range heavy oil is sometimes subdivided into super heavy and heavy at the density of 10° API [1-4].

Reservoir temperature plays a very important role in determining the viscosity of oil or its mobility. The Fig. 1 shows the rating of 10 countries that have reserves of bitumen and extra heavy oil. Classification of hydrocarbons in reservoir conditions is often used in the regulation of hydrocarbon reserves. Bitumen is most often regulated in accordance with the national rules for the extraction of minerals. Light oil is regulated in accordance with national regulations for hydrocarbons or oil [5-15].

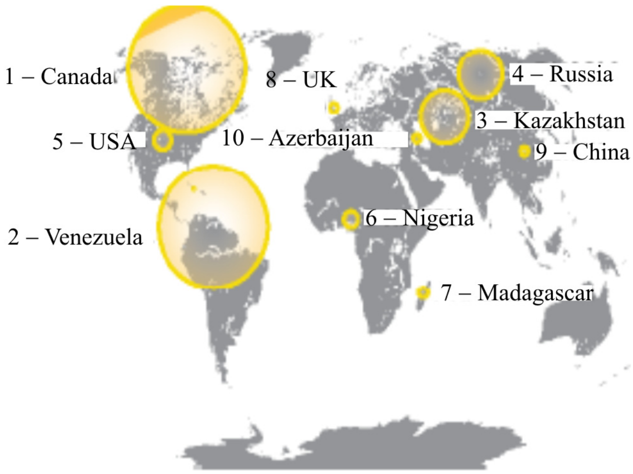

Fig. 1. Rating of 10 countries that have reserves of bitumen and extra heavy oil [17]

Regional geological formulation: structure and tectonics

Bituminous sandstones in southwestern Nigeria are located between the coastal plain and elevation. Geological conditions are characterized by the resins of Ilesha province, structural and minor topographic abysses. Plains and highlands of Benin basin are in the west, valley and delta of the Niger are in the east where shallow layers of the Anambra basin are studied.

The coastal plain is underlain by sedimentary strata forms surface of the earth, usually with a low relief. Drainage is moderately integrated, but most of the rivers are relatively small and have drainage basins either in the coastal plain or in adjacent hills. Most of the land surface has a good developed lateritic soil cover. Bedrocks are usually not affected, except for artificial sections or excavations. Sandstones and clays of Cretaceous and Tertiary age lie in the northern part of the coastal plain; some of them are relatively unconsolidated. The southern part of the coastal plain represents a coastal lowland strip, slightly widening to the east, where swamp and coastline relief are dominating. Alluvial valleys sediments of Quaternary age have a border with larger rivers [16-20].

Resins compose the area of higher relief to the north. Gneisses, quartzites, granites and shales dominate in the set of igneous and metamorphic rocks of Precambrian age. Sedimentary sequences on hills of the basement indicate in several places a tendency to overlap the formations with the underlying surface when it rises to the north. In the south deposits are located in a sedimentary chain at great depths.

The Benin basin is named after a country that borders Nigeria in the west. It was called Dahomey and Cotonou basin. The basin was formed after the rifts in a marginal position. That developed in the shallow gulf of the coast of West Africa after the opening of the equatorial Atlantic Ocean in the early Cretaceous period. The Benin basin is extended east-west parallel to the coastline, extending from Ilesha's spur in the east to the coastal lowlands of Ghana in the west. The northern edge of the basin is characterized by the exposure of the basement, which is located 130 km from the coast along the central axis of the basin near the border of Nigeria and Benin. The southern limit of the basin is poorly defined and is located beneath the seabed beyond the continental shelf.

The Anambra basin, east side of oil sandstones, is the geological area that lies at the base of the western coastal part of the Niger Delta. Like the basin of Benin, it originated in the early Cretaceous period as a rift structure and is distinguished by its extension to the north-east as one of a series of structural deflections caused by the thinning of the crust along the failed axis of the rift perpendicular to the center of the Atlantic. Benin hinge represents the most significant part of this system. It is a clearly defined structure located north-east, where the northwestern flank of the basin meets the spine of Ilesha [21-24].

Stratigraphy

The age of formations of the eastern Benin basin and the Ilesha spur is from Cretaceous to modern. Surface distributions of major lithostratigraphic units were mapped across the region, resulting in a basic stratigraphic structure that uses surface survey data and information about shallow wells. Some stratigraphic nomenclatures can be used, while others are based on descriptions of outcrops.

Sandstones and shales of Maastrichtian (late Cretaceous) age exposed in the resin belt are the oldest layers. These fossils usually denote the formation of Aboeokuta. In its typical location to the west of the resin belt, the Abeokuta form has a thickness of approximately 600 feet, but it becomes somewhat thinner in the east. Detailed studies of the resin belt show that individual sandstones are strong, but the general picture consists of basal sandstones and conglomerates, followed by a sandy-shale gap of several hundred feet and then the upper part of shales [25-29].

Magnetic properties of the core

The nuclei have a spin quantum number I (nuclear spin). The phenomenon of nuclear magnetic resonance (NMR) can be observed only for nuclei with a nonzero spin quantum number, I ≠ 0. Nuclei with I ≠ 0 have a magnetic quantum number m_i = I, I-1, I-2, … –I (values range from I to –I, through 1). For the nucleus of the hydrogen atom ^{1}H (proton): I = 1/2 and m_i = 1/2, –1/2. The nuclei of atoms have their own angular momentum

$$p = \sqrt{\mathrm{I}(\mathrm{I}+1)}h/2\pi ,$$

where I – nuclear spin, h – Planck's constant. Magnetic nuclei (I ≠ 0) are characterized by a magnetic moment μ, which is calculated by the formula $\mu = \gamma \cdot p$, m_i – projection of the vector μ on the magnetic field strength line; γ – gyromagnetic ratio (individual characteristic of the nucleus).

The energy of the nucleus in a magnetic field is characterized by the expression

$$E = -mh/2\pi\gamma\beta_o,$$

where β_o – magnetic field strength. For $m_1 = +1/2$, $E_1 = -h/4\pi\gamma\beta_o$. For $m_2 = -1/2$, $E_2 = h/4\pi\gamma\beta_o$. $\Delta E = E_2 - E_1 = h/2\pi\gamma\beta_o$. The basic NMR equation is described by the expressions

$$\Delta E = h/2\pi\gamma\beta_o,\ \Delta E = hv,$$

where h – Planck's constant, γ – gyromagnetic ratio; β_o – magnetic field strength, v – resonant frequency, $v = \gamma\beta_o/2\pi$. For nuclei ^{1}H $\gamma = 2.674\times 10^8$ $c^{-1}Tl^{-1}$ at β_o = 1.4 Tl, v = 60 MHz (resonance) [30-39].

Objects and methods of research

The Yegbata core from the south-west of Nigeria was selected from the depth of 750 m. The electronic image of the surface of the bituminous core sample is obtained using the high-resolution scanning microscope JSM 7500F (Japan) with a resolution of 1 nm at 100-fold zoom and is shown in Fig. 2.

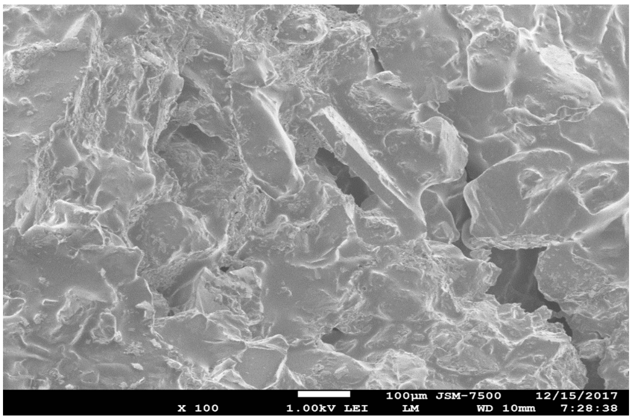

Fig. 2. Electronic surface image of bituminous core of the Yegbata deposit, Nigeria

The NMR spectra were recorded on the JNM ECA-400 spectrometer (Japan, JEOL, 400 MHz) in deuterated chloroform. As an internal standard, tetramethylsilane was used. A sample of a part of the bituminous core (sampling depth of 750 m) was placed in a solvent and mixed until the organic phase. Insoluble residue of the rock was filtered out. Number of accumulations for the spectrum for ^{1}H is 48, for ^{13}C is 5000.

There is in the spectrum ^{1}H the signal of the solvent is at 7.21 ppm, the signal of the standard is at 0 ppm. The spectrum contains signals from groups CH_3 with a shift of 1 ppm, for CH_2 shift is 1,5 ppm and signals branched alkyl fragments in the shear region (2.2 ppm). The shift signal in the region of 7-8 ppm corresponds to hydrogen signals of aromatic groups. The ^{1}H NMR spectrum of the organic part of the bituminous core sample shows a peak value at a chemical shift of 1.5 ppm (Fig. 3).

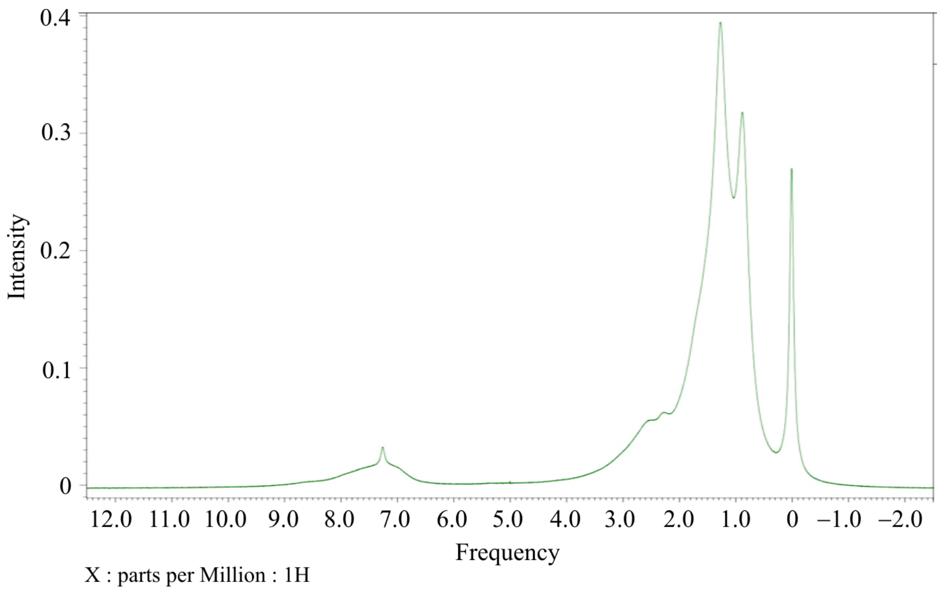

Fig. 3. Nuclear Magnetic Resonance Spectrum of 1N Bituminous Oil of Nigeria

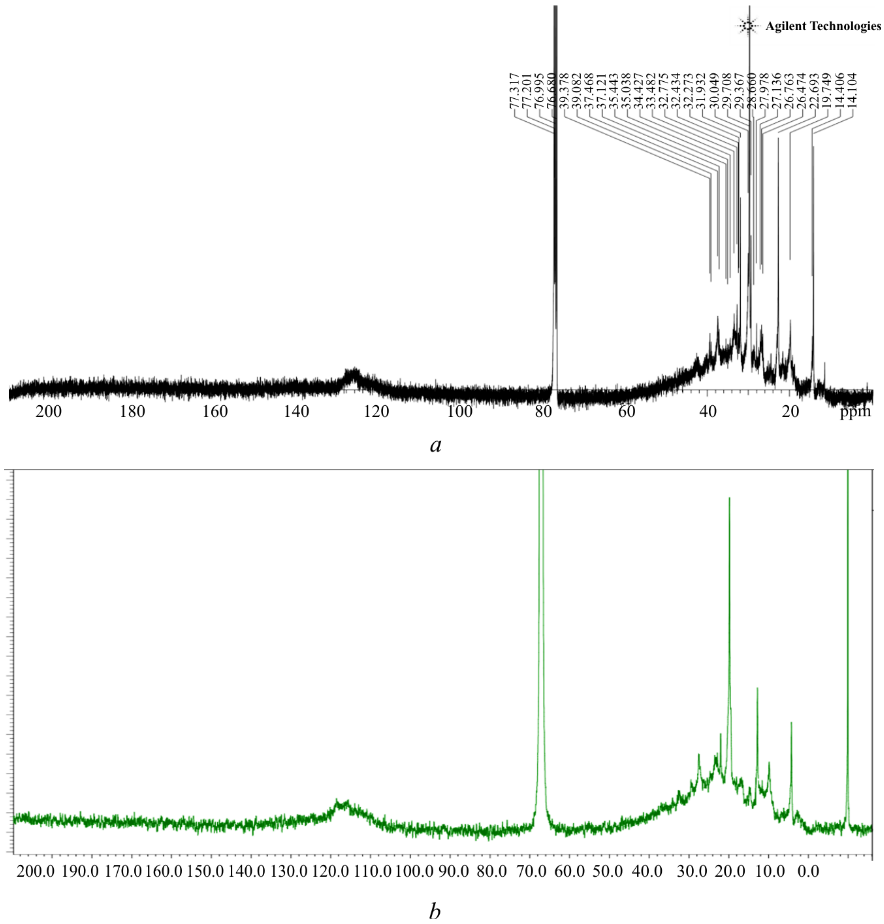

Fig. 4. Nuclear Magnetic Resonance Spectrum of ^{13}C: *a* – Kuban oil; *b* – bituminous oil field of Yegbata in Nigeria

The ^{13}C spectrum shows the intense signal of the solvent at 77 ppm and TMS standard signal at 0 ppm. There are signals observed in the spectrum of the carbon nuclei of various alkyl groups (saturated hydrocarbons) CH_3, CH_2 and CH in the region of shifts from 10 to 50 ppm. A large group of signals in the region of 130 ppm corresponds to carbon signals of aromatic groups of polycyclic compounds of bituminous oil. The general form of the ^{13}C NMR spectrum indicates a large number of aliphatic and aromatic components of bituminous oil.

Results and discussion

For a comparative illustration the NMR spectrum of ^{13}C of the Kuban oil of the Western Akhtanizovsky deposit (well No. 30, bottom 1479 m) is shown in Fig. 4*a*. There is the ^{13}C NMR spectrum of bituminous oil from Nigeria of the Yegbata deposit shown in Fig. 4*b*.

There is a coincidence of the NMR ^{13}C spectra of the Kuban oil and bituminous oil of Nigeria determined in the region of signals of the ^{13}C carbon nuclei of aliphatic groups of the compared spectra (see Fig. 4). However, there are more wide intense lines in the aromatic carbons area in the range of 108-150 ppm observed in the ^{13}C NMR spectrum of the bitumen oil sample of Nigeria.

The range of chemical shifts of ^{13}C NMR and assignment of carbon classes for a sample of bituminous oil from the Yegbata deposit in Nigeria are shown in the table.

Range ^{13}C and classes of carbon of the bitumen sample from the Yegbata field of Nigeria

The range of chemical shifts of ^{13}C, ppm	Identification of carbon classes	Classification of carbon
0-107	C_{al}	Aliphatic carbon atoms
108-118	$C_{ar.op}$	Tertiary aromatic carbon atoms in ortho-position to ethereal oxygen
110-130	C_{ar}	Tertiary aromatic carbon atoms*
130-137	$C_{ar.m}$	Methyl-substituted aromatic carbon atoms*
137-148	$C_{ar.al\text{-}nap}$	Alkyl- and naphthyl-substituted aromatic carbon atoms*
148-170	$C_{ar}O$	Aromatic carbon atoms substituted by a phenolic or ester group
170-200	C_c	Carbonyl carbon atoms

Note: * – absence of heteroatomic and condensed aromatic compounds.

Conclusion

Core samples of bituminous deposits of the Yegbata field of Nigeria are studied using the modern methods of spectral studies. The classes of hydrocarbons that make up the organic part of the core of the bituminous field of Nigeria are determined. The coincidence of the ^{13}C spectra of the Kuban oil and NMR ^{13}C spectra of bituminous oil in Nigeria in the area of chemical shifts of the ^{13}C carbon nuclei of aliphatic groups (saturated hydrocarbons) CH_3, CH_2 and CH in the shift range from 10 to 50 ppm is shown. The ^{13}C NMR spectrum of the bituminous oil sample has a wider range and larger integral value in the region of 118-150 ppm, which corresponds to carbon signals of aromatic groups of polycyclic compounds of bituminous oil in Nigeria. The ^{1}H NMR spectrum of the sample of the organic core component shows an increased peak value with a chemical shift of 1.5 ppm aliphatic part of the bituminous oil of Nigeria. The spectral data of organic part of the real core material obtained indicate a unique source of raw materials with a wide component composition of aliphatic and aromatic hydrocarbons included in the bituminous oil of the Yegbata field in Nigeria.

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