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Thesis Changes Log

Name of Candidate: Aliya Mukhametdinova

PhD Program: Petroleum Engineering

Title of Thesis: Unconventional Reservoir Characterization Using Low-Field Nuclear Magnetic Resonance

Supervisor: Associate Professor Alexey Cheremisin

The thesis document includes the following changes in answer to the external review process.

Dear Reviewers,

I would like to express my gratitude for your comments and suggestions to all of you! I found them very useful and implemented them into my thesis. Also, I would consider them in my future research work. Please find the responses to your comments.

Sincerely,
Aliya Mukhametdinova

Thesis Change:
According to the Jury members recommendations, the title of the thesis was changed from *Investigation of the Reservoir Properties of Unconventional Reservoirs Using Nuclear Magnetic Resonance* to *Unconventional Reservoir Characterization Using Low-Field Nuclear Magnetic Resonance*.

Reviewer: Prof. Dmitry Koroteev

Comment 1:

I can hardly see a link between the permafrost chapter and other chapters. Even though it is clear that tackling permafrost issues at field development need to be tackled accurately (for example at drilling), I cannot see a logical link here to oil and gas-bearing formations described in the other parts of the text. Could you please be more precise at the logical connections between the Bazhenov, Domanik, Heavy Oil reservoirs from one hand and the Permafrost soils from the other?

Response:
Thank you for the question In Russia, some of new large oil and gas fields are located in the Arctic, *i.e.* in the area of permafrost distribution (continent or Arctic shelf); therefore, for the safe development of these deposits, an understanding of the specifics of permafrost is crucial. These specifics are especially important while drilling and operation of production wells (facing thawing areolas around the wells and affecting the stability of the wellbore). The risks include a decrease in reservoir temperatures (up to negative ones) in the bottomhole zone of the wells due to the Joule-Thomson effect (throttling). In addition, there is an evident presence of large gas accumulations within the permafrost (intrapermafrost gas and gas hydrates), which at the testing stage of field development can be considered as promising for production.

This thesis is aimed at the application of NMR in various scientific and applied areas, the purpose of which is to help in the development of cost-effective and safe (accident-free) technologies for the development of new unconventional fields in the Russia, both on the continent and on the shelf. Therefore, the study of the phase composition of the gas-containing permafrost horizons is of great importance as well. In recent days, in many countries (China, USA, Japan, Canada), NMR technologies are actively used to study permafrost

and intrapermafrost gas accumulations (including gas hydrates). This chapter covers the application of low-field NMR relaxometry in defining the saturation in permafrost under different conditions.

Therefore, this area should be viewed as a research direction, along with research of Bazhenov, Domanik, heavy-oil reservoirs. I included a short motivation part to the introduction part in corresponding chapter, please refer to Chapter 4.

Comment 2:
Could you please expand on why you compare NMR measurements on cores with GR logging data, not NMR logging?

Response:
Thank you for the valuable question. 2-MHz laboratory NMR setups are often used for calibration of the well-logging tools working at the same Larmor frequency. Therefore, it is crucial to understand the reliability of laboratory measurements with the ones obtained by conventional logging tools. It is important to note that the NMR logging is not widely used within a standard logging complex due to its high operation price and availability. For this reason, the porosity we compare laboratory NMR porosity is the one from classical porosity neutron logging tool. The following explanation part was included into the thesis body, please refer to section 3.3.1.

Comment 3:
Talking about the practical implementation of NMR workflows for wettability characterization in massive core testing campaigns, do you believe the NMR will be more beneficial over the rapidly developing micro-CT techniques for saturated samples? Can you expand on this?

Response:
Thank you for interesting question. Micro-CT technique in characterizing the rock wettability is gaining high interest in last 10-15 years and considered as a part of digital rock. Wettability characterization of the rock is based on processing of spatial images of rock obtained by X-ray micro-CT. Numerous studies confirm the applicability of such approach in determining the rock wettability. However, the most of studies are conducted on high-porosity and high-permeability sandstone samples or carbonate rocks. In case of many shale rock studies, the most of fluid is hosted in pores of the nano-size. In massive core testing, the standard sample is a cylinder of 25.4 (1-inch) or 30 mm diameter. X-Ray micro CT does not allow the resolution higher than 7-10 μm for such samples, and therefore, the essential part of the porous system is not registered. Such limitation results in distorted data and inaccuracy of interpretation. To increase the spatial resolution of the scanned sample, it is required to prepare a core sample of diameter of 3mm or less which falls out from the conventional rock analysis workflow. In turn, the NMR analysis is conducted on standard core plugs in two saturation states (oil and water) which is typically already embedded in standards core analysis and doesn't require additional studies and core preparation.

Comment 4:
Please check once again for the typos. I still can see several of them all over the text.

Response:
Thank you, the text has undergone the proofreading, and the typos were removed.

Reviewer: Prof. Yury Popov

Comment 1:
Metrological aspects are not described in the thesis at all practically that is not acceptable for the experimental work. The measurement quality should be estimated with description of measurements precision, accuracy and uncertainty at least shortly with mentioning approaches applied for estimations of these parameters.

Response: Thank you a lot for valuable question. To address the methodological aspects, I included the separate chapter to the Chapter 2 – Section 2.3.2 on precision, accuracy and uncertainty of NMR measurements.

Comment 2:

Inessential technical error should be removed in formula (5), Page 71.

Response: Thank you, it is corrected.

Comment 3:
The information on determination coefficient should be removed from Figure 82 where regression equation based on two elements (!) is given.

Response: Thank you, it is removed.

Comment 4:
Regression equations would be useful for experimental data shown in Figures 75 and 76.

Response: Thank you; I added the regression equations for noted graphs.

Reviewer: Prof. Reza Rezae

Comment 1:
In Figure 34 for all 13 samples, there are both Preserved and As is states. How this is possible? Does this mean that you have left the preserved samples exposed to air for a while and then conducted another NMR? Explain this in your thesis.

Response: Thank you for the question. Preserved samples are the samples covered in the paraffin, and therefore never exposed to the air after prompt drilling on site. In preserved state, we have to deal with the signal of noise caused by the layers of paraffin. As-is samples are the ones examined right after unpacking (in nearest 5-10 min) and exposed to air for the testing duration. I have revised this part of the thesis and removed the data from paraffin-covered samples to avoid any misunderstanding. Please refer to section 3.1.2.

Comment 2:
Page 51, Figure 36 – Is not there any physical change to rock's pore structure after water introduction?

Response: Thank you for the comment. For shale samples, we have attempted to clean the kerosene-saturated samples and saturate them again with a brine model. Due to high content of clays in the rock, 7 out of 5 samples were destroyed during the saturation process. Therefore, we have statistics for 5 samples only. To illustrate the changes, we added a new graph (please refer to Section 3.1.1) which illustrates the change in porosity after saturation with water. However, the high porosity of brine-saturated samples can be explained by occurrence of micro-crack and thin fractures in the matrix, caused by clays extensively adsorbing water.

Comment 3:
Page 56: Figure 39 is against your statement "It is noted that maximum NMR porosity values are characteristic for stage 3, at which analyzed samples saturated with decane." The figure clearly shows higher NMR porosity for the brine-saturated state. Please clarify this issue.

Response: Thank you for noting, the Figure 39 was an example for one sample of the collection with relatively high porosity. However, we investigated three layers with more than 50 core plugs and following statements were made based on all accumulated data. I revised this part of the thesis to address this comment and added a table with data on porosity for both decane and water-saturated samples. Please refer to the section 3.2.2.

Comment 4:
Page 59: What do you mean by gamma ray-neutron porosity? Needs to be clarified.

Response: Thank you for the valuable question. 2-MHz laboratory NMR setups are often used for calibration of the well-logging tools working at the same Larmor frequency. Therefore, it is crucial to understand the reliability of laboratory measurements with the ones obtained by conventional logging tools. It is important to note that the NMR logging is not widely used within a standard logging complex due to its high operation

price and availability. For this reason, we compare laboratory NMR porosity with the one from classical porosity neutron logging tool. The following explanation part was included into the thesis body, please refer to section 3.3.1.

Comment 5:
Page 61: "Thirdly, NMR tends to provide the highest porosity value (Figure 45). We explain this observation by the technical capability of NMR to detect the full range of pores – from nano-sized pores filled with high-viscous components to large voids occupied by mobile fluids." This is not simply because other methods such as LS provide connected or effective pore spaces whereas NMR provides total porosity?

Response:
Thank you for noting. We agree that in comparison with LS and gas porosity NMR shows higher values because NMR provide total porosity, whereas LS and PD (pulse pressure decay) provide connected/ effected pore space. However, this statement was initially made based on comparison of NMR and micro CT as well, so we enhanced the corresponding section with new CT data and new comparison chart.

Comment 6:
In Publications Page, correct for 5 and 6 that are one publication "5. Rock porous structure characterization: A critical assessment of various state-of- the-art techniques, Mehdi Razavifar, Aliya Mukhametdinova, Ehsan Nikooee," 6. Alexander Burukhin, Amin Rezaei, Alexey Cheremisin, Masoud

Response: Thank you, it is corrected.

Comment 7:
Books' references are cited in the body of the text incorrectly, e.g., at Page 1: Technically Recoverable Shale Oil and Shale Gas Resources: An Assessment of 137 Shale Formations in 41 Countries Outside the United States 2013 You do not need to write the book's name. Author name and year would be enough. In the reference list the basic form of a book citation is: Last Name, First Name. Title of Book, Publisher, Publication Date.

Response: Thank you, the reference is fixed.

Comment 8:
Correct Labani & Rezae to Labani & Rezaee.

Response: Thank you for noting, the reference is fixed.

Comment 9:
Page 29, Instead of using Objects of Investigation use Rock samples used for this study;
Table 3: "List of objects under study" is better to change to "List of rocks used for this study".

Response: Thank you for recommendation, the both titles are changed.

Comment 10:
Page 31: What is the clay type of Bazhenov Formation.

Response: Main clay minerals are illite and kaolinite, however clays are also presented by illite/smectite mixed-layered mineral compounds. The following piece was added to the main thesis body at page 30.

Comment 11:
Page 49: change "The results on the determination of porosity by NMR were complexed and displayed on a graph." to "The results on the determination of porosity by NMR were compared and displayed on a graph."

Response: Thank you, it is corrected.

Comment 12:
Page 50, Figure 34 change Pooosity to porosity.

Response: Thank you, it is corrected.

Comment 13:
Page 57: Remove “TOC (total organic carbon) values are presented in Figure 2 below.”

Response: Thank you, the sentence is removed.

Comment 14:
Page 62: change “complexed” to “compared”.

Response: Thank you, it is corrected.

Comment 15:
Page 136: “An increase in the fluid viscosity is accompanied not only by changes in the N1 and T2”; change N1 to T1.

Response: Thank you, it is corrected.

Reviewer: Prof. Sergey Stanchits

Comment 1:
I recommend making a double-check of all the references in Chapter 1;

Response: Thank you, the list of references was revised and corrected.

Comment 2:
I suggest restructuring the introductory part of dissertation (sections 1.2.2-1.2.4), considering the option of slightly shortening the literature review part;

Response: Thank you for recommendation. The mentioned sections were shortened and merged; the titles were changed. Please refer to corresponding sections.

Comment 3:
Section 2.3. I recommend adding a paragraph describing the NMR measurement limits in terms of visible fluid volume and pore size;

Response: I attempted to address this question by adding the new section on NMR precision, accuracy and uncertainty, please refer to the Section 2.3.2.

Comment 4:
Section 3.1, 3.2. I recommend adding separate conclusions for these sub-chapters;

Response: Thank you for recommendation; I included short summaries per each section.

Comment 5:
Section 5.4. I recommend to provide more details regarding the calculation of the diffusion coefficients;

Response: Thank you, we expanded the explanation of both experimental basis and equations used in calculation of the diffusion coefficient. Please refer to Sections 5.3.2, 5.4.2, 5.4.3.

Comment 6:
Chapter 7. I recommend to highlight the novelty of the study in the Conclusions section;

Response: Thank you, we enhanced the Conclusions section (into Summary, Conclusions, Recommendations) and attempted to highlight the novelty of research. Please refer to the Section 7.

Reviewer: Prof. Sudarshan Mehta

Comments included the following points:

-formatting the page numbers;

-grammar mistakes and typos;

-capitalization, style of specific word (numeration, in-situ, etc.);

-formatting of the references;

-structure and titles of the sections;

-adding abbreviations;

-English proofreading;

-quality of figures.

Response: Thank you, all recommendations and comments were addressed. Please refer to the text of thesis.

Reviewer: Prof. Dimitri Pissarenko

No corrections requested.