Reviewer’s Report

Organic-rich shales present the perspective hydrocarbon resources for the future due to depletion of conventional oil reserves. However, existing recovery technologies are not efficient for their development. Application of thermal methods which have technological potential for the production of crude oil from organic-rich shales, requires detailed fundamental studies of thermal effect on organic and mineral matter structure and properties. Bazhenov formation is one of the biggest in the world and largest in Russia organic-rich shales resource. Consequently, it is very important and interesting object for the study of thermal methods effects on rocks and organic matter properties. This fact fully substantiates the choice of the Bazhenov Formation as an object of research for the dissertation work of Tagir Karamov. The methodology and experimental results obtained for this object can be used to evaluate and analyze other organic-rich shales reservoirs. Based on the problems described above, it can be concluded that the purpose of Tagir Karamov’s dissertation, which includes studies of the influence of thermal methods on the transformation of organic matter and the properties of the mineral rock of the Bazhenov Formation, is very relevant and practically important.

In this work the degree of organic matter transformations and mineral matter condition before and after thermal impact with the application of thermal methods, in particular HPAI method, was studied with a large variety of high-precision modern and in some cases unique petrophysical and geochemical methods such X-ray diffraction, CT-microtomography, SEM, Rock-Eval, isotope analysis, pyro-GC-TOFMS, thermal conductivity and others. This shows how deeply and systematically the author approached the solution of the tasks, which is undoubtedly the strong side of the work.
Tagir Karamov completed a lot of experiments and characterized Bazhenov’s source rocks and its microstructural changes in organic and inorganic parts. It should be noted that core samples of Bazhenov formation was studied before, nevertheless, important summary and characterization of transformations was done for the first time, especially via microtomography of porous space before and after thermal treatment.

In the second chapter a detailed description of Bazhenov formation oil shales was conducted via modern existed literature data. Stratigraphic and tectonic information was added as well and reviewed. Studies of alteration of mineral matrix and organic matter due to thermal treatment also was reviewed in this section. It was concluded that there is a lack of information about this important topic and new comprehensive studies are required. However, few recent references about transformation of mineral matrix due to oxidation experiments were not added. Information about thermal treatment technologies for oil shales proposed by the companies was also discussed. Despite that fact author presented a brief review on only a few thermal technologies for the development of organic-rich shales. General classification of methods by the source of thermal treatment could be more valuable as well as reflection of a critical analysis of such technologies in order to mention details about their advantages and disadvantages and areas of application.

Third chapter contains comprehensive lithological characterization of Bazhenov formation shales. It is difficult to remember such careful and detailed characterization of Bazhenov formation samples, especially from the northern part of the reservoir. This part in itself is of great scientific value.

However, the main novelty of Tagir Karamov’s thesis is presented in chapters 4 to 6. Author studied in detail the effect high pressure air injection on the porosity, minerals structure and properties of organic matter. The transformations were fixed and analyzed in different zones of the combustion process, and they were completely different according to the position of rock sample relative to the combustion front. Also, the behavior of each lithotype was correctly partially separated and observed via physical methods of study. It was concluded that HPAI leads to the increase of porosity due to organic matter transformation and replacement as well as mineral matrix transformation into other new-formed minerals. One of the important findings of this work was the fixing of significant thermal conductivity decrease after thermal treatment. It is very useful data for the correct simulation of HPAI process before application.

Characterization of kerogen microstructural transformation during thermal maturation confirmed evolution of both natural occurring porosity and resulting from artificial thermal maturation. The appearance of primal voids resulting from the general processes associated with sedimentation.

In the last part the main chemical transformations of different types of minerals and their influence on maturation/porosity were shown. Conceptual scheme of alterations in shale’s during high-temperature treatment and the description of it as well as pore space evolution were presented first time in the current research literature. It is very important from an engineering point of view that the recommendations for the thermal technology applications for different lithotypes were developed.

Author showed strong competencies and knowledge in geology, petroleum engineering and petrophysical methods of research. Main results of this work were published in 7 papers in well-known peer-reviewed journals. This fact confirms the reliability of obtained results and their novelty.

Generally, thesis is well written in English and good organized. Some minor orthographic errors presented in the text. Author showed a detailed literature review with the focus on laboratory testing of immature...
Bazhenov’s source rock and the changes in its petrophysical properties due to thermal treatment. Less attention was paid on the analysis of papers about oxidation behavior of organic matter and combustion.

Like any work devoted to the study of a wide range of methods and one object, it contains less detailed description of specific processes and their stages. However, this does not affect the results obtained in this work.

In all I consider this PhD thesis worth the degree. The author has proved himself to be a highly qualified specialist who can conduct excellent and valuable research work independently with the possibility of maintaining high scientific perspective. He deserves to get PhD degree in Petroleum Engineering.

**Provisional Recommendation**

☑️ I recommend that the candidate should defend the thesis by means of a formal thesis defense

☐ I recommend that the candidate should defend the thesis by means of a formal thesis defense only after appropriate changes would be introduced in candidate’s thesis according to the recommendations of the present report

☐ The thesis is not acceptable and I recommend that the candidate be exempt from the formal thesis defense