

## Jury Member Report – Doctor of Philosophy thesis.

**Name of Candidate:** Maksim Zhmaev

**PhD Program:** Petroleum Engineering

**Title of Thesis:** Experimental evaluation of filtration properties of cryolithozone rocks under conditions of formation and decomposition of gas hydrates

**Supervisor:** Dr. Evgeny Chuvilin

**Name of the Reviewer:** Prof. D N Singh

I confirm the absence of any conflict of interest

**Date:** 15.11.2024

*The purpose of this report is to obtain an independent review from the members of PhD defense Jury before the thesis defense. The members of PhD defense Jury are asked to submit signed copy of the report at least 30 days prior the thesis defense. The Reviewers are asked to bring a copy of the completed report to the thesis defense and to discuss the contents of each report with each other before the thesis defense.*

*If the reviewers have any queries about the thesis which they wish to raise in advance, please contact the Chair of the Jury.*

### Reviewer's Report

Reviewers report should contain the following items:

- Brief evaluation of the thesis quality and overall structure of the dissertation.
- The relevance of the topic of dissertation work to its actual content
- The relevance of the methods used in the dissertation
- The scientific significance of the results obtained and their compliance with the international level and current state of the art
- The relevance of the obtained results to applications (if applicable)
- The quality of publications

The summary of issues to be addressed before/during the thesis defense

Please see the attachment.

**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~~*

## Referee's Evaluation Report on Doctoral Thesis

Name of the Candidate: **MAKSIM ZHMAEV**

Name of the Supervisor: **Dr. Evgeny Chuvilin**

**Leading Research Scientist**

**Skolkovo Institute of Science and Technology**

Title of thesis: **EXPERIMENTAL EVALUATION OF FILTRATION PROPERTIES OF CRYOLITHOZONE ROCKS UNDER CONDITIONS OF FORMATION AND DECOMPOSITION OF GAS HYDRATES**

Name of the Referee: **Prof. Devendra Narain Singh**

**Indian Institute of Technology Bombay, India**

### Referee's Comments to the Candidate

#### **Summary of the thesis:**

An experimental methodology has been developed to investigate gas migration (mentioned as filtration but to be read as gas permeability) properties of the natural sediments (collected from the cryolithozone on the Yamal Peninsula (Russia) in the vicinity of the gas production wells) during formation and dissociation of gas hydrates in the presence of ice. Artificial sand-clay mixtures have been employed as the host sediments to conduct the experiments. Though, the novelty and outcome of the study appears to be good **the following issues need to be addressed.**

#### **Chapter 1:**

1. In statement of the problem, the term Permafrost rocks is used to explain the relevance of the present study to the real-life conditions. However, unconsolidated sediments from Yamal Peninsula and artificial sand-clay mixtures have been employed as the host porous media. In this context, it becomes mandatory to justify the reasons that why these materials are equivalent to the rocks.

#### **Chapter 2:**

1. Will it be OK to modify the title of the chapter as, **Formation and Existence of Gas Hydrates in Permafrost Regions** (and not the marine conditions).
2. It is mentioned that "n is the number of water molecules", is this the hydration number?

3. Lines 4-6 in the first paragraph of Subsection 2.1 should be substantiated with the relevant literature.
4. Page no 29, the statement “Due to hydrogen bonds ... attraction of van der Waals forces”, is not clear. Hence rewrite and cite proper references.
5. With reference to Figure 3, huge variations are observed in the Pressure (P) and Temperature (T) conditions favourable for the formation of hydrates with different gases. However, the explanation in this context is missing and should be included.
6. “With a decrease in temperature, thermal conductivity of hydrates will decrease” (Refer Page No: 32). Explain this phenomenon.
7. Replace the word *weaker* with *minor* on Page No: 33.
8. Explain the relevance of comparing the properties (refer Table 1) of hydrates and ice to the present study by including a separate paragraph. How these properties would be relevant to the present study?
9. Figure 4 presents distribution of gas hydrates in both marine conditions and permafrost regions. However, it is better to modify the figure by highlighting the permafrost regions as the present study is relevant to the hydrates in the presence of ice.
10. Provide a detailed explanation to elaborate the difference between Figures 8a & 8b and Figures 9a, 9b and 9c.
11. Figure 10 and its explanation is not clear and hence do the needful.

### **Chapter 3:**

- As the ice and hydrate bearing sediments are highly impermeable as compared to their host sediments and/or rocks, and the permeability depends on the ice or hydrate saturation and/or both in case of permafrost (ice) hydrate bearing rocks, the information provided in Section 3.2.2 should be modified to prove that the ice and hydrate bearing sediments are not barriers to the fluids.
- Lines 4-5 in the 2<sup>nd</sup> paragraph (Page No. 104), “the difference between the pore ice formation and hydrate formation”, should be elaborated with proper justification.

### **Chapter 4:**

1. The captions for the Figures 18a & 18b are interchanged. Please check and rectify.

2. In the general schematic of the sample holder (Figure 19b), where are the temperature sensors and the set up for the acoustic measurements? Define DM?
3. Subsection 4.2, Page No. 110, it is mentioned that the natural sediments have been taken from permafrost horizons of north of western Siberia. However, the locations of the samples Sand 2 and Polymyneral loam are different. Please clarify.
4. What is the meaning of letters used for Genesis Age in Table 12?
5. Plot and show the particle size distribution of all geomaterials used in the study instead of showing values in Tables 13 & 14.
6. Provide a flow chart for the methodology adopted for the filtration tests (Refer Subsection 4.3).
7. With reference to the sentence, “kept for 30 minutes to evenly distribute moisture in the pore space at a temperature of +22 °C (refer 115)”, how would one ensure uniform distribution of water content after 30 minutes? Clarify.
8. “The cooled mixture was carefully reshaped in an airtight container, after which ice chips of no more than 1 mm in size were added to it to create the necessary humidity”, what is the significance of the necessary humidity? After that, the container was kept at room temperature (+22 °C) for an hour to evenly distribute moisture in the pore space (refer Page No: 115). How was this achieved? Provide the photographs to justify the above statement.
9. Provide temperature profile for the freezing process like the profile provided for hydrates in Figure 21.
10. Provide the procedure to determine the  $P_{crit}$  and  $T_{crit}$  (refer Figure 20).
11. What is the meaning of “u.f”, that appear in Page 125.
12. In Table 20, it is mentioned that  $V_{p\ table}$  and  $V_{s\ table}$ . What does  $table$  corresponds to here? Please elaborate.
13. The explanation for Figure 22 in the text is not clear and is insufficient.
14. On Page no. 130, it is written, “A comparison of the results of determining  $V_p$  and  $V_s$  at two installations showed that the discrepancy between the results is within 5.2% (Fig. 5)”. Figure 5 is not relevant to this statement as it is the classification of hydrate reservoirs. Provide the justification.

## **Chapter 5:**

1. Remove either ice-bearing or frozen in the heading of Subsection 5.1 (Page 132.).
2. With reference to the Figure 23, explain the reason for the abrupt drop in pressure after 4 MPa.
3. On Page 134, it is wrongly written Figure 1a, instead of Figure 23a.
4. It is mentioned that the drop of pressure is related to a sharp activation of filtration in Page 134. However, it might be due to the partial melting of ice due to high pressures. Author must clarify.
5. It is observed that the “increase in temperature during the filtration experiment led to an increase in the permeability of frozen samples, but the intensity of the change in gas permeability was different” (Figures 30 & 31). However, the author should provide the reasons/ to support this.
6. The images provided in Figures 25, 26 and 27 are not clear. Please modify.
7. Provide a detailed explanation to understand the influence of the fine contents (clay fraction) on the gas filtration capacity during freezing and thawing of sediments.
8. With reference to Figure 29, substantiate increase in water content after filtration experiment.
9. As per the data provided in Figure 30, it is realized that the maximum gas permeability is around 5 mD. However, the range of gas permeability 22-25 mD is used to plot the graph between Gas permeability and Temperature (refer Figure 31). Please check and clarify.
10. With reference to the Figure 32, what is the reason for redistribution of the water content only between 6-8 cm length of the sample?
11. It has been observed that *moisture* and *humidity* are used at many places to represent the water content. What is the difference between these two and is it possible to use only one of these to eliminate the confusion.
12. The information provided in Figures 42, 43, 44 and 45 is not well documented.

## **Chapter 6:**

1. Provide details (profiles of temperature and pressure) of the synthesis and dissociation of gas hydrates.
2. In Figure 47, What are the characteristics of sample TS2?

3. What is non-equilibrium pressure, mentioned in Figure 47.
4. Plot the variation in velocities with change in gas permeability during dissociation.
5. Explain the reason for the nonlinear behaviour of gas permeability with axial pressure during unloading (Refer Figure 53).
6. With reference to the sentence, "In experiments with flue gas injection, the presence of nitrogen contributes to a more active decomposition of methane hydrates, which affects the increase in gas permeability at the initial stages of replacement" on Page no. 191, explain the reason for more decomposition of methane hydrates due to the presence of nitrogen.
7. Provide a detailed experimental methodology for conducting CH<sub>4</sub>-CO<sub>2</sub> replacement in Subsection 6.3.
8. Which method has been employed for hydrate synthesis?
9. What is the influence of the dissociated water on the effective gas permeability during the dissociation process?

Chapter 7:

1. What are the "capacity properties of rocks"?