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LABORATORY STUDIES ON THE EFFECTS OF HYDROGEN EXPOSURE ON POROSITY, PERMEABILITY, AND CHEMICAL COMPOSITION OF TERRIGENOUS RESERVOIR ROCKS: A CASE STUDY OF THE BOBRIKOVSKII FORMATIONS IN THE NORTHEAST VOLGA-URAL OIL AND GAS PROVINCE

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Abstract:
This article outlines the methodology for conducting laboratory investigations into the effects of hydrogen on reservoir rocks. It details the stages of sample analysis and the instruments employed during the experiments. A comparative evaluation of the porosity and permeability of core samples was undertaken, revealing a 4.6% decrease in porosity and a 7.9% reduction in permeability following hydrogen exposure. Correlation analysis indicated a typical shift in the relationship between these characteristics: post-exposure, the variability of values increased while the correlation coefficient decreased, suggesting alterations in the void structure. The findings suggest that the observed reductions in porosity and permeability are attributed to slight compaction due to effective stresses. Chemical analyses indicated no significant changes in the composition of primary oxides before and.

Keywords: Hydrogen; subsurface gas storage; reservoir rock; properties of porosity and permeability; chemical makeup of rocks; fundamental oxides.

Introduction

Recently, there has been a growing focus on eco-friendly production technologies and the reduction of carbon dioxide emissions, including the exploration of alternative energy sources to replace hydrocarbons. In publication [1], the authors

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highlight the importance of addressing global warming and discuss technologies for capturing and disposing of carbon dioxide. They emphasize that Russia should adopt these technologies through government programs, drawing on international experiences. Hydrogen is anticipated to serve as one of the alternative fuels, raising challenges related to its production and transportation. Articles [2-4] outline various production methods for hydrogen, each with differing levels of energy efficiency and environmental impact. The authors advocate for technologies that do not produce carbon dioxide emissions. Additionally, publications [5-7] indicate that transporting hydrogen necessitates densification (liquefaction) and enhanced safety for tanks and transport systems. A critical consideration in hydrogen energy is the selection of storage facilities. Scientific articles [8-10] indicate that salt caverns may be suitable for storing hydrogen-methane mixtures, but they have both advantages and disadvantages. While salt caverns offer excellent sealing properties, they require significant labor to create, incur high costs, and have limited storage capacity due to structural collapse under external pressures. Some researchers propose utilizing existing underground gas storage facilities (UGS) traditionally used for natural gas [11, 12]. For larger volumes, depleted gas fields, aquifers, or currently operational methane storage facilities are recommended. A significant challenge in hydrogen storage is the embrittlement of steel well strings and downhole equipment. Articles [13-15] provide evidence that exposure to hydrogen can lead to physicochemical changes in commonly used steel grades, resulting in cracking and deterioration of mechanical properties. Such effects can lead to unexpected emergencies in wells and equipment. The metabolic activity of hydrogen-consuming bacteria can also pose issues. Experts [16-18] have noted that certain bacterial species in reservoirs can convert hydrogen into hydrogen sulfide, an aggressive gas that can damage well structures and equipment.

When hydrogen is stored in reservoir beds, it can chemically react with the minerals in the rock matrix and the cap rock above. Authors of articles [19-21] present data showing that hydrogen can interact with pyrite and aluminum-containing minerals, as well as with dissolved carbon dioxide and sulfates, leading to the formation of methane and hydrogen sulfide. These reactions can alter the porosity and permeability of the formation. Publications [22-24] indicate that hydrogen can cause a twofold change in the porosity of core samples, with these effects being dependent on the lithological characteristics of the rocks.

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Thus, it is recommended to store gas primarily in terrigenous reservoirs free of clay and carbonate impurities. Similar to traditional underground gas storage for methane, storing hydrogen can create a complex geodynamic situation at the operational sites. As shown in articles [25-27], fluctuations in formation pressures during gas injection and withdrawal can cause surface deformations. Therefore, it is essential to establish geodynamic monitoring systems in these UGS. An analysis of the literature reveals that the effects of hydrogen on the porosity and permeability of reservoir rocks, as well as on the chemical composition of the rock matrix, have not been sufficiently studied. This highlights the importance of examining the impact of hydrogen on reservoir properties and the chemical changes in the rocks of the investigated formation. The study focuses on the terrigenous deposits of the Bobrikovskii horizon in the Volga-Ural oil and gas province, where gas is stored in the Karashurskoe UGS in the Republic of Udmurtia [28-30]. A methodology was developed for analyzing core samples, and a unit was created for the long-term exposure of samples to hydrogen. The results of lithological and petrographic studies of thin sections of the core samples are presented, along with a comparative analysis of the laboratory results concerning the porosity, permeability, and chemical composition of the rock matrix.

Methodology

The influence of hydrogen injection on the natural properties of reservoirs was studied using the Bobrikovskii terrigenous formations as a case study—a geological body designated for underground gas storage in the Volga-Ural oil and gas province. Due to the inability to use core samples from the At the Karashurskoe UGS, core samples were collected from a similar Bobrikovskii formation at one of the oil fields in the northeastern part of the Volga-Ural oil and gas province for laboratory experiments (Fig. 1). The core was extracted from sections with the highest porosity and permeability, specifically at a depth closest to the production zone in the Karashurskoe UGS, ranging from 1,488.4 to 1,489.8 meters. A total of twenty-four core columns were drilled from the original core material (Fig. 1), with half having standard dimensions (3 cm in length and diameter) and the other half featuring non-standard dimensions (6 cm in length and 3 cm in diameter). The non-standard samples were used to evaluate the stress-strain properties of the rocks through static testing before and after hydrogen exposure. Any samples displaying visible cracks were excluded from further

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analysis, resulting in a final total of twenty samples. After extraction, the samples underwent standard preparation, which involved soaking in an alcohol-benzene mixture for 20 days using a Soxhlet apparatus followed by drying. Figure 1 illustrates the prepared core samples, which were categorized into five groups of four samples each (Table 1). Each group contained two standard samples and two "long" samples. The intention is to compare the results of determining reservoir properties (porosity and permeability) as well as stress-strain properties (elastic modulus, Poisson's ratio, tensile and compressive strength) before and after hydrogen exposure for both the standard and "long" samples from each group.

Fig.1. Source core material and samples after drilling out

Table *1* Geometric characteristics of core samples and their porosity and permeability determined before and after exposure to hydrogen

Group number	Sample number	Length *h*, cm	Diameter *d*, cm	Before exposure to hydrogen		After exposure to hydrogen		Absolute change		Relative change	
				Kp, %	*Kper*, mD	*Kp*, %	*Kper*, mD	□*Kp*, %	□*Kper*, mD	□*Kp*, %	□*Kper*,%
1	1	6.03	3.00	22.4	720						
1	19	5.89	3.00	23.1	848	21.3	757	–1.8	–91	–7.79	–10.73
1	11/2	3.03	3.02	23.3	759						
1	2/1	3.01	3.00	22.4	739	22.3	692	–0.1	–47	–0.45	–6.36
2	4	5.89	2.99	22.4	664						
2	12	5.84	3.00	22.2	706	21.5	646	–0.7	–60	–3.15	–8.50
2	2/2	2.90	3.00	22.9	686						
2	14	2.96	2.99	22.9	686	22.5	644	–0.4	–42	–1.75	–6.12
3	6	6.03	3.01	21.3	604						
3	15	6.01	2.99	21.4	518	20.7	495	–0.7	–23	–3.27	–4.44
3	13	2.98	2.99	23.0	643						
3	5/1	2.87	2.99	22.8	644	21.1	603	–1.7	–41	–7.46	–6.37
4	7	5.97	2.99	22.1	612						
4	3	6.02	3.00	22.6	634	22.0	618	–0.6	–16	–2.65	–2.52
4	11/1	3.00	3.02	22.7	717						
4	8/1	2.98	3.00	22.4	669	19.4	603	–3.0	–66	–13.39	–9.87
5	16	5.9	2.99	22.3	496						
5	21	5.89	3.02	21.6	463	21.0	448	–0.6	–15	–2.78	–3.24
5	2/5	2.93	3.00	22.5	611						
5	8/2	2.91	3.01	22.3	655	21.7	625	–0.6	–30	–2.69	–4.58
Average value				22.4	656	21.3	612	–1.07	–44.6	–4.74	–6.46

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Table 2 outlines the comprehensive research program for the sample studies. The research consists of the following stages:

Stage 1: Following the standard preparation of the samples, their porosity and gas permeability were measured under conditions similar to atmospheric pressure: open porosity (Kp) and absolute permeability (Kper). The assessment of reservoir properties was conducted in accordance with GOST 26450.2-85 using a PIK-PP unit (AO Geologiya). To ensure accurate measurements, the samples were subjected to a minimum overburden pressure of 2.5 MPa, which prevented gas from flowing around the sides of the samples. This phase of the study aimed to identify variations in the reservoir properties of the core samples and assess the representativeness of the sample selection.

Table 2

Key stages of the research program, along with the equipment used and the parameters measured for each group of four samples.

Stage	Content of research stage	Unit used	Determined parameters
1	Samples are extracted and dried, open porosity and absolute gas permeability are determined at effective stresses close to atmospheric conditions (overburden pressure 2.5 MPa)	PIK-PP	***Kper, Kp***
2	Lithological and petrophysical studies of thin sections of core samples	Polarising microscope Leica DM 2700P	**Lithological and petrophysica l properties**
3	For three crushed samples, the chemical analysis of rock is performed, the com- position of the main rock-forming oxides: Fe2O3, MnO, TiO2, Al2O3, SiO2, CaO, MgO, Na2O, K2O, P2O5, Stot	Spectroscan MAKS-GV	**Mass fractions of oxides**
4	From each group, one “long” sample, one standard sample and part of the crushed core are placed in a cylinder, into which hydrogen is then injected. Sam- ples are kept in the cylinder for 7 days	Cylinder for injecting hydrogen and keeping samples	
5	For samples removed from the cylinder, the porosity and gas permeability are determined at effective stresses close to atmospheric conditions (overburden pressure 2.5 MPa)	PIK-PP	***Kper, Kp***
6	**For samples extracted from the cylinder, chemical analysis of the rock is per- formed, composition of the main rock-forming oxides: Fe2O3, MnO, TiO2, Al2O3, SiO2, CaO, MgO, Na2O, K2O, P2O5, Stot**	**Spectroscan MAKS-GV**	**Mass fractions of oxides**

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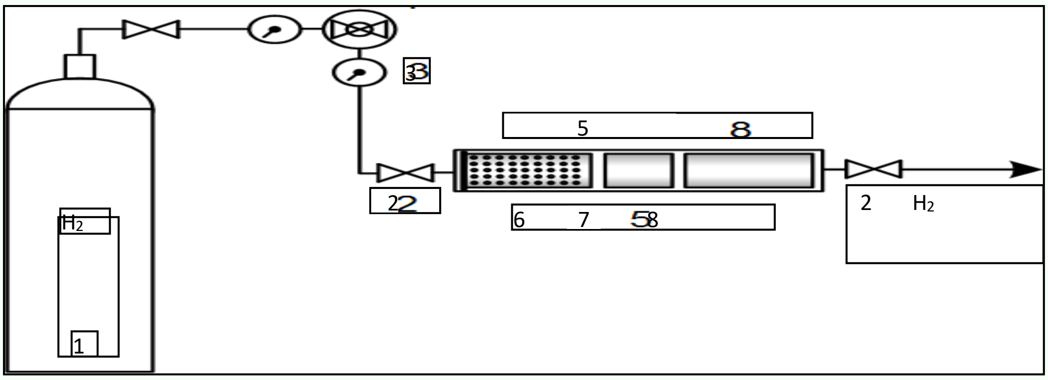
Stage 2: Identification of the minerals present in the rock being analyzed. Stage 3: Prior to hydrogen exposure, studies were carried out, and some samples were crushed and thoroughly mixed for further analysis. Three crushed core samples were examined, assuming their compositions would be similar due to the crushing and mixing process. Chemical analysis of the primary oxides was conducted without hydrogen influence, following GOST 5382-2019, specifically clause 7 (weight loss on ignition – LOI) and clause 23 (X-ray spectral method for element determination) using the MAKS-GV X-ray fluorescence spectrometer. Stage 4: The samples underwent prolonged exposure to hydrogen using a system that included a hydrogen cylinder, a pressure regulator with pressure sensors, and a sealed chamber (Fig. 2). Hydrogen was supplied from a compressed gas cylinder with a volume of 40 dm³ and a total gas volume of 6.3 m³ (Fig. 2). The hydrogen characteristics in the cylinder included:

- Hydrogen volume fraction (dry gas) of at least 99.99%;
- Total oxygen and nitrogen volume fraction not exceeding 0.01%;
- Water vapor mass concentration at 20 °C and 101.3 kPa not more than 0.2 g/m³;
- Cylinder pressure at 20 °C of 14.7±0.5 MPa.

Given the high pressure of the hydrogen, a regulator was attached to the cylinder, equipped with two sensors to monitor the gas pressure at the inlet and outlet. The reduced gas pressure was approximately 0.6-0.7 MPa. For the interaction with hydrogen, core samples were placed in a specially designed cylinder (Fig. 2) with openings for gas supply and removal. Each study group included one standard sample, one “long” sample, and a sample of crushed rock moistened with distilled water, contained in a sieve to prevent loss during gas flow. The regulator and cylinder were connected after a specific volume of hydrogen was introduced to displace the air. Following this, both valves were closed, allowing the rock to be exposed to hydrogen for seven days, with the gas being refreshed every 24 hours.



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In total, five groups of samples—comprising five standart, five "long," and five crushed rock samples—were exposed to hydrogen to assess changes in chemical composition. Stages 5 and 6: After hydrogen exposure, porosity and gas permeability were measured again, along with a chemical analysis of the primary oxides.

Fig. 2. Diagram of the unit for containing core samples exposed to hydrogen:

1 – Hydrogen cylinder;

2 – Valves;

3 – Pressure sensors;

4 – Pressure regulator with valve;

5 – Cylinder housing core samples;

6 – Sieve with crushed rock;

7 – Standard core sample (diameter: 3 cm, length: 3 cm);

8 – "Long" core sample (diameter: 3 cm, length: 6 cm).

Results:

The results for open porosity and absolute permeability of the samples (Stage 1), along with their geometric characteristics, are summarized in Table 1. The porosity of the samples ranged from 21.3% to 23.3%, while the permeability varied between 463 mD and 848 mD, with average values of 22.4% and 653.7 mD, respectively.

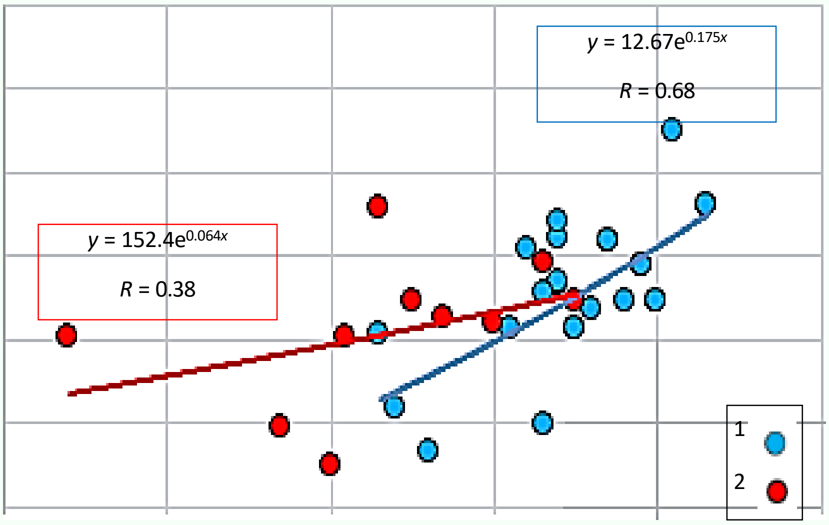

Fig.3. Dependence of permeability on porosity of core samples before (1) and after (2) exposure to hydrogen

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The correlation between absolute gas permeability and open porosity of the core samples is depicted in Fig. 3 (blue circles). This graph illustrates a strong relationship between these two characteristics, with a correlation coefficient of 0.68, indicating the homogeneity of the samples selected for analysis. Figure 4 presents images of some thin sections of the core samples studied (Stage 2). The analysis of these thin sections revealed the following key findings:- The samples are primarily composed of fine-grained silty sandstones with a quartz and feldspathic-quartz mineral composition. The rock structure is fine-grained silty-psammitic, with grain sizes ranging from 0.05 mm to 0.2 mm, and 10-30% of the grains are in the silty fraction measuring 0.1-0.16 mm. Grains are irregular, subisometric, elongated, and semi-rounded, with a microlayered texture resulting from the orientation of some elongated fragments. The rock primarily consists of quartz grains (82-95%), feldspar grains (up to 5%), and mica (up to 8%). The quartz grains exhibit an irregular elongated shape and may show signs of regeneration (0.003-0.015 mm). Some grains have dissolved around the edges, leading to uneven contours. Feldspar is mainly represented by plagioclase, which shows weak pelitization and partial dissolution. Mica includes muscovite blades and some chlorite flakes. The rock primarily features indentation cementation, which is a cement-free contact connection between quartz grains and fragments, characterized by a conformal structure. Accessory minerals include pyroxene grains measuring 0.08-0.18 mm. Authigenic minerals consist of isolated hydromica flakes smaller than 0.05 mm and calcite crystals measuring 0.25-0.3 mm (accounting for less than 1%). Post-sedimentation transformations have led to indentation structures and the formation of conformal structures due to quartz regeneration and the compaction of fragments

The void space within the rock is approximately 3-5% and is unevenly distributed, consisting of irregularly shaped, intergranular isolated pores of presumed secondary origin, ranging from 0.06 to 0.25 mm in size. Articles [21, 31, 32] indicate that quartz particles, under the mining and geological conditions of the UGS, show minimal interaction with hydrogen.

The results of chemical analysis of the rock (stage 3) are presented in Table 3 and Fig.5. As can be seen from these data, the samples have the chemical composition characteristic of a terrigenous reservoir. Figure 5 shows the averaged composition of oxides for three samples of crushed rock, while the composition with and without silicon oxide is plotted separately, since its content is much higher than

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that of other oxides. The chemical composition of the investigated samples is dominated by silicon oxide, the amount of which is on average 96.64 %, the amount of other oxides, together with weight loss on ignition, is 2.01 % (Fig.5, *a*).

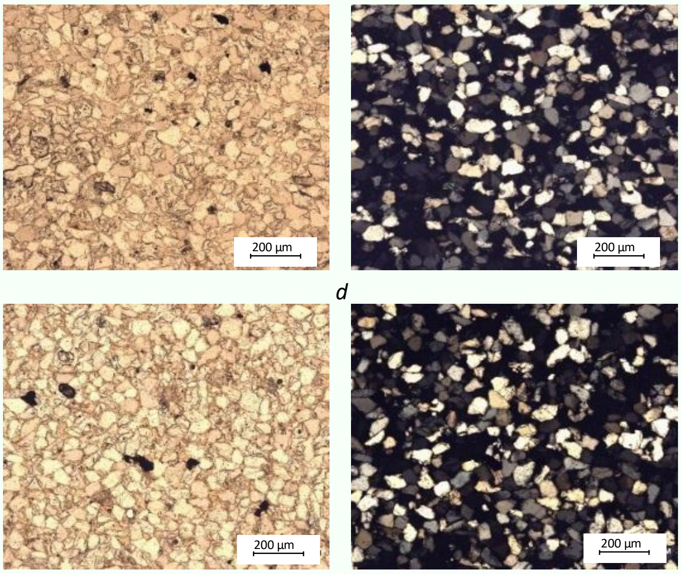

Fig.4. Photos of the investigated thin sections of core samples N 1 (*a*, *b*) and N 4 (*c*, *d*): *a*, *c* – without analyzer; *b*, *d* – with analyzer

Table 3 **Results of the chemical composition analysis of rocks before and after hydrogen exposure.**

Sample number	Content in rock, %											
	Mass fraction of a chemical element in terms of oxide											LOI
	Fe – Fe_2O_3 tot	Mn – MnO (II)	Ti – TiO_2	Al – Al_2O_3	Si – SiO_2	Ca – CaO	Mg – MgO	Na – Na_2O	K – K_2O	P – P_2O_5	S – Stot	
						Before exposure to hydrogen						
1	0.81	< 0.01	0.28	< 0.1	96.44	0.32	0.13	0.20	0.22	0.02	0.23	1.08
2	0.77	< 0.01	0.28	< 0.1	96.83	0.29	0.03	0.22	0.21	0.02	0.22	1.09
3	0.75	< 0.01	0.24	< 0.1	96.65	0.25	0.02	0.06	0.20	0.02	0.25	1.10
Average value	**0.78**		**0.27**		**96.64**	**0.29**	**0.06**	**0.16**	**0.21**	**0.02**	**0.23**	**1.09**
						After exposure to hydrogen						
1	0.98	0.01	0.28	< 0.1	95.01	0.46	0.11	0.14	0.25	0.02	0.29	2.58
2	0.72	< 0.01	0.24	< 0.1	95.73	0.25	0.16	0.16	0.14	0.02	0.21	1.18
3	0.75	0.01	0.23	< 0.1	97.62	0.31	0.10	0.11	0.14	0.02	0.18	1.26
4	0.82	< 0.01	0.25	< 0.1	95.64	0.24	< 0.1	0.22	0.21	0.02	0.32	1.23
5	0.72	< 0.01	0.21	< 0.1	97.11	0.28	< 0.1	0.25	0.26	0.02	0.44	1.13
Average value	**0.80**	**0.01**	**0.24**		**96.22**	**0.31**	**0.12**	**0.18**	**0.20**	**0.02**	**0.29**	**1.48**
Absolute change	**0.02**		**–0.02**		**–0.42**	**0.02**	**0.06**	**0.01**	**–0.01**	**0.00**	**0.06**	**0.39**

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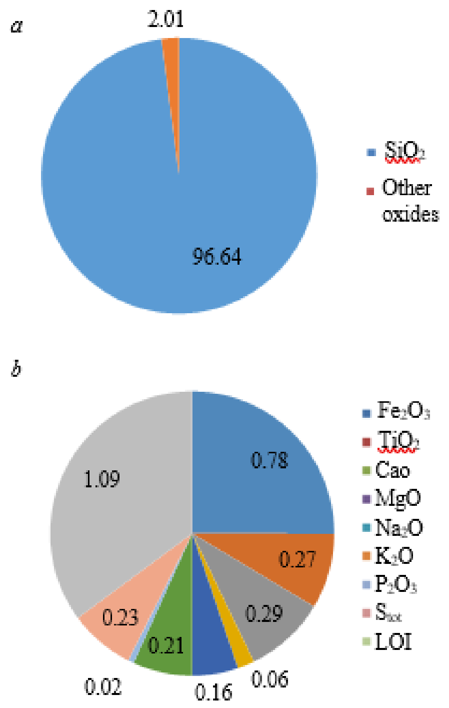

Fig.5. Results of determining the chemical composition of samples before exposure to hydrogen: *a* – all oxides, except silicon, are combined into one value; *b* – silicon oxide is excluded

As shown in Fig. 5b, iron oxide ranks second in abundance after silicon oxide, with a composition of 0.78%. The samples also contained the following oxides in descending order: calcium oxide (0.29%), titanium oxide (0.27%), sulfur oxides (0.23%), potassium oxide (0.21%), sodium oxide (0.16%), magnesium oxide (0.06%), and phosphorus oxide (0.02%).

After prolonged exposure of the samples to hydrogen (Stages 4 and 5), changes in reservoir properties can be compared before and after exposure (refer to Table 1, Fig. 3 with red dots, and Fig. 6). To facilitate comparison, each graph in Fig. 6 includes a dotted line representing equal values. By comparing the experimental values to this line, one can easily determine whether a specific characteristic has increased or decreased following hydrogen exposure. Points below the line indicate a decrease in the rock property, while points above the line indicate an increase. Additionally, Fig. 6 features a linear approximation function starting from the origin. The coefficient for the variable x in this function helps quantify the extent of the changes in each characteristic. Table 3 displays the results of the chemical composition analysis of the rock before and after exposure to hydrogen (Stage 6). For easier data interpretation, Fig. 7 illustrates the composition of the main oxides in percentage and their absolute changes.

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Discussion of Results

Comparing the porosity and permeability results of core samples before and after hydrogen exposure (see Fig. 5) indicates that after exposure

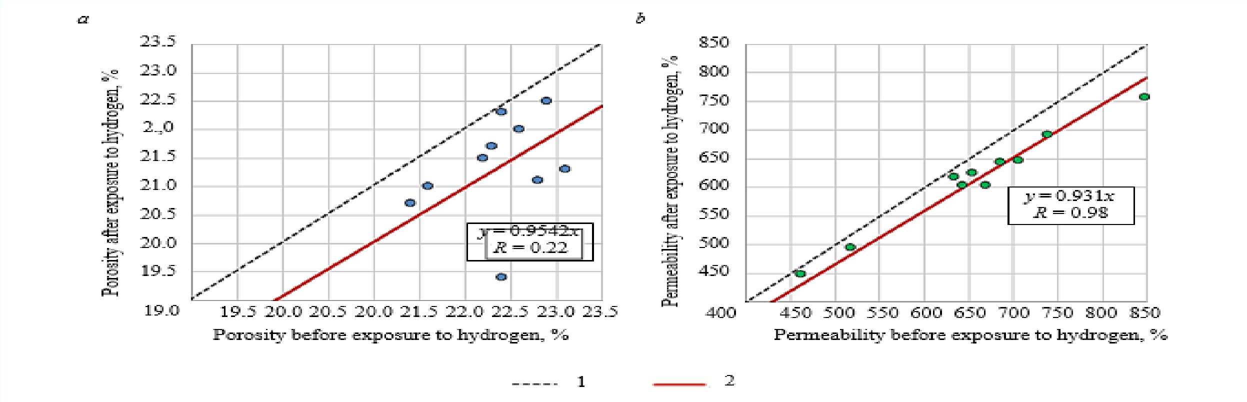

Fig.6. Comparison of porosity (a) and permeability (b) of core samples before and after exposure to hydrogen
1 – line of equal values; 2 – approximation

Fig. 6. Comparison of porosity (a) and permeability (b) of core samples before and after hydrogen exposure:1 – Line of equal values; 2 – Approximation line.

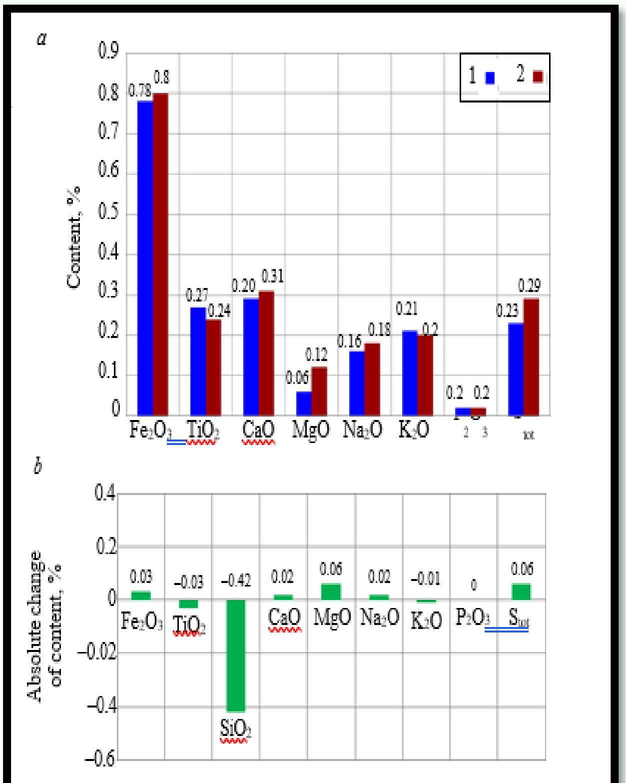

Fig.7. Average composition of basic oxides (silicon oxide excluded) before and after exposure to hydrogen (a) and an absolute change in their contents after exposure to hydrogen (b) in the total mass of the sample
1 – before exposure; 2 – after exposure

Exposure to gas resulted in a decrease in porosity and permeability by 4.6% and 7.9%, respectively. This indicates that the samples became more compacted after hydrogen exposure. According to the authors, this compaction occurred due to the weakening of the rocks under hydrogen's influence. Additionally, when measuring the porosity and permeability of the samples after this exposure, an overburden pressure of 2.5 MPa caused further compaction, even though the samples had already experienced compressive loading during the initial measurements before

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hydrogen exposure. It is likely that this exposure compromised the strength of the intergranular contacts, leading to a weakening of the rock matrix. The correlation between changes in both porosity and permeability after hydrogen exposure is understandable, as changes in the volume of void space affect rock compressibility. This, in turn, results in variations in porosity and permeability in response to changes in effective stress. In addition to the correlation trends illustrated in Fig. 6, it was observed that the relationship between permeability and porosity changed significantly after hydrogen exposure (see Fig. 3): the correlation coefficient decreased, and the variability in permeability values increased, indicating that permeability did not decline significantly with reduced porosity.

To sum up, it is important to examine the results of the chemical analyses of crushed rock samples before and after hydrogen exposure (Table 3, Fig. 7). Figure 7 presents the average values of basic oxides for three samples prior to hydrogen exposure and five samples afterward. The changes in chemical composition before and after exposure are minimal, with some values increasing and others decreasing. The exception is silicon oxide, which remains at a maximum concentration in the rock. Overall, these changes could be attributed to variations in sample composition or measurement errors. Thus, the comparison of chemical analysis results indicates that hydrogen's effect on the chemical composition of core samples is minor. This suggests that the geological formation studied (the Bobrikovskii horizon) could be suitable for storing a hydrogen-methane mixture. However, this conclusion would require further verification through more detailed studies involving a larger sample size and longer hydrogen exposure (up to one month or more).

Conclusion:

This study examines the methodology and findings from laboratory investigations into the effects of hydrogen exposure on the reservoir properties and chemical composition of terrigenous reservoir rocks, focusing on the Bobrikovskii horizon in the Volga-Ural oil and gas region. The main conclusions are as follows:

- The authors developed an experimental unit and a specialized program for analyzing core samples, allowing for the examination of reservoir properties, density, dynamic characteristics, stress-strain behavior, and chemical composition before and after hydrogen exposure. The methodology was tested on terrigenous core samples from the Bobrikovskii formation in a Volga-Ural oil field.

- Lithological and petrophysical analyses revealed that the core samples were predominantly composed of quartz grains, with minor amounts of feldspar and muscovite.
- The experiments assessed the reservoir properties of the core samples before and after hydrogen exposure, showing a decrease in porosity and permeability by 4.6% and 7.9%, respectively.
- The authors attribute the reduction in porosity and permeability to the weakening of rocks due to hydrogen exposure. Additionally, when measuring these properties after exposure and applying an overburden pressure of 2.5 MPa, further compaction occurred, despite prior compressive loading during initial measurements. The hydrogen exposure likely disrupted the strength of intergranular contacts, contributing to rock weakening. It is important to note that these reductions in porosity and permeability are not substantial enough to significantly impact gas injection and removal, especially since hydrogen is much more mobile than natural gas.
- Comparing the chemical analysis results for basic oxides indicated that changes in the chemical composition of rocks before and after hydrogen exposure were minimal, possibly due to sample composition variations or measurement errors. These findings suggest that the formation is chemically resistant to hydrogen, supported by the high silicon oxide content (96.64%), which does not interact with hydrogen under the studied conditions.
- Overall, the analyses of porosity, permeability, and chemical composition demonstrate that hydrogen's influence on the reservoir rock is negligible. The Bobrikovskii horizon in the Volga-Ural oil and gas province appears suitable for storing a hydrogen-methane mixture. However, further validation is needed through more comprehensive studies involving a larger number of samples and extended hydrogen exposure.

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