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Scientific paper

The porosity and nuclear magnetic characteristics of artificial samples with the equal grain size

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Abstract. ***Relevance.*** The nuclear magnetic resonance relaxometry is a fairly new method for well logging and laboratory investigations. It is applied for determining petrophysical properties, e.g., porosity, permeability, water- and oil-saturation. However, the nuclear magnetic resonance relaxometry data interpretation rests on the representation of the pore space as a set of spheres of various diameters that fundamentally does not correspond to the structure of granular rocks. ***Aim.*** To examine an influence of grain size on the measurements results of nuclear magnetic resonance relaxometry porosity and compare with other conventional methods. ***Objects.*** Artificial samples made with various sand and quartz glass beads fractions. ***Methods.*** Water-saturation, gas-volumetric and nuclear magnetic resonance relaxometry were applied to determine porosity. X-ray diffraction method was used to clarify the mineralogical composition; to measure the grain size a laser particle size analyzer was used, and the grain shape was investigated in polished specimens. ***Results.*** The paper describes the results of porosity measurements with different methods for artificial samples. We observed an effect of underestimated porosity acquired with nuclear magnetic resonance relaxometry method for consolidated and unconsolidated samples with grain size less than 0.160 mm. The investigation of sand composition with the X-ray diffraction method and additional assessment of the particles shape and size led to conclude that these factors are not the cause of the underestimated porosity effect. Additional statistical samples and using of the other nuclear magnetic resonance relaxometer presented the reparability of previous results. Based on the research results, it was assumed that the cause of the underestimated porosity effect is the complex shape and increased specific surface area of the samples, which grows as the particle size decreases.

Keywords: porosity, grain size, artificial samples, petrophysical properties, nuclear magnetic resonance relaxometry, gas-volumetric method, water-saturating method

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Научная статья

Пористость и ядерно-магнитные характеристики искусственных образцов с одинаковым размером зерен

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Аннотация. ***Актуальность.*** Ядерно-магнитная резонансная релаксометрия является относительно новым методом каротажа и лабораторных исследований. Он применяется для определения петрофизических свойств, например, таких как, пористость, проницаемость, насыщенность водой и нефтью. Однако интерпретация данных ядерно-магнитной резонансной релаксометрии подразумевает представление порового пространства как набора сфер различного диаметра, что не соответствует структуре терригенных пород. ***Целью*** исследования было изучение влияния размера зерна на результаты измерения пористости ядерно-магнитной резонансной релаксометрией и сопоставление с другими стандартными методами. ***Объектом*** исследования являются искусственные образцы из различных фракций песка и стеклянных кварцевых шариков. ***Методы.*** Для определения пористости применяли метод жидкостенасыщения, газоволюметрический метод и ядерно-магнитную резонансную релаксометрию. Рентгенофазовый метод использовался для уточнения минералогического состава; для оценки размера зерен применялся лазерный анализатор размеров частиц, форма зерен исследовалась в шлифах. ***Результаты.*** Описаны результаты измерений пористости различными методами на искусственных образцах. Мы наблюдали эффект занижения пористости, измеренный методом ЯМР для консолидированных и неконсолидированных образцов с размером зерна менее 0,160 мм. Исследование состава песка рентгенофазовым методом и дополнительная оценка формы и размера зерен позволили сделать вывод, что эти факторы не являются причиной данного эффекта. Дополнительные статистические выборки и использование другого ЯМР-релаксометра подтвердили повторяемость предыдущих результатов. По результатам исследований сделано предположение, что причиной эффекта занижения пористости является сложная форма и повышенная удельная поверхность зерен образцов, которая увеличивается с уменьшением размера зерен.

Ключевые слова: пористость, размер зерен, искусственные образцы, петрофизические свойства, ядерно-магнитная резонансная релаксометрия, газоволюметрический метод, метод жидкостенасыщения

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Introduction

The filtration and capacitance properties of rocks are an important object of study in the design and development of oil and gas fields. Quantitative assessment of properties such as porosity, permeability, saturation, allows you to decide on the feasibility of creation of a well. Today there are many logging and laboratory methods that have their advantages and disadvantages. One of these is the method of nuclear magnetic resonance (NMR) relaxometry.

The NMR relaxometry method is a relatively new method for studying petrophysical properties of core samples and drill cuttings. The first borehole measurements were carried out in 1960. Further development of technologies that increase the resolution and sensitivity of instruments has made it possible to develop the NMR relaxometry method in our time [1, 2].

Reservoir properties are known to be determined by the structure of the pore space for terrigenous rocks. It depends on the shape and size of the grains composing these rocks. The NMR relaxometry method makes it possible to evaluate not only such petrophysical properties as porosity [3, 4], permeability [1], water and oil saturation, but also the structure of the pore space [5–7] – the size of the sample pores. However, the data interpretation model assumes pores in the form of spherical or cylindrical inclusions, which is inadequate model true. In addition, it is believed that the NMR relaxometry method depends only on the properties of the fluid saturating the rock and is limited by the sensitivity of the instrument [1, 8].

In work [9], we found an underestimated porosity effect using the NMR relaxometry method for artificial sand samples in comparison with other conventional methods. Furthermore, we examined the maximum of the transverse relaxation time and their shifting after the creation of residual water saturation:

- average displacement is 455 ms (with an average maximum of 517 ms) for samples of the large fraction;
- average displacement is 134 ms (with an average maximum of 183 ms) for samples of the medium fraction;
- average offset is 33 ms (with an average maximum of 40 ms) for samples of the fine fraction.

In foreign and Russian sources, no mention was found of the underestimated porosity effect with the NMR relaxometry method in fine-grained and silty samples. Conversely, many studies indicate a good correlation of this method with conventional methods for determination of porosity [3, 4, 10], including for fine-grained fractions [11, 12]. At the same time, researchers note the need to consider surface relaxivity [13] and clay content during studying with NMR, since it affects not only the echo signal, but also the spectrum of time distribution [14–16]. The work [17] describes a shift in relaxation times towards their decrease due to an increase in the clay content of artificial

samples consisting of sand and clay material. All these works are united by the conclusion about the importance of the specific surface area during studying core samples.

Materials and methods

The samples collections

We used petrophysical methods (liquid saturation, gas-volumetric and nuclear magnetic resonance relaxometry) for three suites of sample collections – artificial consolidated sand samples, artificial unconsolidated sand samples and artificial unconsolidated quartz glass beads samples.

To create collections of sand samples ordinary river sand was used. This sand was dried with oven and sifted with a sieving machine. As a result, we had got 11 fractions with grain size ranging 1–0.040 mm.

To create consolidated samples, the mixture of different granulometric sand compositions with cryogel (10% solution of polyvinyl alcohol) was used. Cryogel is an aqueous solution of polyvinyl alcohol, which has the ability to transform from a viscous flow state into structured elastic bodies. This gradual transformation is conducted by passing through freeze-thaw cycles through a threshold value of 0°C. Using an empirical method, it was found that five cycles are enough to consolidate sand. Currently, cryogel is used mainly in the conditions of the Far North to strengthen road slopes [18], soils [19], as well as in the construction of structures and pipelines in the arctic regions [20].

To create the samples, cylindrical forms with a diameter and length of ~3 cm were used so that the volume of the samples was close to the volume of standard core samples and a calibration sample for NMR measurements. The cryogel volume was about 35% of the volume of the form. The mixture was stirred until a homogeneous mass, after which it was laid out in layers in the form. With each portion, sand and cryogel had to be compacted to avoid the formation of voids and air bubbles. Then the samples went through five freeze-thaw cycles and were dried. According to standard methods for measuring porosity and permeability using the gas-volumetric method with a porosimeter, the ends of the samples under study must be smooth and strictly perpendicular to the sides of the samples. Therefore, the ends of the samples were worked up with a grinding machine. The number of samples of each fraction and their characteristics are presented in Table 1.

For unconsolidated samples, fluoroplastic cylindrical forms (with volume ~15 cm^3) were designed and made. The paper filter (cell size is 8–12 micrometers) on the end of this forms helped to avoid spilling sand out and allow sand saturation. To achieve better sand compressibility and prevent high porosity due poor packing, sand was mixed with water in an amount of 35% of the volume of the form. Then, the mixture was placed into the form in layers and compacted to avoid the forming of air bubbles and voids. The samples were subsequently dried and prepared for further measurements. The unconsolidated samples characteristics are shown in Table 2.

Table 1. *Consolidated samples characteristics*

Таблица 1. *Характеристики консолидированных образцов*

Fraction size, mm Размер фракции, мм	Fraction name Название фракции	Number of samples in collection Количество образцов в коллекции
1–0.630	Coarse-grained sand Крупнозернистый песок	3
0.630–0.315	Coarse- and medium-grained sand Крупно-среднезернистый песок	3
0.315–0.250	Medium-grained sand Среднезернистый песок	3
0.250–0.200	Fine-grained sand Мелкозернистый песок	3
0.200–0.160		3
0.160–0.125		4
0.125–0.100		3
0.100–0.063	Silt (very fine-grained sand) Алевриты	3
0.063–0.050		3
0.050–0.040		2

Table 2. *Unconsolidated samples characteristics*

Таблица 2. *Характеристики неконсолидированных образцов*

Sample no. Номер образца	Fraction size, mm Размер фракции, мм	Fraction name Название фракции
1	1–0.630	Coarse-grained sand Крупнозернистый песок
2	0.630–0.315	Coarse- and medium-grained sand Крупно-среднезернистый песок
3	0.315–0.250	Medium-grained sand Среднезернистый песок
4	0.250–0.200	Fine-grained sand Мелкозернистый песок
5	0.200–0.160	
6	0.160–0.125	
7	0.125–0.100	
8	0.100–0.063	Silt (very fine-grained sand) Алевриты
9	0.063–0.050	
10	0.050–0.040	

To examine references consisting of smooth spherical grains, quartz glass beads of different granulometric compositions were used. Since it was necessary to explore only porosity using the NMR and gravimetric methods, jars with a volume close to the calibration NMR sample were used. To compare the results, the same samples were examined, but consisting of grains of sand, which made up the structure of the pore space from non-smooth grains of different non-ideal shapes.

The samples were saturated according to standard methods under vacuum [21]. Then, the samples were ready for porosity measurements with the NMR relaxometry method. Table 3 shows the sand and quartz glass fractions that overlapped and were compared with each other.

Table 3. *Fractions of quartz glass beads and corresponding sand fractions*

Таблица 3. *Фракции кварцевого стекла и соответствующие им фракции песка*

Fraction sizes of samples with quartz glass beads Размер фракции образцов из шариков кварцевого стекла	Fraction sizes corresponding sand samples Размеры фракций, соответствующие песчаным образцам
mm/мм	
0.500–0.600	0.315–0.630
0.250–0.500	0.315–0.630
	0.250–0.315
0.100–0.250	0.200–0.250
	0.160–0.200
	0.125–0.160
	0.100–0.125
0.070–0.110	0.063–0.100
0.063–0.073	0.063–0.100
0.040–0.070	0.050–0.063
	0.040–0.050
0.000–0.050	0.040–0.050

Nuclear magnetic resonance relaxometry

Nuclear magnetic resonance rest on the reaction of atomic nuclei to an external magnetic field. Due to the presence of a magnetic moment and spin in the nuclei, they precess around the vector of the acting magnetic field. Therefore, when exposed to an external magnetic field, these precessing magnetic nuclei can produce signals that can be measured [1].

According to the theory, NMR measurements can be carried out on any nuclei with an odd number of protons and neutrons. These include a hydrogen, carbon, sodium. For the most nuclei found in terrestrial formations, the nuclear magnetic signal induced by the external magnetic fields is small. However, hydrogen (which has only one proton) allows observing a fairly strong signal due to its relatively large magnetic moment and wide distribution in fluids saturating rocks [22].

The NMR experiment and obtaining the relaxation curve can be divided into three stages:

- sample magnetization over a period of time Tw by exposure to a constant magnetic field B_0 (T_1 time);
- turning of the macroscopic magnetization vector M_0 into a transverse plane perpendicular to vector B_0 using a 90° pulse of alternating magnetic field B_1;
- learning the relaxation curve as a result of applying a sequence of 180° pulses supplied at TE time intervals (T_2 time).

There are relationships between petrophysical properties and NMR characteristics. Three processes are involved in relaxation of fluids located in the pore space of rocks [1]:

- Volumetric processes in fluids affecting T_1 and T_2. It depends on chemical and physical properties of saturating fluid.
- Surface relaxation affecting T_1 and T_2. It depends on a pore space – special surface area – and relaxivity parameter depending on mineralogical composition.
- Diffusion in the presence of magnetic field gradients affecting T_2. It depends on NMR instrument.

In our case we examined only T_2 time – transverse relaxation time. Since we used the same saturating fluid and the same NMR instrument without magnetic field gradient, we can assume that NMR characteristics depends only the surface relaxation phenomena [23]:

$$\frac{1}{T_{2\,\mathrm{surface}}} = \rho_2 * \left(\frac{S}{V}\right)_{\mathrm{pore}}, \quad (1)$$

where T_2 surface is the transverse relaxation time of the pore fluid associated with relaxation on the grain surface; ρ_2 is the surface relaxivity; S/V is the pore surface area to the pore volume ratio (the specific surface area).

From formula (1) it follows that in large pores a small S/V ratio leads to increase in relaxation times. Conversely, in small pores this ratio is high, and therefore relaxation times are shorter. A detailed illustration is presented in the Fig. 1. As can be seen from point A, the smaller the pore filled with fluid becomes, the faster the relaxation curve decays, which means the transverse relaxation time also shifts towards decreasing times. If we consider different pore simultaneously, then the relaxation curve is a combination of all pores separately. This fact is used in mathematical processing.

Water-saturation method

The water saturation method [21] is applied for determining the porosity coefficient. It is based on measuring the sample mass in air and the saturating fluid (in our case – water). Initially, the samples are dried and weighed in air (M_1). Then, they are saturated with water under the vacuum created by a pump for several days and weighed in air (M_2). Finally, mass in the fluid gives a summand M_3. The porosity coefficient is known to be the pore volume to the sample volume ratio and it can be calculated from (2):

$$\varphi = \frac{M_3 - M_1}{M_3 - M_2} * 100\%. \quad (2)$$

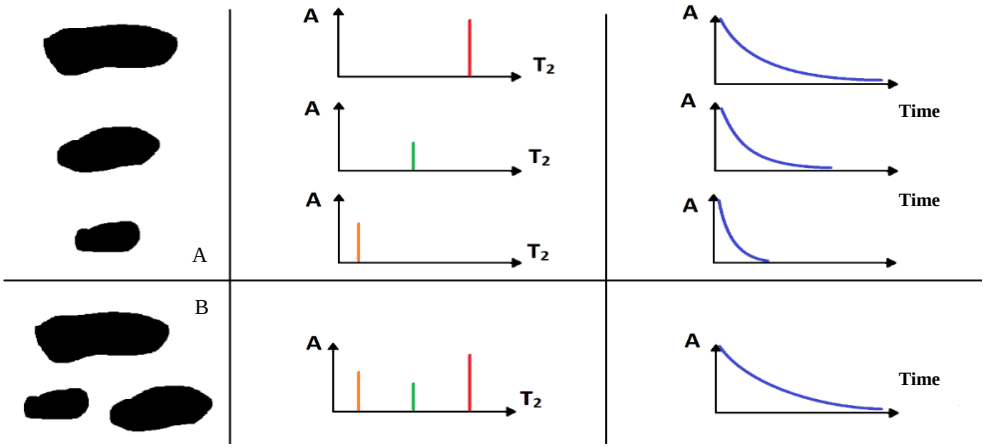

Fig. 1. *Form of a relaxation curve and transverse relaxation time T_2 depending on a pore size: A) separately; B) in combination [23]*
Рис. 1. *Вид релаксационной кривой и времени поперечной релаксации T_2 в зависимости от размера пор: А) по отдельности; В) в комбинации [23]*

Gas-volumetric method

The gas-volumetric method is based on the Boyle–Marriotte's Law. It describes the relationships between pressure and volume of a confined gas with a constant temperature [24] (3). Before an experiment the instrument need a calibration. Specific metallic sample is placed into the camera and compressed with a rubber coupling to eliminate gas leak. Therefore, we get P_1 and V_1. Then, an investigated sample is put into the camera to get P_2 and V_2 after reaching pressure equilibrium. The result volume consists of the calibration volume and the pore volume. In the research we used the porosimeter "AP-608".

$$P_1 * V_1 = P_2 * V_2 . \quad (3)$$

Results and discussion

In [9] we found a phenomena during porosity measurements with the NMR relaxometry method of collection artificial sand samples consolidated with cryogel. For the samples with grain size less than 0.200 mm porosity acquired with saturation and gas-volumetric methods distinguish from NMR. Firstly, to prevent an influence of cryogel, we examined porosity of unconsolidated samples consisted of the same sand fractions. The measurement results are showed in Fig. 2. There is a decrease in porosity towards the re-duce of grain size less than 0.160 mm.

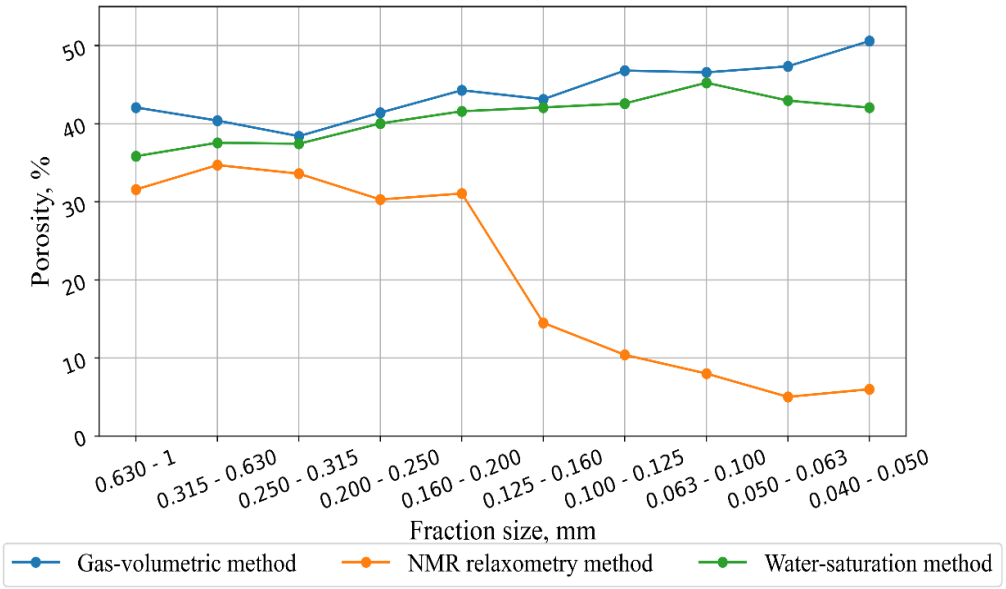

Fig. 2. *Porosity of the unconsolidated samples measured with various methods*
Рис. 2. *Пористость неконсолидированных образцов, измеренная разными методами*

The difference between the NMR porosity and the gas-volumetric porosity is about 22% for samples with the grain size more than 0.160 mm and 80% for the samples with the grain size less than 0.160 mm. In the previous research this difference was 30 and 70%, respectively.

Further examining of the underestimated porosity effect led to the study of the particle size and lithological composition of sand fractions. Fractions sizes yielded during sieving were confirmed with the laser diffraction method. Median and average grain sizes correspond to the fraction sizes ranges. The study of sand polished specimens with an electron microscope allowed considering grain forms. The shape of the grains changes from rounded to angular as the size decreases. This is consistent with theory described in [25, 26].

It was established with X-ray diffraction method that quartz significantly predominates in all sand fractions. Plagioclase and potassium feldspar are present in subordinate amounts. In addition, chlorite, dolomite, calcite, mica were like impurities. Finally, there are no magnetic minerals in the sand.

Quartz glass beads samples have a pore space structure where the shape of each grain is close to spherical, in contrast with sand samples with rough and irregularly shaped grains. The effect of underestimated porosity with the NMR relaxometry method was not detected on model samples (Fig. 3). In addition, the results are in good agreement with the weight method (an average difference is 5%). For the same bulk sand samples made using the method described in section Materials and methods the effect of underestimated porosity with the NMR was detected for samples with grain sizes less than 0.160 mm. The difference from the gravimetric method is on average 80%. For the samples larger than 0.160 mm, this difference between two methods is on average 10%.

Since cryogel has no effect on porosity studies, the next collection of samples was consolidated with cryogel due to the work convenience. There were an average of three samples per fraction (Table 1). The main goal of creation of this collection is to increase the amount of statistical material and see the repeatability of the effect.

The results of examining the porosity of these samples showed the repeatability of the effect of underestimation of values with the NMR relaxometry method for samples of fine-grained and silty fractions (Fig. 4). The errors shown in the figure reflect the difference in values from sample to sample within the same fraction. The difference in the porosity values according to the gas-volumetric and NMR relaxometry methods for samples with grain sizes larger 0.160 mm was on average 21%, for samples smaller than 0.160 mm – on average 80%.

Thus, the studies showed the repeatability of the results obtained in the previous works. In all sample collections, the effect of underestimated porosity with the NMR relaxometry method was detected, in contrast to the gas-volumetric method and the water-saturation method. The difference in values is 76±5% for samples with a grain size less than 0.160 mm and 25±5% for samples with a grain size greater than 0.160 mm.

Experiments were carried out with other NMR relaxometry with working frequency 12 MHz (the previous NMR instrument had working frequency 2.2 MHz) to ensure that the used instrument did not affect the results. The experiments were carried out on the existing samples. It can be seen that the behavior of the curve for the new NMR instrument is similar for particles with a particle size less than 0.160 mm (Fig. 5). For the rest, there is a good comparison with conventional methods.

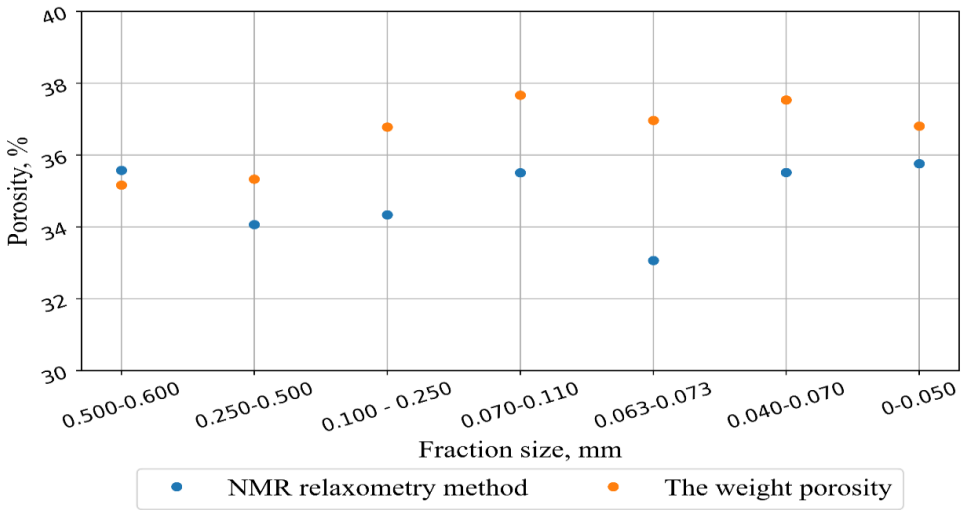

Fig. 3. *Comparison of the porosity with weight method and NMR relaxometry method for the quartz glass beads samples*
Рис. 3. *Сопоставление пористости по весовому методу и методу ЯМР релаксометрии для образцов из шариков кварцевого стекла*

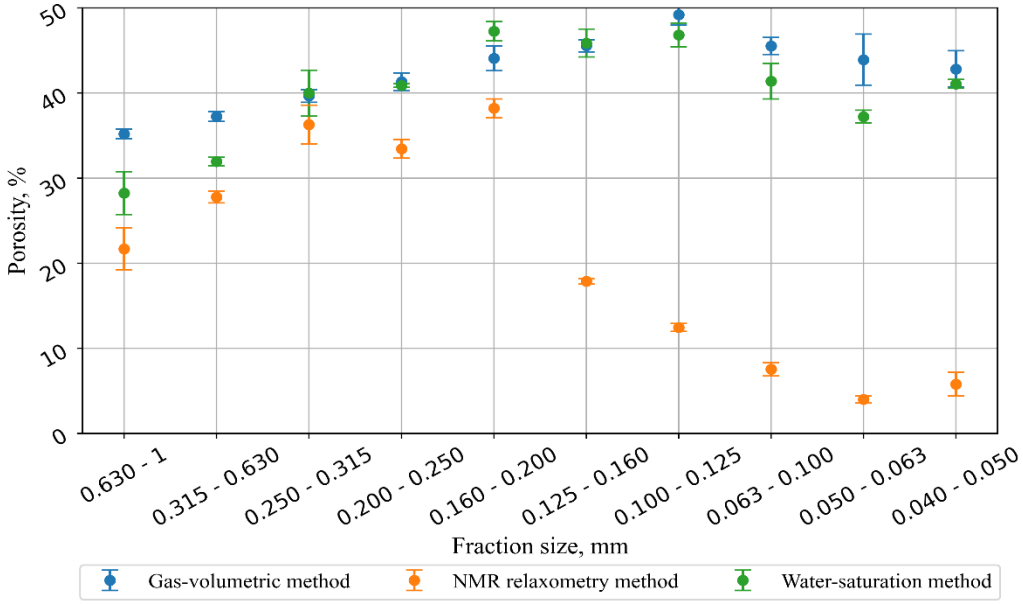

Fig. 4. *Porosity of the consolidated samples measured with various methods*
Рис. 4. *Пористость консолидированных образцов, измеренная разными методами*

Exploration of the shift of the maximum transverse relaxation times of saturated samples with water after centrifugation at a speed of 2500 rpm showed that:

- average offset is 362 ms (with an average maximum of 565 ms) for coarse samples. In previous work it was 455 ms;
- average offset is 149 ms (with an average maximum of 191 ms) for the medium fraction samples. In previous work it was 134 ms;
- average offset is 27 ms (with an average maximum of 30 ms) for fine samples. In previous work it was 33 ms.
- All of the above allows us to draw some conclusions. To exclude the cryogel impact on porosity measured with NMR, the unconsolidated collection of samples were created. The results demonstrate the repeatability of the underestimated porosity effect. Hence, the cryogel was not the reason of this effect. The same result was acquired on the additional collection. It helped to ensure the repeatability of the results.

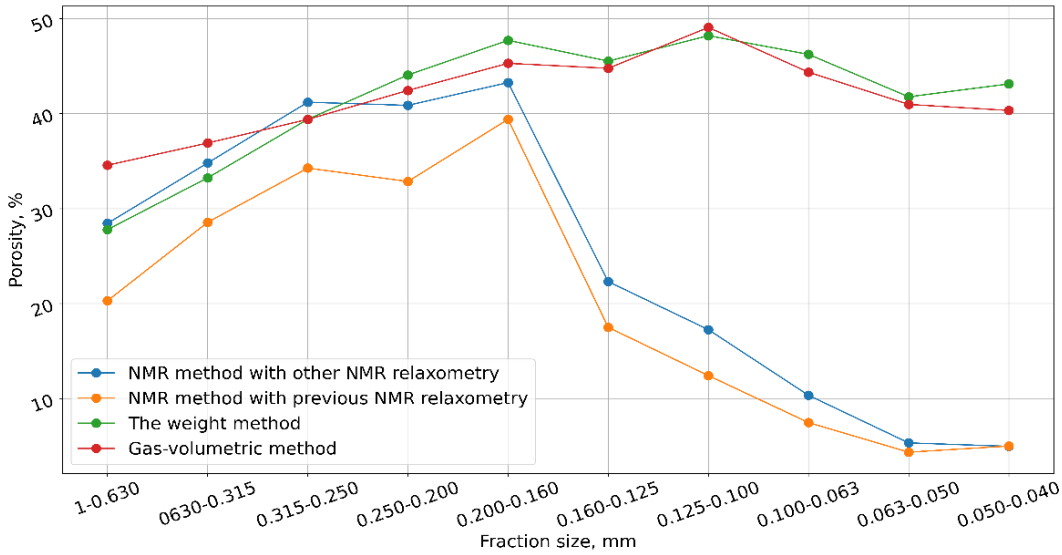

Fig. 5. *Results of porosity measurements with two NMR instruments (the blue line – 12 MHz; the orange line – 2.2 MHz) and matching with conventional methods*
Рис. 5. *Результаты измерения пористости двумя ЯМР приборами (синяя линия – 12 МГц; оранжевая линия – 2,2 МГц) и сопоставление со стандартными методами*

The examining of the mineralogical composition of sand, the size and shape of grains demonstrated constancy of the composition. All fractions have the same kinds of minerals. The shape of the grains changes from rounded to angular as the size decreases. Moreover, angular grains have the high specific surface area. This is consistent with theory described in [25, 26]. According to Formula (1) the specific surface area affects directly the NMR measurements. This is indirectly confirmed by the works [14, 17]. They demonstrated the impact of the amount of a clay material and its mineralogical composition on the NMR characteristics. In our case, we assume the constancy of the clay kind because of the using the different fractions of the same sand.

The indirect additional evidences of the influence of the specific surface area are:

- measurements result with the NMR of collections samples created with quartz glass beads. The coincidence of NMR and the gravimetric method allow assuming that the quartz glass beads have small specific surface area because of spherical grain shape without any angular biases;
- repeatability of the results of the carried out measurements on the additional NMR instrument. Furthermore, it confirms the initial NMR instrument was not the reason of the effect of underestimated porosity.

Conclusion

The investigations were aimed at searching for the dependence of nuclear magnetic characteristics on the size of the grains composing the sample, as well as investigating the reasons for the effect of underestimated porosity values obtained using the NMR relaxometry method in comparison with other methods.

In all sample collections, the effect of underestimated porosity with the NMR relaxometry method was detected, in contrast to the gas-volumetric method and the water-saturation method. The difference in values is 76±5% for samples with a grain size less than 0.160 mm and 25±5% for samples with a grain size greater than 0.160 mm. The repeatability of the results of studying the shift of maximum relaxation time and the effect of underestimated porosity with the NMR relaxometry method was achieved.

After all investigations of possible reasons of the underestimated porosity, we assume that the reason of the underestimated porosity with the NMR relaxometry method is the specific surface area. Unfortunately, due to technical difficulties, we have not yet been able to measure this parameter. In further research, we plan to pay special attention to this and conduct experiments with different amounts of clay material.

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