RESEARCH ON PETROPHYSICAL PROPERTIES OF CHOSEN SAMPLES FROM THE POINT OF VIEW OF POSSIBLE CO_2 SEQUESTRATION

VÝZKUM KOLEKTORSKÝCH VLASTNOSTÍ VYBRANÝCH HORNINOVÝCH VZORKŮ Z HLEDISKA MOŽNÉ GEOSEKVESTRACE CO_2

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Abstract

Man-made CO_2 emissions (the so called anthropogenic CO_2 emissions) and their increasing trend can be, by some scientists, considered a serious menace for the sustainable development of mankind, and their reduction a prerequisite for the environment protection. Carbon dioxide is one of the most important gases that cause a greenhouse effect which warms up the earth surface as a consequence of a different heat flow between the earth and the atmosphere. Our laboratory measurements determined the porosity, permeability and grain density for clastic sedimentary rock samples which were drilled from an underground gas storage facility. Additionally, our results showed a reduction in porosity and permeability after a confining pressure was applied. We assume that this effect is caused by internal structure changes due to the repeatedly increased and decreased net pressure applied to the samples.

Abstrakt

Emise CO_2 vznikající lidskou činností – tzv. antropogenní emise CO_2 a jejich vzestupný trend, mohou být některými odborníky považovány za vážné nebezpečí pro udržitelný vývoj lidstva a jejich omezování za nezbytnou podmínku ochrany životního prostředí. Oxid uhličitý je významný z plynů způsobujících skleníkový efekt, který se projevuje oteplováním zemského povrchu v důsledku změn toků tepelného záření mezi zemí a atmosférou. Laboratorní měření poskytla hodnoty porozity a koeficientu propustnosti horninových vzorků, které byly odvrtány z podzemního zásobníku plynu. Naše měření vykázalo snížení kolektorských parametrů horninových vzorků, které bylo způsobeno změnou vnitřní struktury horniny díky opakovanému zvýšení a snížení tlaku na rostlou část vzorku.

Key words: carbon capture and storage (CCS); enhanced oil recovery (EOR); porosity; permeability; laboratory experiment

1 INTRODUCTION

Several projects that deal with theoretical and pilot research on CO_2 storage in geological formations are currently underway. These projects are addressed by national programmes in USA, Canada, Australia and Japan. One of the projects under the supervision of the European Union was the RECOPOL project which evaluated the CO_2 storage in coal seams of the Lower-Silesian Basin. The main objective of these projects is to find out whether the CO_2 storage in geological formations is economically feasible and environmentally safe. In the Czech Republic, the most perspective formations for storing this gas are connected with oil and gas reservoirs.

Potential storage spaces are the depleted and actively produced oil- and gas fields, in which it is possible to enhance oil recovery by 10 to 15 % by using the CO_2 injection into the reservoir. Oil fields are a favourable variant, because before they were produced, the hydrocarbons were stored inside them during geological time, and similarly the carbon dioxide can be stored there now. Another advantage they have is a well explored geological environment, and, therefore, an abundance of information on the selection of suitable storage locality, its utilization and long-term monitoring. The capacity of the CO_2 storage space in an oilfield depends on the pore space freed after oil production and the pore space that is filled with water under the oil bearing horizon. Depleted oil- and gas- fields represent suitable porous rock structures either for CO_2 sequestration or for underground storage of imported natural gas.

2 THEORETICAL ASPECTS OF CO_2 STORAGE

It is assumed that horizons used for the carbon dioxide sequestration will lie in depths below 800 m. At temperatures and pressures corresponding to the depths, lower than the above mentioned, carbon dioxide changes its phase behaviour, its density resembles liquids and its state is called a supercritical state. This transition into the supercritical state takes place under p,T conditions of 7.38 MPa and 31.1°C, respectively.

Carbon dioxide injected in a supercritical state occupies much less space than in a gaseous state. In the depth interval of 600-800 m, the CO_2 density increases with depth. From the depth of 1000 m, it reaches its maximum value and it doesn't change with depth any more. Under standard conditions (temperature of 25°C and pressure of 0.1 MPa), the density of CO_2 is 1.977 kg/m^3. That means that 1 tonne of CO_2 occupies a volume of 526 m^3. On the temperature and pressure conditions in a depth of 1000 m (35°C, 10 MPa), one tonne of CO_2 occupies a space of 1.5 m^3 (CO_2 density is 650 kg/m^3) [4].

In order to inject CO_2 effectively, its density should be in an interval of 600 to 800 kg/m^3 (on p,T conditions of 30°C and 8 MPa, resp.).

Mathematical modelling of a geochemical sequestration process is needed for developing a notion of how the injected CO_2 will behave in a reservoir. One of such models based on our laboratory experiments was created for a saline aquifer of the Upper Silesian Coal Basin conditions [2]. The Geochemist's Workbench 7 (GWB) simulator was used for modelling. The modelling process had two stages. The first stage aimed at observing the changes in the rock environment at the beginning of CO_2 injection. The second stage evaluated the changes caused by the CO_2 influence on permeable rocks after the injection.

A timespan of 20 thousand years was analysed in the model. During the first three years after the injection ended, a continuous increase in porosity takes place in the rock environment. Afterwards, this value stabilizes at a maximum level without further changes [2].

3 SIMULATION OF RESERVOIR ROCK CAPACITY ABILITY

For ascertaining the CO_2 storage capability, the knowledge of ***porosity*** and ***permeability*** values of a natural reservoir is essential. These parameters were measured by means of the apparatuses COREVAL 700 and Benchtop Relative Permeameter 350 (BRP 350) in the Laboratory of Wells and Hydrocarbon Deposits Stimulation at the Institute of Clean Technologies for Extraction and Utilization of Energy Resources, under the Faculty of Mining and Geology at the VŠB – Technical University of Ostrava.

3.1 Porosity and permeability of natural reservoirs

Pores can be defined as spaces of different shapes, size and origin in soil or between rock grains that are not filled with solid phase. We differentiate these porosities:

- absolute porosity;
- open porosity;
- effective porosity.

The porosity as well as the permeability is evaluated at our facilities by means of the automatic porosimeter and permeameter COREVAL 700. The device's method of work is based on API recommendation [5] which uses the *Boyle´s Law Single Cell Method* for measurements of a free space. The method uses a reference cell filled with gas of a reference volume and pressure which is afterwards released into the pore volume of a given sample. The sample is placed into a core-holder and is fastened in an elastic sleeve which induces a confining (lithostatic) pressure. The whole experiment is isothermal. The determined value is the open porosity which is the ratio of the bulk sample volume to the volume of interconnected pores including dead-end pores.

The core samples porosity measurements show a variance of 0.1 cm^3 for a 50 cm^3 volume sample, the porosity margin is ± 0,2 % of the real value.

The measured parameters are:

- pore volume V_p (cm^3),
- sample porosity φ (%),
- bulk volume V_b (cm^3),
- grain volume V_g (cm^3),
- grain density Gd (g/cm^3),
- gas permeability K_g (mD),
- slip factor b (psi),
- initial resistance β (ft^{-1}),
- turbulence factor α (μm).

It is convenient to determine the above mentioned parameters at expected reservoir pressures. Next, it is necessary to determine a hysteresis (with an appropriate step – at minimum 6 values) up to the pressure that is 15 % higher than the expected reservoir pressure. Last but not least, it is desirable to determine an extreme hysteresis (at an appropriate step – at minimum 6 values) for approximately three times the value of the expected reservoir pressure.

Permeability is a property of a porous medium and is a measure of its ability to transmit fluids. The reciprocal of permeability represents the viscous resistivity that the porous medium offers to fluid flow when low flow rates prevail.

A transient pressure technique for gases: Pressure-Falloff, Axial Gas Flow measurements [5]. Transient measurements employ fixed-volume reservoirs for gas. These may be located upstream of the sample, from which the gas flows into the sample being measured. The pressure falloff apparatus (Fig.1) employs an upstream gas manifold that is attached to a sample holder capable of applying hydrostatic stresses to a cylindrical plug of diameter D and length L. An upstream gas reservoir of calibrated volume can be connected to the calibrated manifold volume by means of a valve. Multiple reservoir volumes are used to accommodate a wide range of permeability values. The downstream end of the sample is vented to the atmospheric pressure. An accurate pressure transducer is connected to the manifold immediately upstream of the sample holder. The reservoir, manifold, and the sample are filled with gas. After a few seconds for thermal equilibrium, the outlet valve opens to initiate the pressure transient. The pressures and times are recorded. This technique has a useful permeability range of 0.1 to 5000 milliDarcys (mD).

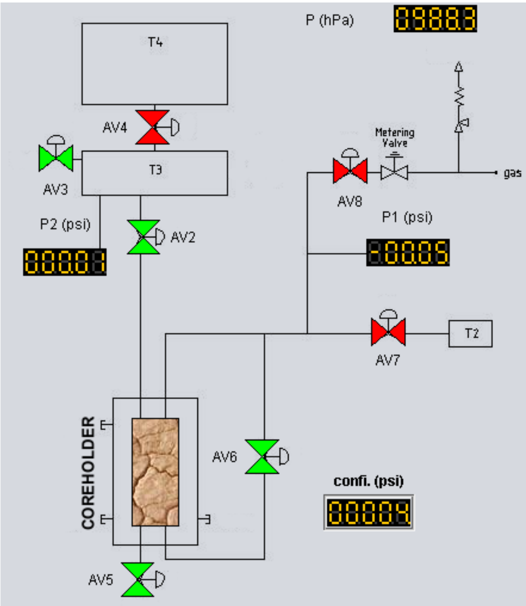

Fig. 1 The scheme of COREVAL 700 apparatus
(T2-4: Reference chambers; P,P1,P2,confi.: manometers;
AV1-8: valves)

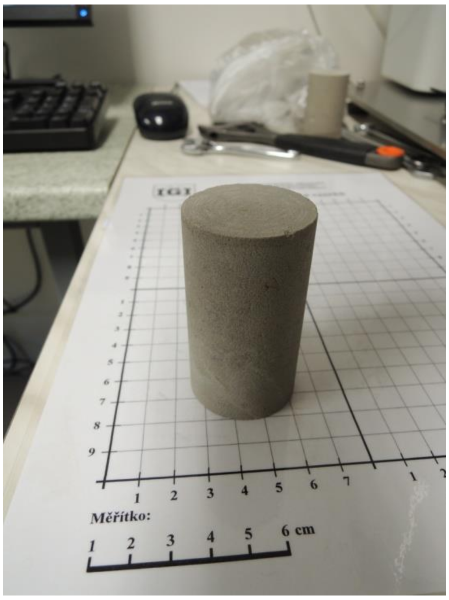

Fig. 2 Tested core

3.2 Laboratory measurements of chosen parameters of core samples on Automatic porosimeter-permeameter

To understand the behaviour of rock massif considered for the CO_2 storage, real core samples were selected. Their petrographic composition corresponds to the potential storage formation. They are fine-grained sandstones with addition of clay. They were drilled out from one larger core perpendicularly to its axis. The parameters of the samples were (fig. 2):

- diameter – 38,4 mm,
- length – 68,2 mm,
- initial weight – 156,56 g.

The confining pressure of the first reference measurement was set to 1000 psi (6.895 MPa). The measurement outcomes are presented in Tab. 1. The core sample before inserted into the Automatic porosimeter-permeameter is shown in Fig. 2. The objective of a series of subsequent measurements was to determine hysteresis curves for different confining pressures. The hysteresis curves tell us how differently the measured parameters change when the confining pressure increases and subsequently decreases. Another objective was to ascertain how the internal structure of a tested core will look like after repeated rises and drops of the confining pressure. To portray the hysteresis curves, the following pressure steps were chosen: 1000 psi, 1200 psi, 1400 psi, 1600 psi, 1800 psi, 2000 psi, 2100 psi, 2200 psi, 2100 psi, 2000 psi, 1800 psi, 1600 psi, 1400 psi, 1200 psi, and 1000 psi. It is a pressure range from 6,895 MPa to 15,168 MPa. These are pressures that can be expected to occur in the formation suitable for the CO_2 storage. Because of assumed shape memory of a measured sample, the time span between different measurements was at least 24 hours.

Tab. 1 Measurement outcomes at the confining pressure of 1000 psi (6,895 MPa)

confining pressure	**pore volume**	**porosity**	**bulk volume**	**grain volume**	**bulk density**	**grain density**	**confining pressure**	**air (N_2) permeability**
Pc (psi)	**Vp (cm^3)**	**φ (%)**	**Vb (cm^3)**	**Vg (cm^3)**	**Bd (g/cm^3)**	**Gd (g/cm^3)**	**Pc (psi)**	**K [air] (mD)**
1000	19.9167	25.2958	78.7226	58.8445	1.9789	2.6620	1000	41.1791

Hysteresis curves determination for chosen measurement parameters

The hysteresis curves were determined based on the measurement of chosen parameters in a pressure range from 6.895 MPa to 15.168 MPa (between 1000 psi and 2200 psi; **1 MPa = 145.0377 psi**) for the above mentioned pressure steps. After the first measurement, the weight of the core sample was 156.45 g. The weight loss after the first set of measurements was 0.11 g. The measurement outcomes are documented in the following charts for different measured parameters. During the measurement, the porosity and permeability of samples generally decrease up to the point of the highest used confining pressure and subsequently increase in general during the release of the confining pressure. In each case, the values of porosity and permeability are higher when determined at the starting pressure (1000 psi) than the values determined at the same pressure after the confining pressure was adjusted to the maximum value (2200 psi) and released to the value of 1000 psi.

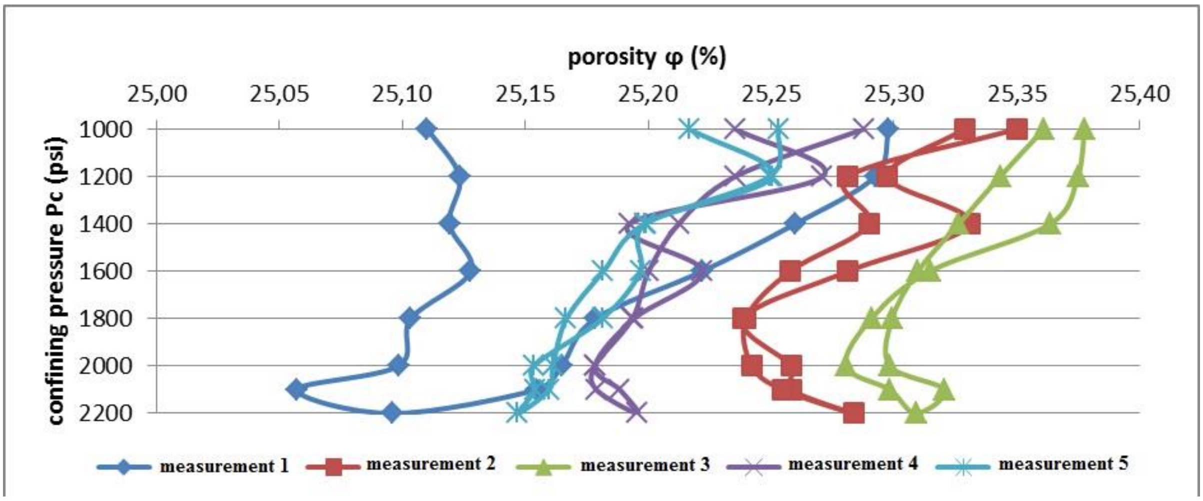

Fig. 3 Hysteresis curves of porosity for different pressure steps

Ad Fig. 3:

The comparison of the first measurement with other four measurements tells us that the internal structure of the sample had undergone significant changes. In absolute numbers, the changes are negligible in the order of one hundredth of a percent (the average value of porosity at 1000 psi is 25.2880 % before the pressure started to rise and 25.2756 % after it dropped; at a maximum pressure of 2200 psi, the porosity was 25.2062 %). Nevertheless, the shape of the curves confirms that after several measurements, changes in the internal structure took place, which indicates at least some damage of the internal structure. It can be assumed that during the last two measurements, a partial internal sample consolidation took place - proportionally between effective and closed porosity.

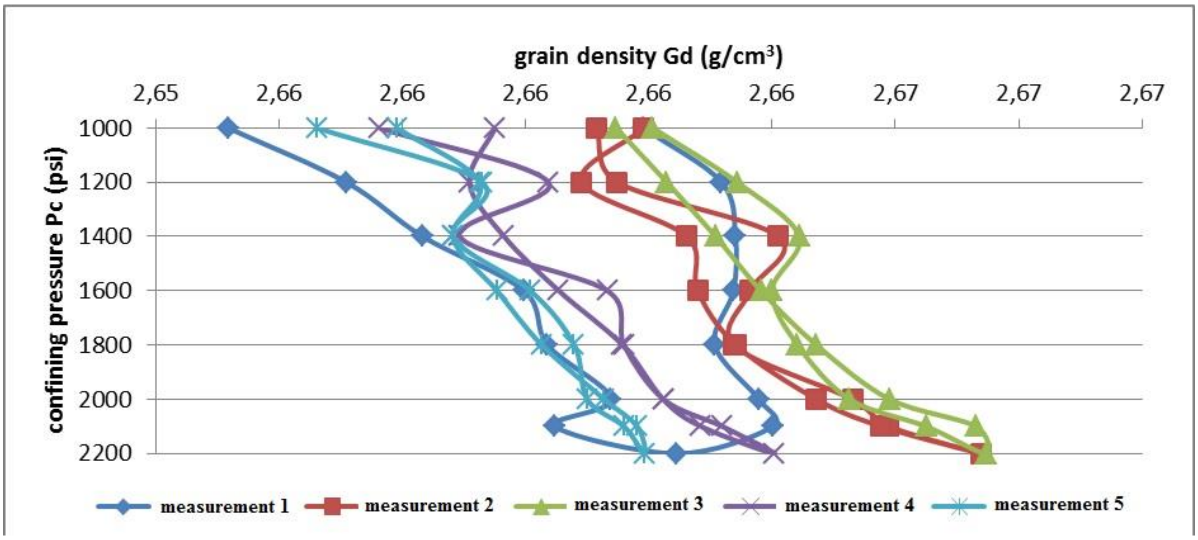

Fig. 4 Hysteresis curves of grain density for different pressure steps

Ad Fig. 4:

Logically, the grain density Gd is higher than the bulk density Bd, due to the low density of fluids filling the pores. Within the whole series of measurements (from 1 to 5), the grain density was changing on a second or third decimal position, therefore the change is negligible. The average grain density of the core sample was found to be 2.6620 g/cm^3. An interesting fact is that the grain density Gd curves shape corresponds with the shape of the grain volume Vg curve and partially with the porosity φ curve.

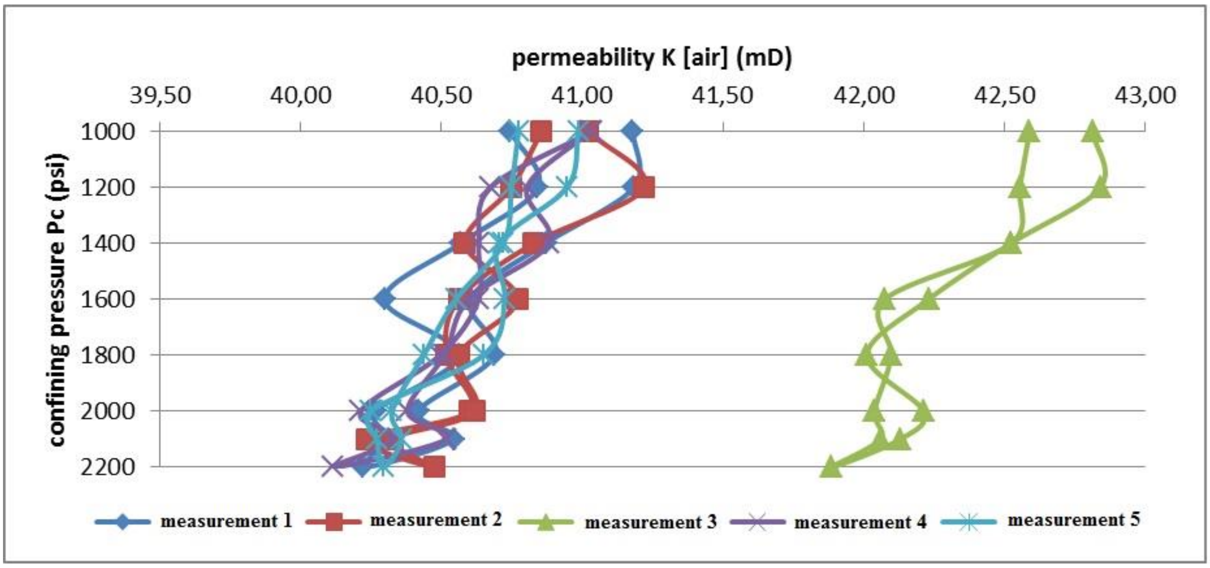

Fig. 5 Hysteresis curves of air (N_2) permeability for different pressure steps

Ad Fig. 5:

The air permeability (medium – N_2) showed itself to be consolidated and, if the measurement no. 3 is not taken into account, it was found to be 40.6139 mD. The measurement no. 3 shows permeability values at least 1 mD higher than the rest of the measurements. It is probably due to the fact that during this measurement, the greatest internal structure changes, which caused the higher permeability, took place. After a partial consolidation the permeability has settled at primeval values.

4 CONCLUSIONS

During the laboratory experiments, a set of cores from a single borehole was tested. Charts 1 and 3 show the most interesting results. And even though, at first glance, these samples from a similar depth look the same, they shown not only different petrophysical results, but also implied possible complications in the internal structure of reservoir rocks considered for the CO_2 storage. For the ***porosity***, the difference between the examined cores was ***as much as* 3 %**, while the difference of the ***permeability*** was more than ***40 mD***. At the same time, the ***grain density*** was the same for all the cores (***2,64 – 2,66 g/cm³***), it varies on the second or third decimal position. The measurements on these cores indirectly indicated that even though the confining pressure was relatively low (6.895 MPa to 15.168 MPa), the internal structure suffered some damage. It is reflected in hysteresis curves (charts 1 to 3). A “bent V” shape of the curves, with a shape memory, was expected. Only the first measurement approximately resembles this shape, four others are much more chaotic. It is likely that after every pressure step, the deformation of porous space took place, in a way that the effective pore space was squeezed so much that it prevented the flow of measuring medium (N_2). The assumption of internal structure deformation is backed by an evidence of visual reconnaissance of the core surface. The cracks, some more than 0.5 mm deep, appeared on the core’s surface. This phenomenon is known from cyclical operations of underground gas storage sites, where due to the thermal and pressure changes, micro-particles of the rock matrix are crumbled away and form a so called “silt cloud”. The outcome of the measurement confirms the non-homogeneity of the geological environment. Even though, the samples are from a single borehole from approximately the same depth, the results vary substantially.

The following research works will focus on ascertaining the CO_2 phase permeability in a supercritical state (7.38 MPa and 31.1°C). For this task, another laboratory device of the Laboratory of Wells and Hydrocarbon Deposits Stimulation will be used. It is the BRP 350 multiphasic permeameter made by Vinci Technologies (France).

REFERENCES

[1] Bethke C.M., 2008, Geochemical and biogeochemical reaction modelling. Cambridge Univ. Press, Cambridge: 1-543.

[2] Labus, K., Bujok, P.. CO_2 mineral sequestration mechanisms and capacity of saline aquifers of the Upper Silesian Coal Basin (Central Europe) - Modeling and experimental verification. Energy. 2011, vol. 36, issue 8, s. 4974-4982. DOI: 10.1016/j.energy.2011.05.042.

[3] Palndri J.L., Kharaka Y.K., 2004, A compilation of rate parameters of water-mineral interaction kinetics for application to geochemical modeling. US Geological Survey. Open File Report 2004-1068: 1-64.

[4] Xu T, Apps JA, Pruess K (2003) Reactive geochemical transport simulation to study mineral trapping for CO_2 disposal in deep Arenaceous Formations. J. Geophys. Res., 108: B2.

[5] AMERICAN PETROLEUM INSTITUTE. Recommended Practices for Core Analysis [online]. USA: API Publishing Services, 1998 [cit. 2013-05-13]. Recommended practice: 40, 2nd ed. Dostupné z: http://w3.energistics.org/RP40/rp40.pdf

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RESUMÉ

V průběhu laboratorního výzkumu byla otestována řada vzorků vrtných jadérek z jednoho návrtu. Grafy č. 1 až č. 3 ukazují nejzajímavější výsledky. A i když se jednalo o na první pohled stejné vzorky z přibližně stejné hloubky, vykázala testovaná jádra nejenom odlišné fyzikálně petrografické výsledky, ale naznačila i možné komplikace v oblasti vnitřní struktury nádržních hornin pro uskladňování CO_2. U ***pórovitosti*** je sice rozdíl mezi oběma testovanými jádry ***až 3%***, ale při srovnání s **permeabilitou** je rozdíl mezi oběma jádry i více než ***40 mD***. Přitom ***hustota rostlé části*** je pro všechna testovaná jádra takřka stejná (***2,64 – 2,66 g/cm^3***), liší se maximálně na druhém až třetím desetinném místě. Měření u tohoto konkrétního jadérka nepřímo naznačila, že i když se jednalo o relativně nízké tlakové hodnoty (6,895 MPa až 15,168 MPa), došlo k poškození vnitřní struktury. Svědčí o tom průběh hysterezních křivek (grafy č. 1 – č. 3). Byl očekáván hladký průběh ve tvaru „vyhnutého V“ s předpokládanou tvarovou pamětí. Tohoto stavu pouze přibližně dosáhlo první měření, další čtyři měření již vykázala na první pohled chaotický průběh. Nejspíše při každém tlakovém projevu (Pc) došlo k deformaci vnitřního pórového prostředí, kdy se v určitou chvíli měnilo efektivní pórové prostředí na uzavřené nebo polouzavřené pórové prostředí a tím znemožňovalo nebo výrazně ovlivňovalo průběh prostupu měřícího média (N_2). Předpoklad o deformaci vnitřní struktury potvrzuje i vizuální ohledání vnějších stran jadérka. Je patrné poškození vnější strany jádra, kdy některé trhliny vykazují hloubku i více než 0,5 mm. Tento jev je znám z cyklického provozu PZP, kdy vlivem tlakových a teplotních změn dochází k „vydrolování“ mikročástic z matrixu horniny a vzniku tzv. siltového mraku. Výsledek měření tak potvrzuje fakt nehomogenity geologického prostředí. I když se jedná o jeden návrt, tedy přibližně stejnou hloubku, jsou výsledky často odlišné a proměnlivé.

Další část výzkumu bude zaměřena na stanovení fázových propustností CO_2 za superkritického stavu (tedy tlaku 7,38 MPa a teplotě 31,1°C). K tomu bude využito dalšího laboratorního přístroje Laboratoře stimulace vrtů a ložisek uhlovodíků. Jedná se o fázový permeametr BRP 350 firmy VINCI Technologies (Francie).