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Methodological aspects of porosity and pore space measurements in shale rocks

Introduction

Porosity and pore space investigations are not commonplace in shale rocks. Repeating of conventional analyses could produce a great mistakes in estimation of reservoir parameters in such types of reservoirs. Methodical investigations were performed to establish reliable parameters of porosity and pore space analyses. Sample collection was prepared from the old boreholes. Paper prepared in the frame of Polish-American Scientific Program Nr 282/N-USA/2008/O (realized by Oil and Gas Institute and Oklahoma University).

Plan of investigations

Generally correctness of porosity and pore space investigations were examined. The base trouble in shale rocks investigations is necessity of complete extraction and drying of samples before analysis. It is difficult because of nanostructures of pore space. The only solution is crumbled of samples, but granulation must satisfy two conditions: give reliable results and secure 100% extraction and drying of samples. Mercury and helium pycnometry as well as mercury porosimetry were applied in investigations [2]. Accuracy and reliability of performed analyses were verified. Only degree of granulation was changed. The other parameters of analyses were conserved.

A frame and correctness of pore space investigations are created by two analyses:

- bulk density measurement with the use of mercury pycnometry and performed on non crumbled sample (it is the lowest value of density),
- grain density measurement with the helium pycnometry and performed on dry, powdered sample contain residual organic matter (it is the greatest value of density).

The others densities, measured during porosimetry investigations will cover the range between these two values. In this type of investigations residual organic matter is treated as a part of reservoir rock. Methodical researches were devoted to find the best granulation of investigated rocks. The highest pressure applied during porosimetry analyses corresponds with pore diameter equal to 40 nanometers.

Porosity investigations

In our methodology of shale rocks porosity measurements all kinds of porosity are intergranular, intragranular and pores in residual organic matter are treated as the same parts of total porosity. As a set of necessary parameters: grain density, bulk density are obtain using mercury pycnometry for non crumbled samples, helium pycnometry for dry, powdered samples.

The third necessary parameter is mass of the sample. So:

m – mass of prepared, dry sample [g],
$\gamma(o)$ – bulk density [g/cm^3],
$\gamma(z)$ – grain density[g/cm^3].
And we obtain

$$POR = [(m/\gamma(o) - m/\gamma(z)]/[m/\gamma(o)] \times 100\ [\%] \quad (1)$$

A formula (1) is obvious under condition of proper determination of m (it means that sample is really dry – all reservoir fluids were removed from it). For shale rocks 100 percent efficiency of extraction is obtain for crumbled sample.

Correctness and reliability of measurements can be verified with the use of another set of analyses containing quantitative mineralogical investigations and Rock Eval analyses. These measurements give the base for grain density calculations. Sketch of analytical sets of measurement is presented in Fig. 1.

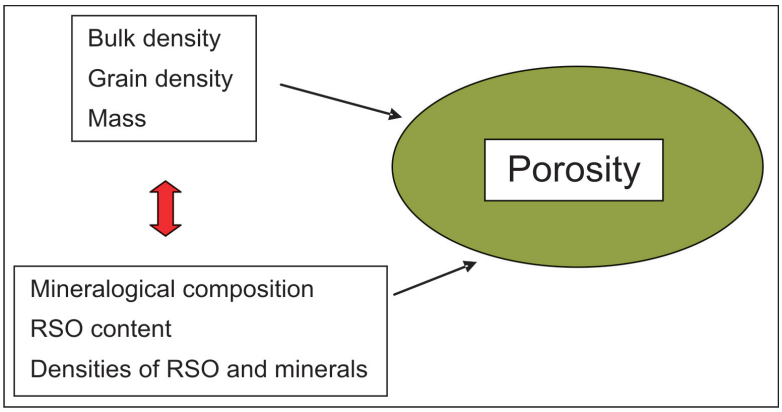


Fig. 1. Sketch of measuring and verifying of porosity

Pore space investigations

Mercury porosimetry measurement depend on pressing of mercury into a pore space under pressure [3]. At last dependence of mercury volume in pore space in the function of pressures is obtained. Applying capillary tube model of pore space and Washbourne'a formula [4] it is possible to correlate volume of mercury in pore space with pore diameters. Measurement is reliable when mercury can penetrate all open pores in investigated pore space.

A series of investigations was performed using standard procedure of preparation as well as standard analysis procedure for shale rocks. Efficiency and reliability of these measurements were estimated basing on the base of percent of pore space occupied by mercury (measured as porosimetry porosity). At first total, open porosities of these samples were measured using shale rocks procedures. The results are presented in Tab. 1.

Table 1. Efficiency of conventional procedure

Sample number	Bulk density [g/cm^3]	Grain density [g/cm^3]	Total, open porosity [%]	Porosimetry porosity [%]	% of pore space occupied by mercury
1	2.60	2.63	1.10	0.43	39
2	2.61	2.65	1.88	0.89	47
3	2.40	2.65	9.43	4.44	47
4	2.49	2.65	6.00	2.15	36
5	2.52	2.65	4.90	1.81	36

It was shown that:

- percent of pore space occupied by mercury is low (36÷47%),
- final mercury saturation does not depend on porosity.

Measurements were performed up to pressure according 40 nm in Washbourne formula.

Generally low degree of pore space saturation can be caused by three factors: extraction and drying were not efficient, standard procedure parameters are not suitable for such kind of pore space (too short time for mercury penetration) and due to great percent of nanopores (diameters less than 40 µm).

Application of longer time of mercury penetration during analyses is impossible because of their long time. Nanopores of diameters less than 40 nm will be taken into account by applying higher capillary pressures (up to 60 000 psi – corresponding diameter is equal to 5 nm).

So, the only solution for improfement analyses the application apply crumbled samples. The degree of crumbling must be established on level appropriate to 100% efficiency of extracting and drying but allows us to investigate real parameters of pore space, not intergranular spaces.

Series of 5 samples were investigated for the various diameters of prepared grains. Two analyses were performed for all granulations and repeatability of measurements was investigated.

No crumbled samples, grains greater than 1 µm, grains 0.5÷1 µm and powdered samples were investigated. In all measurements standard preparations of cores as well as standard parameters of mercury porosimetry measurements were applied [2].

For all cumulative curves correction for broad effect were done (pores greater than 100 µm). In such conditions all curves started from the level of mercury saturation equal to 0%. It let as to compare their shapes in interesting ranges of diameter. After performing the first series of analyses the second one was made to verify obtained results.

The results

For grains 1÷3 μm improved quolity of measurements was not observed. Calculated porsimetry porosities are practically the same as for no crumbled samples. Cumulative curves for these samples have similar shapes, they cross or lies near by one another. These differences can be explained by heterogeneity of rock, efficiency of preparation of sample as well as mercury penetration are similar. Also threshold diameters are the same for both curves. The results do not depend on total porosity. Typical curves were presented in Fig. 2 and 3.

In Fig. 2 cumulative curve for crumbled rock lies in great part above cumulative curve for no crumbled sample. In Fig. 3 is another situation can be observed. But in both cases differences are rather cosmetics and the only conclusion is that it is not a good granulation for shale rocks.

In the case of granulation 0.5÷1 μm results change. In Fig. 3 two cumulative curves for granulation 0.5÷1 μm were presented (curves C and D). These curves conserve value of threshold diameter. Efficiency of mercury penetration of pores space is good. It is the result of better extraction and drying for such type of sample as well as time of mercury penetration into pores. Both curves show good repeatability.

These improvements were observed for all investigated samples (grain diameters 0.5÷1 μm).

Tab. 2 shows values of calculated porosity for them. Calculated porosities covered the range from 65 to 81% of total open porosity. It is good efficiency taking into account that nanopores were not measured during analyses.

To investigate all possibilities also powdered samples were performed (the sample applied in grain density measurements) but result was nonsensical. Obtained cumulative curve described not pore space but intergranular spaces so its parameters depend on diameters of grain. In such conditions calculated porosity was several times greater than total porosity. This phenomena is illustrated in Fig. 4, in which

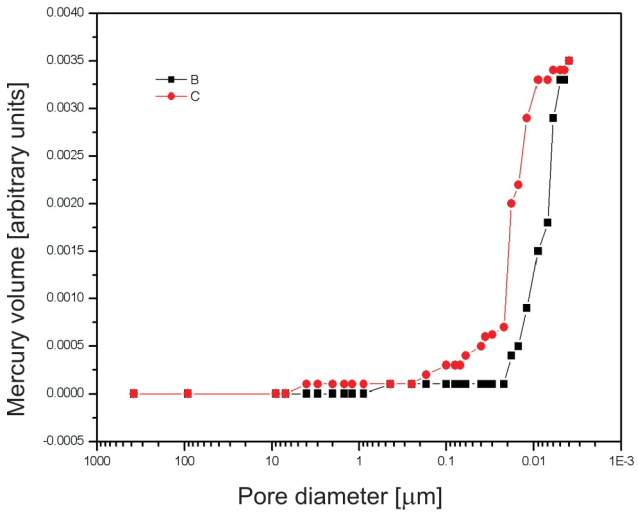


Fig. 2. Pore diameter cumulative curves (sample "C"): B – non crumbled sample, C – crumbled sample (granulation 1÷3 μm)

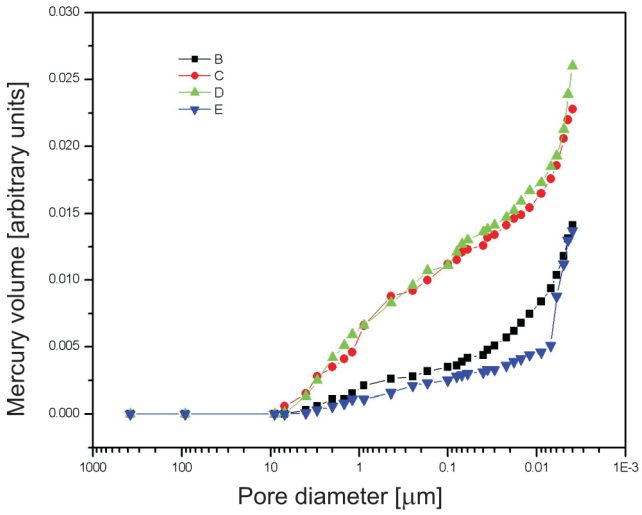


Fig. 3. Pore diameter cumulative curves (sample "A") for: B – non crumbled sample, C&D – crumbled samples (granulation 0.5÷1 μm), E – crumbled sample (granulation (1÷3 μm)

Table 2. The results of porosities calculated on the base of mercury porosimetry for no crumbled and for grains 0,5÷1 μm (parameters of analysis were the same for all analyses)

Sample number	No crumbled			Grains 0.5÷1 mm	
	Total open porosity [%]	Porosimetry porosity [%]	% of mercury saturation	Porosimetry porosity [%]	% of Merkury saturation
1	1.10	0.43	39	0.80	72
2	1.88	0.89	47	1.48	78
3	9.43	4.44	47	7.70	81
4	6.00	2.15	36	3.90	65
5	4.90	1.81	36	3.60	73

curves for no crumbled sample, for 0.5÷1 μm granulation and for powdered sample are presented for the same rocks.

Typical cumulative curves for low porosity shale rocks are presented in Fig. 4. In Fig. 5 and in Fig. 6 are respectively presented cumulative curves for high and medium porosity.

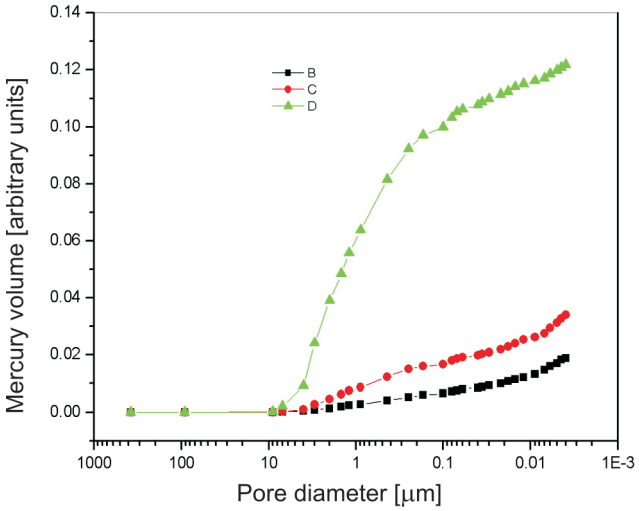


Fig. 4. Pore diameter cumulative curves (sample #2) for: B – non crumbled sample, C – crumbled sample (0.5÷1 µm), D – powdered sample

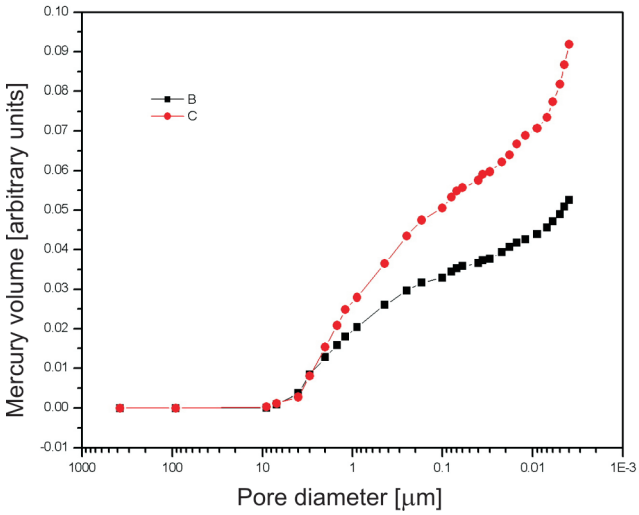


Fig. 5. Pore diameter cumulative curves (sample #3): B – non crumbled sample, C – crumbled sample (0.5÷1 µm)

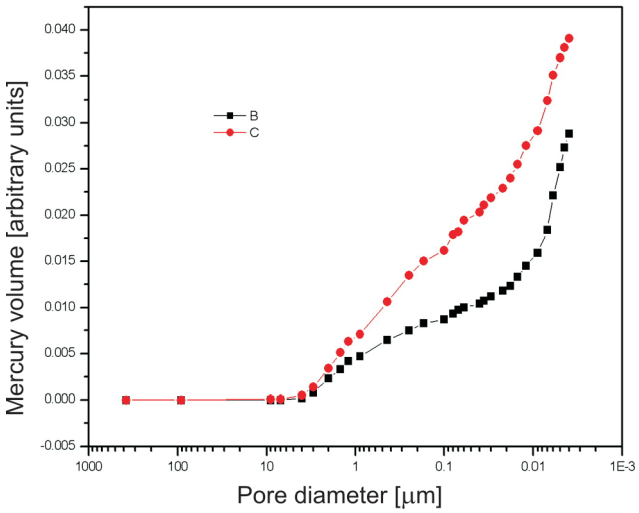


Fig. 6. Pore diameter cumulative curve (sample #4): B – non crumbled sample, C – crumbled sample (0.5÷1 µm)

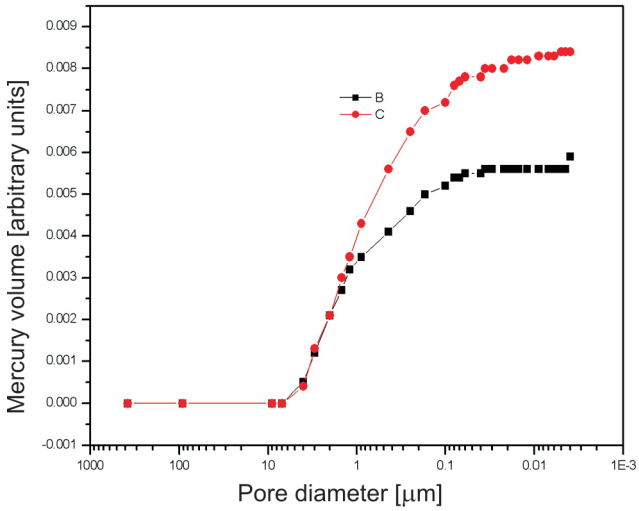


Fig. 7. Cumulative curves for sample "B": B – no crumbled, C – grains 0,5÷1 µm

Control investigations with the use of the second set of samples (signed as A, B, C, D) confirmed correctness of applied granulation. In Fig. 7 are presented cumulative curves for sample of porosity equal to 9% and threshold diameter equal to 5 µm. For pore diameters greater than 1 µm both curves are similar. Differences occurs for mikro and nanopores (as for other shale rock samples). Fractal dimensions for these range of pores from log (mercury volume) – log (pore diameter) plot were calculated [1]. The results are presented in Fig. 8. For crumbled sample fractal dimension for nanopores was equal to 2.27 for no crumbled was equal to 2.52.

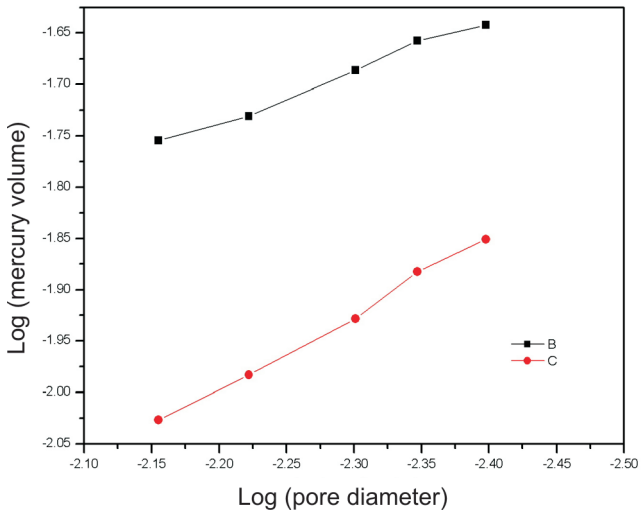
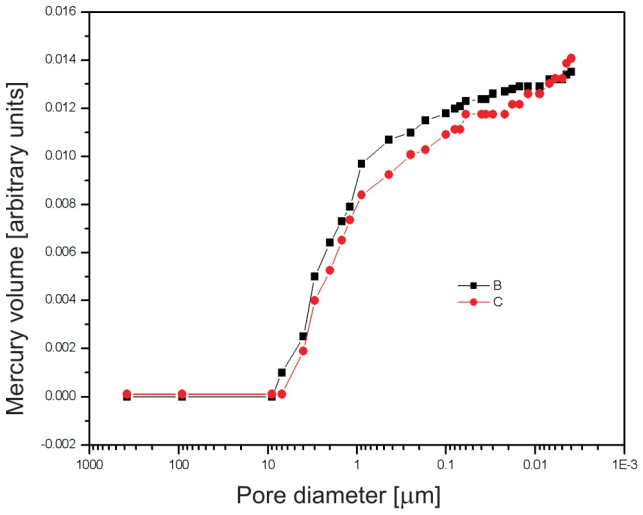


Fig. 8. Log – log curves for sample "B"

At last cumulative curves for crumbled and no crumbled sample are presented in Fig. 9. Porosity of this sample is greater than 12%. Its threshold diameter is equal to 10 µm



and in this situation more than 50% of pore space is built with pores of diameters greater than 1 μm. It is geologically shale rocks but its petrophysical parameters allow us to describe it as permeable reservoir rocks in conventional meaning. And for this sample both curves are very similar. It is the best confirmation of used methodology.

Fig. 9. Cumulative curves for porous and permeable sample (sample "D"): B – no crumbled, C – 0.5÷1 μm grains

Conclusions

1. Porosity could be measured with the use of mercury pycnometry (non crumbled sample) and helium pycnometry (powdered sample). The results can be verified using quantitative mineralogy and residual organic matter investigations.
2. Extraction and drying are critical points in pore space investigations. For shale rocks the only method of efficient extracting and drying is rock granulation.
3. It was found that grain diameters must covered the range from 0.5 to 1 mm. These granulation gives reliable and repeatable results of pore space investigations with the use of standard parameter of analyses.

Paper prepared in the frame of Polish-American Scientific Program Nr 282/N-USA/2008/O ((realized by Oil and Gas Institute and Oklahoma University).

Artykuł nadesłano do Redakcji 12.04.2011 r. Przyjęto do druku 13.04.2011 r.

Recenzent: prof. zw. dr hab. inż. Józef Raczkowski

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