

INFLUENCE OF THE MINERAL COMPOSITION OF SHALES ON THE RESULTS OF TRIAXIAL COMPRESSION TESTS – A CASE STUDY FROM THE ORDOVICIAN AND SILURIAN ROCKS OF POLAND

Marek Dohnalik^a, Sylwia Kowalska^a, Zbigniew Mikołajewski^b, Andrzej Domonik^c,
Zbislaw Tabor^d

^aOil and Gas Institute National Research Institute, Poland,

^bPGNiG SA, Geology and Hydrocarbon Production Branch, Poland

^cUniversity of Warsaw, Institute of Hydrogeology and Engineering Geology

^dCracow University of Technology, Faculty of Physics, Mathematics and Computer Science

This paper has been prepared for presentation at the International Symposium of the Society of Core Analysts held in Avignon, France, 8-11 September, 2014

ABSTRACT

Hydraulic fracturing is needed to extract hydrocarbons from shale rock formations. The efficiency of fracturing depends on many factors, i.e. for example the mechanical properties of rocks. This paper shows the results of studies on the influence of the mineral composition of rocks on their mechanical properties. The investigated cores represent different formations from the Silurian-Ordovician Basin from Poland. X-ray diffractometry (XRD) and X-ray fluorescence (XRF) methods were used to establish the composition of the studied rocks, whereas the mechanical parameters were derived from triaxial compression tests. Correlation was conducted between the mineral composition and the mechanical parameters. The X-ray microtomography (micro-CT) method was used in order to visualize the structural changes in the rocks caused by triaxial compression. As a result of the research, a discussion on the influence of the rock matrix composition on its mechanical properties is presented.

INTRODUCTION

Recently in Poland there is high interest in the research on shale formations. The hydraulic fracturing process is needed to extract hydrocarbons trapped in such rocks. The efficiency of fracturing depends on many factors, including i.e. the mechanical properties of rocks. The rock mechanical properties derive from the mineralogical composition, degree of compaction and the internal structure in macro- and micro- scale and they can be measured directly. Rock susceptibility to hydraulic fracturing can also be estimated based on the analysis of rock chemical or mineralogical composition, which are indicators of the rock brittleness. This paper describes a complex study of the rock mechanical properties performed on cores representing shales of Ordovician and Silurian age from various lithostratigraphic units from Poland; for comparison, samples from

intercalations occurring within the shale complex (marls, limestones) have also been tested.

The aim of this research was to compare results of standard tests of the mechanical properties in rock cores with the mineralogical and chemical indicators of rock brittleness achieved by the XRD and XRF methods. As a complementary method, a micro-CT was applied in order to visualize the internal core structure before and after the triaxial compression tests. The observed changes were correlated with the lithological properties of the studied rocks.

METHODOLOGY

The investigated samples were cored from original rock cores. The samples were cylindrical in shape with an approx. 8 cm height and 5 cm in diameter. The samples were subjected to different research methods in the following order:

- Micro-CT scanning;
- 3 axial compression test;
- Micro-CT scanning;
- X-ray fluorescence;
- X-ray diffraction.

Microtomography scanning Micro-CT is a non-destructive and non-invasive method which allows to visualize the internal structure of the investigated object. More information describing the foundations of this method can be found elsewhere [1]. Microtomography (micro-CT) tests were performed in order to visualize the core structure changes caused by triaxial compression tests. Each sample was scanned twice, before and after the triaxial compression tests. In order to detect cracks after compression tests, the author's algorithm for crack detection was applied.

Triaxial compression tests The aim of the conducted strength tests was first of all the determination of the elasticity parameters: Young's static elasticity modulus (E) and static Poisson's ratio (ν). The compressive strength tests in the triaxial state of stress were conducted using the single failure test procedure. They were carried out according to the recommendations of the International Society for Rock Mechanics (ISRM) [2]. The triaxial compression tests and interpretation of the results were performed using procedures of the MTS company, which were based on the ASTM 2664-95a [3] standard, as well as the recommendations of the ISRM's "Suggested Method for Determining the Strength of Rock Materials in Triaxial Compression".

In this paper the main focus was put on the values of the Young's static modulus (E) as well as Poisson's static ratio (ν). The Poisson's ratio (ν) was defined as the relation between the size of the axial (ϵ_z) and lateral (ϵ_{xy}) strains; for further analysis we selected Poisson's ratio for the full linearity of all the characteristics of deformation (ν_{avl}) corresponding to the ASTM and ISRM recommendations, however it was determined in

more detail, only to the limit of microdilancy (microcracking) established according to the phenomenological description by Hallbauer and others (1973) [4]. For Young's modulus we selected the average modulus (E_{av}), which is closely connected to the phenomenological conduct of rock sample deformation and appears to be the most representative for the measured rocks.

X-Ray Diffraction (XRD) Quantitative X-Ray Diffraction (XRD) analysis was conducted by the Rietveld method with the use of the SIROQUANT programme [5, 6], which is useful for analysing samples containing clay minerals. The quantitative measurements were carried out on Panalytical X'Pert Pro apparatus with modern ultra fast detector (real time ultra strip X'Celerator). Voltage 40 kV and current 40 mA were applied, samples were scanned from 5° to 65° 2θ . Preparations were made according to the method introduced by Środoń et al. [7]. Zincite (ZnO) was added to the samples as an internal standard. Samples were grinded for 5 minutes in the McCrone mikronizing mill in order to reduce the grain size to $<20\mu\text{m}$. Internal standard (ZnO) was added prior to grinding to obtain full homogenization of the samples. Measurements were conducted on random preparations made by side-loading.

X-Ray Fluorescence (XRF) X-Ray Fluorescence (XRF) chemical analysis was conducted with the use of the BUKER S1 TITAN, a fully field portable analyzer based on energy dispersive XRF (EDXRF) technology. The S1 TITAN uses an Rh target X-ray tube (max: 50kV, 100 μA , 4 W) and 10 mm^2 X-Flash®Silicon Drift Detector (SDD) (typical resolution of 147 eV at Mn K-alfa peak). Applied X-ray spectrometer works with Fundamental Parameters (FP) software to allow for elemental quantification of completely unknown samples without standards. The device enables elemental analysis for the range from magnesium (Mg) through uranium (U).

Rock brittleness, defined as the susceptibility of rocks to fracturing, is usually determined by its mineralogy as a quartz content [8] and by its chemical composition as a SiO_2 content [9]. In the case of the Silurian-Ordovician rocks from Poland the mineralogical indicator of brittleness was defined as a sum of quartz and feldspar.

RESULTS

Nine core samples representing different lithostratigraphic formations have been investigated. The samples for the tests were selected based on sedimentological analysis of cores and log analysis. The selected samples represent lithostratigraphic units of the Ordovician (Prabuty Marls and Claystones Formation, Sasino Claystones Formation, Kopalino Limestones Formation) and the lowermost Silurian (Pelplin Claystones Formation, Pasłek Claystones Formation, Jantar Bituminous Claystones Member). These units correspond to the stratigraphic interval between the Arenigian and the Wenlockian. Rocks from the clay formations are similar in terms of mineralogical composition, structure and texture. They differ in colour (light grey to black) and organic carbon content (TOC). Besides of shales the sample of marl and limestone were also included to the investigation.

The XRD confirmed the differences in the mineral composition of the studied rocks. Table 1 presents selected mineral composition results as well as Poisson's ratio and Young's modulus values.

Table 1. Results of mechanical and mineralogical measurements

Sample no	Brittleness as a SiO ₂ content (XRF)	Brittleness as a sum of quartz and feldspars content (XRD)	Sum of carbonates (XRD)	Sum of clays (XRD)	Poisson's static ratio (ν)	Young's static modulus (E _{av})
	[wt%]	[wt%]	[wt%]	[wt%]	[-]	[Gpa]
1	64.27	26.7	13.1	55.0	0.19	30.0
2	66.83	27.9	1.3	66.5	0.17	21.7
3	44.73	17.3	14.5	47.6	0.12	23.6
4	63.46	32.5	22.0	44.0	0.19	38.6
5	22.55	7.6	71.8	18.3	0.26	50.4
6	68.17	28.8	2.5	66.3	0.16	22.8
7	67.71	28.2	3.1	66.8	0.15	18.3
8	70.65	29.2	8.8	59.7	0.16	26.9
9	64.97	27.7	2.8	57.9	0.12	15.1

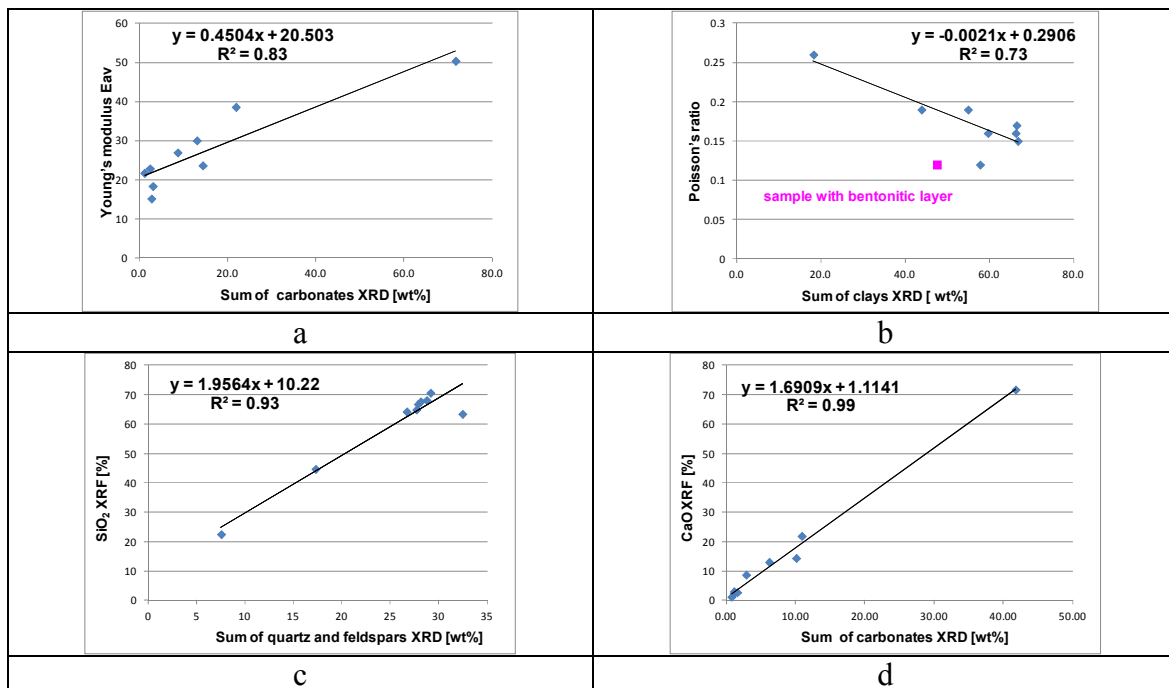


Fig. 1. Correlations of the obtained parameters.

A correlation analysis was performed for the parameters listed in Table 1. Example results are shown in Fig. 1. Young's modulus values and Poisson's ratio are highly dependent on the rock mineralogical composition. Young's modulus values are higher for higher carbonate concentrations (Fig. 1.a). For Poisson's ratio a good correlation was observed with the clay minerals content. Poisson's ratio values are lower for the higher

clay minerals content (Fig. 1.b). The correctness of the mineralogical analysis was proved by the XRF analysis (Fig. 1.c and 1.d).

Micro-CT results

According to the micro-CT results, the investigated cores may be divided into two groups. The first group includes samples with minor structure changes and the second group represents samples containing big cracks extending throughout the whole core structure. To the first group belong two samples with the highest concentration of carbonates (samples 4 and 5).

The second group samples show some tectonics cracks in their structure. These tectonics cracks could be the main reason why the visualization of the second group samples showed the most expanded crack after the triaxial compression test. Figure 2 shows examples of the first and second group samples. The top example shows group one sample and the bottom example refers to samples representing group number two.

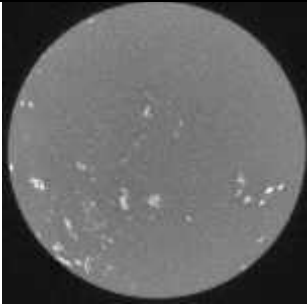
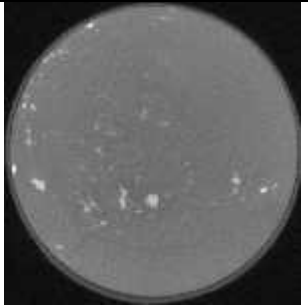
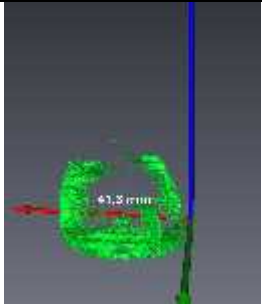
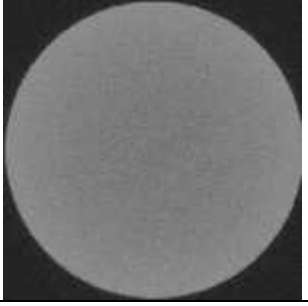

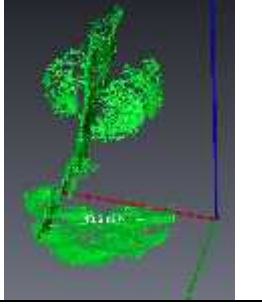
Sample group no.	Cross-section before the triaxial compression test	Cross-section after the triaxial compression test	3d visualization of core structure changes
1			
2			

Fig. 2. Visualization of the core structure changes after the triaxial compression test. Green colour on the right image shows segmented cracks.

CONCLUSIONS

The correlation of triaxial compression and mineralogical tests (XRD and XRF) results has shown the presence of a strict relation between the mechanical rock properties and their mineralogical composition. It has been concluded, however, that the carbonate content (strict correlation with Young's modulus obtained in mechanical tests) and the

sum of clay minerals (strict correlation with the Poisson's ratio) are better indicators of brittleness of the Ordovician and Silurian rocks from the Polish Basin than standard one. The SiO₂ content or the quartz content (sum of quartz and feldspars), used as the brittleness indicator in other basins, has a restricted application in the Polish Basin. Because the Ordovician and Silurian rocks reveal a very good correlation with the content of carbonates determined by the XRD method and the CaO content using the XRF method, it seems that XRF measurements may aid in fast assessment of the brittleness of the studied medium. From the other hand the tomographic data revealed that for the investigated clay-silt core samples, the cracks that appeared during the compression tests followed tectonic structures of the investigated rocks. The micro-CT results showed the necessity of sampling very homogeneous samples, because the presence of tectonic cracks could influence the results of deformational tests.

ACKNOWLEDGMENTS

The authors of this paper are grateful to PGNiG S.A. for the possibility to use this data.

REFERENCES

1. Stock S.R., 2009: *MicroComputed Tomography: Methodology And Applications*, CrcPress, Boca Raton, USA, 332p.
2. ISRM 2007. Suggested Methods for Determining the Uniaxial Compressive Strength and Deformability of Rock Materials [in:] *The Complete ISRM Suggested Methods for Rock Characterization Testing and Monitoring: 1974-2006*. Ed.: Ulusay R., Hudson J.A., s. 137-140.
3. ASTM Designation: D 2664-95a, *Standard Test Method for Triaxial compressive strength of Undrained Rock Core specimens without Pore Pressure Measurements*.
4. Hallbauer D. K., Wagner H., Cook N. G. W. 1973. *Some observations concerning the microscopic and mechanical behaviour of quartzite specimens in stiff, triaxial compression tests*. Int. J. Rock Mech. Min. Sci. 10. London.713-726.
5. Rietveld H. M., 1969. A profile refinement method for nuclear and magnetic structures. *Journal of Applied Crystallography* 2: 65–71.
6. Taylor C.J., Hińczak I., 2006: *Rietveld made easy: a practical guide to the understanding of the method and successful phase quantifications*. Published by Sietronics Pty Limited – Australia.
7. Środoń J., Victor A., Douglas K., McCarty, J. C.C. Hsieh, D.D.Eberl, 2001. Quantitative X-ray diffraction analysis of clay-bearing rocks from random preparations. *Clays Clay Miner.* 49: 514-528.
8. Jarvie D.M., 2008. Unconventional shale resource plays: shale-gas and shale-oil. Opportunities. Fort Worth Business Press Meeting, June 19. (www.barnettshalenews.com/documents/dan_jarvie.pdf)
9. Ross D.J.K., Bustin R.M., 2008. Characterizing the shale gas resource potential of Devonian–Mississippian strata in the Western Canada sedimentary basin: Application of an integrated formation evaluation. *AAPG Bulletin* 92: 87-125.