CORRELATION OF MINERALOGICAL INDICES OF BRITTLENESS WITH ACOUSTIC PROPERTIES OF ROCKS IN BASINS OF DIFFERENT DIAGENESIS LEVEL

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ABSTRACT
Effective hydraulic fracturing is the basic requirement to get economically profitable gas production. The paper presents results of comparison of two different ways of estimation of rock susceptibility for fracturing: the mineralogical (sum of quartz and feldspars content) and chemical parameters (content of SiO_2) with acoustic moduli (Young's Modulus and Poisson's ratio). The study was conducted for two rock formations with extremely different level of diagenesis: the Polish Miocene and the Silurian-Ordovician basins. Detailed mineralogical studies of all samples were undertaken by three different methods: firstly XRD and XRF, and after that for chosen samples FTIR. The acoustic measurements of the Vp and Vs were made on cuttings. Based on this data mechanical moduli were calculated and then correlated with mineralogical data. The results show that mechanical moduli are less dependent on mineralogy than on petrophysical and sedimentological features of rocks. The application of any indicator of brittleness needs empirical calibration for each specific stratigraphic unit.

INTRODUCTION
During shale gas prospecting, rock brittleness, defined as the susceptibility of rocks to fracturing, is very often determined by its mineral (as a quartz content [1]) and chemical composition (as a SiO_2 content [2]). In fact the brittleness is derived not only from their mineralogical/chemical composition, but also degree of compaction, internal structure or presence of amorphous material of different kind (for example organic matter, amorphous silica, carbonates or iron(III) oxide-hydroxide).

Method, which takes all these rock parameters into account and also provides the information about the mechanical properties of rocks, is the analysis of velocities of acoustic wave propagation Vp/Vs. On the bases of Vp/Vs measurements the dynamic Young's modulus or Poisson's ratio are calculated. Both these parameters have significant impact on hydraulic fracture geometry [3]. Evaluation of the relations between mineralogy and acoustic properties of rocks for the specific basin could be used also to determine the most appropriate horizons for hydraulic fracturing in the regional scale on the ground of the seismic measurements.

In the present paper results comparing two different ways of quantifying rock brittleness are described. The study was conducted for two rock formations with extremely different levels of diagenesis: the Silurian-Ordovician and the Miocene basins from Poland.

Moreover, Vp/Vs tests were run on cuttings, owing to the fact that from the most of boreholes they are the only available rock material. It is especially important to verify if the results observed on cuttings are representative.

EXPERIMENTAL

Materials

Two sedimentary basins, chosen as a study material, are very precisely mineralogically, geochemically and petrophysically characterized. The Silurian-Ordovician basin, which is now the main object of the shale gas exploration, had very complicated thermal history. Shales of that age are thermally mature and contain highly illitic (illite-smectite <15-20 % of smectite particles) in mixture with illite and chlorite, quartz, feldspar and carbonates (mainly calcite, dolomite and ankerite). In the whole profile the carbonate enriched layers are present. The autochthonous Miocene basin of the Carpathian Foredeep is one of the oldest targets of petroleum exploration in Poland with big resources of biogenic gas. A siliciclastic series (Upper Badenian–Sarmatian), which was sampled, constitutes the main segment of the succession. The Miocene rocks display very low diagenesis level. The studied rocks were buried not deeper than 500-1200 m and they contain highly smectitic illite-smectite (more than 70 to nearly 100 % of smectite particles) as a main component of clay fraction. Other components of examined shales are similar as in the Silurian-Ordovician samples, only with some admixtures of kaolinite and anhydrite.

Methods

Detailed mineralogical studies of all samples were undertaken by three different methods: firstly XRD, XRF for a bigger set of samples and after that FTIR for samples prepared for Vp-Vs tests. For XRD and XRF measurement about 400 g of one sample was initially milled and sieved to pass 0.5 mm. Then it was homogenized and splitted by quartering to get a representative sample (weighing about 6 g).

Quantitative XRD analysis Quantitative XRD analysis was conducted by the internal standard method according to the procedure proposed by Środoń et al. [4], which was prepared specially for clay-bearing rocks. Zinc oxide ZnO was used as a standard. Results were calculated with the use of the RockJock program [5], which simulates the experimental diffraction pattern on the basis of the pure natural standards of minerals. The quantitative measurements were conducted using Panalytical X'Pert X-ray diffractometer equipped with Cu lamp, curved graphite monochromator and proportional counter. Voltage 40 kV, current 40 mA, step-width 0,02°2Θ were applied, samples were scanned from 5° to 65° 2Θ.

Preparations were made according to the method introduced by Środoń et al. [4]. Zincite (ZnO) was added to the samples as an internal standard, what enabled to control the quality of the analysis. Samples were ground for 5 minutes in the McCrone micronizing mill in order to reduce the grain size to < 20 µm. The 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.

Semi-quantitative FTIR analysis - The Fourier transform infrared spectroscopy (FTIR) was applied to control the mineralogical representativeness of cuttings which was used for Vp/Vs tests. The procedure was developed on basis of paper Reig et al [6]. The

measurements were carried out on a Thermo Nicolet 6700 FTIR spectrometer equipped with a DLaTGS detector and an XT-KBr beam splitter on diamond ZeSe Specac ATR (Attenuated Total Reflectance) accessory. The mid infrared FTIR spectra were recorded in a range of 580 - 4000 cm^{-1}. For each sample 128 scans were obtained at a resolution of 4 cm^{-1} for one measurement, and additionally it was independently repeated at least for three times. Before the measurement samples were ground in an agate mortar for 3 min.
The spectra were analyzed with the Omnic 7.3 software. As an index comparing the results of XRD and FTIR methods the ratio of absorption value of 872 cm^{-1} band of calcite to absorption value of quartz (795 cm^{-1}) was used.
CWT measurements Laboratory measurements of P and S wave velocities were performed using the CWT-200 Temco ® equipment on small core pieces (cuttings) with 0.8 – 5 mm thickness. Measurements were carried out at surface conditions (temperature and pressure) following the procedure proposed by [7]. The quality and integrity of cuttings were controlled. If it was possible 2 or 3 core pieces were prepared from one sample and in that case the average value was computed to minimize the selection errors (mainly differences in mineralogical composition and internal structure between samples). Before the measurements cuttings were mechanically processed to achieve the flat element (with parallel surfaces) with 1 – 3 mm thickness and saturated with brine. Next, each prepared sample was placed between two transducers of CWT apparatus (emitter and receiver) and P and S wave velocities were measured.

RESULTS AND DISCUSSION

Correlation of the mineralogical and chemical indexes of brittleness for a whole set of samples is presented in Figure 1. As a mineralogical index of brittleness a sum of quartz and feldspar content was assumed, because both those minerals have very similar mechanical properties. Bowker [1] used only quartz content for the Barnett Shale formation, due to the fact that feldspars were present there in negligible amount.
For both sets of data, very high coefficients of square correlation (R^2 = 0.80-0.90) were achieved, which indicates good measurement quality. The lithological differences between the two analyzed basins are responsible for the occurrence of different regression trend lines. One of the possible reasons could be the presence of the swelling clay minerals (highly smectitic illite-smectite) or amorphous silica in examined samples, which is common in rocks at an early stage of diagenesis. It is a general problem in quantitative X-ray diffraction measurements, as this method is weakly sensitive to the small amount of amorphous material. For that reason the content of whole silica may be underestimated and that affects the results of the calculated brittleness too.
The FTIR ATR analysis was applied in order to verify if the cuttings chosen for acoustic measurements correspond mineralogically to the homogenized samples used for XRD and XRF measurement. Very good correlation of the results was obtained by the methods, shown in Figure 2, confirms that they were properly selected.
Mineralogy and acoustic properties of rocks for the specific basin could be used also to determine the most appropriate horizons for hydraulic fracturing in the regional scale using seismic measurements.

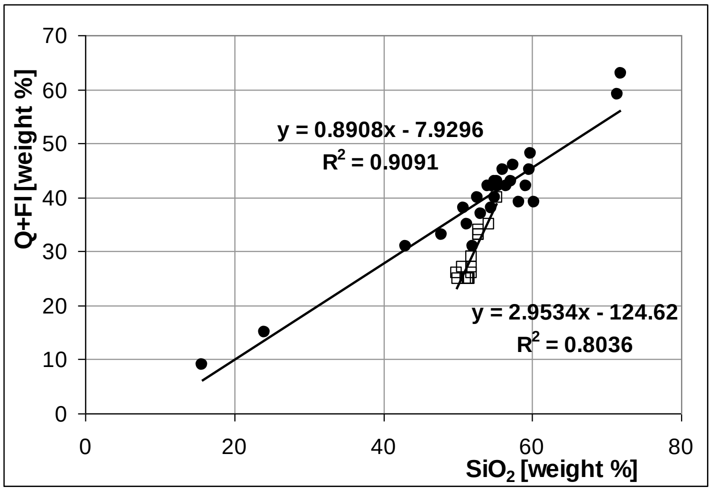


Figure.1. Correlation between the mineralogical and chemical indexes defined respectively as the sum of quartz and feldspars content and SiO_2 content. Blue diamonds – the Silurian – Ordovician set of data, pink squares - the Miocene set of data.

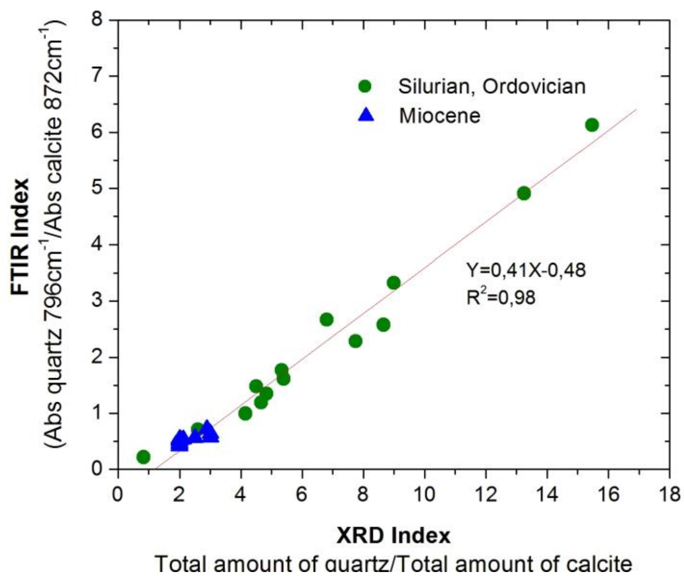


Figure.2. Correlation of FTIR and XRD results on the basis of quartz to calcite content ratio for mineralogical representativeness control of cuttings used for Vp/Vs tests.

Obtained values of the acoustic wave propagation Vp/Vs are generally lower for the Miocene shales compared to the Silurian-Ordovician ones (Figure 3). The most possible reason is the difference in level of diagenesis between them. The Miocene rocks contain more water and have far higher porosity. A wide range of values achieved for the same samples of the Silurian-Ordovician rocks could be the effect of big differences in carbonates and organic matter content, clearly visible even in the macroscopic observations.

The relations between mineralogy and the mechanical moduli are shown on two plots in Figure 4. The trend for the whole set of data is not clearly visible, so the detailed analysis for the smaller dataset corresponding the smaller stratigraphic units was performed.

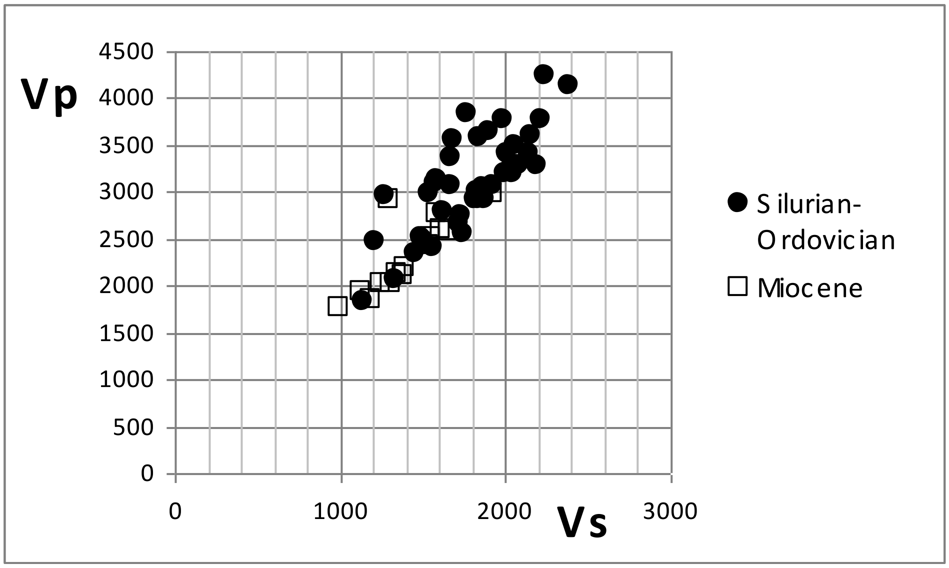
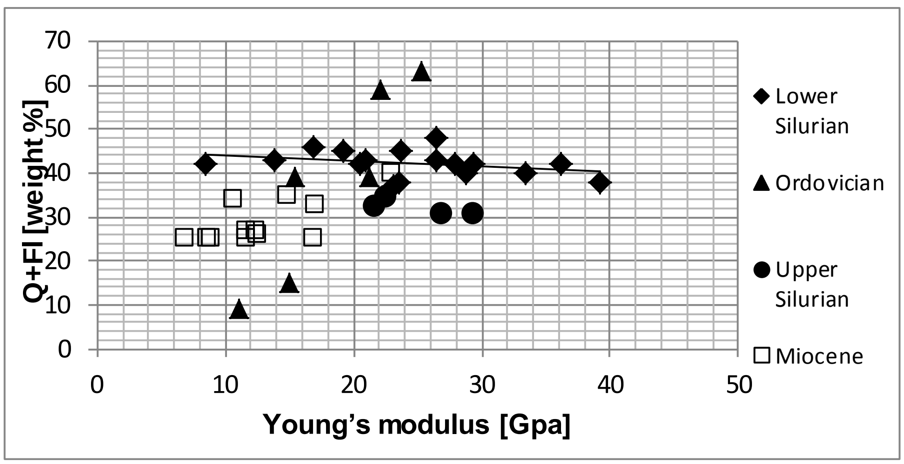


Figure.3. Correlation of the acoustic velocities Vp/Vs. Blue diamonds – the Silurian – Ordovician set of data, pink squares - the Miocene set of data.



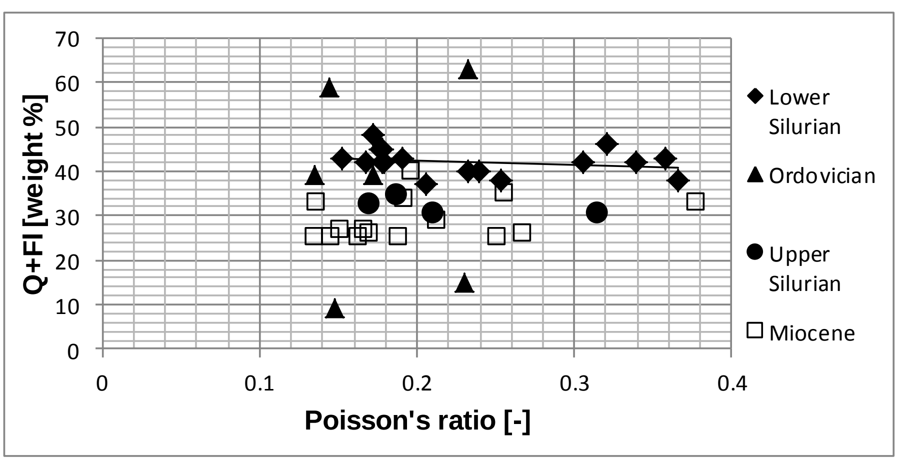


Figure.4. Relation between the mineralogical parameter of rock brittleness and the acoustic moduli: a) Young's modulus, b) Poisson's ratio.

For the Young's modulus the similar phenomena as for acoustic velocities is observed (Figure 4 a). Generally lower values of the parameter were obtained for the Miocene shales. The wider range of values was achieved for the Lower Silurian, what could be connected with big differences of the organic matter content (unpublished data) which are largest for rocks of that age. Generally data for each stratigraphic unit are grouped together and have own correlational trend, although not very strong. For the Poisson's ratio (Figure 4 b) the situation is even more complicated, but the same tendencies are still visible. Evidently distinct relation between the mineralogical parameter of brittleness and the mechanical moduli for each stratigraphic unit show that the strong influence on the values of that moduli have specific sedimentary structures (observable in the macroscopic and microscopic scale) and petrophysical properties of rocks.

CONCLUSION

Performed correlations showed clearly that the mineralogical/chemical parameters of brittleness do not agree unequivocally with the acoustic indicators. For the specific formation, however, it is possible to evaluate the unique relation between the mineralogical and petrophysical features of rocks, and its application as an indicator of brittleness. The mineralogical and chemical indices of brittleness should be treated primarily as lithological indicators, and only secondly indirectly as a gauge of brittleness.

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