

# **POSSIBILITIES OF QUANTITATIVE EVALUATION OF POLISH SHALE-GAS ROCKS PETROPHYSICAL AND GEOMECHANICAL PARAMETERS BY LABORATORY ANALYTICAL METHODS AND WELL LOGS INTERPRETATION**

*G. Leśniak<sup>1</sup>, R. Cicha-Szot<sup>1</sup>, M. Stadtmüller<sup>1</sup>, M. Mroczkowska-Szerszeń<sup>1</sup>, L. Dudek<sup>1</sup>,  
G. Tallec<sup>2</sup>, A. Butcher<sup>2</sup>, H. Lemmens<sup>2</sup>, P. Such<sup>1</sup>,*

<sup>1</sup>Institut Nafty i Gazu - Państwowy Instytut Badawczy, Krakow, Poland

<sup>2</sup>Oil & Gas FEI Visualization Sciences Group, Merignac, France

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

## **ABSTRACT**

The composition of minerals combined with the knowledge about 3D distribution of minerals in the reservoir rock (petrological analysis – detritical grains, cements) and petrophysics translates into its mechanical properties and behavior after application of technologies like hydraulic fracturing - aiming at creating or enhancing the pore connectivity and permeability and therefore enabling the hydrocarbons flow into a well bore. Moreover, the issue regarding resolution improvement is particularly important in the aspect of unconventional hydrocarbon exploration mainly due to reservoir rock revealing micro pore structure.

In the paper, comprehensive study of the Polish shale rock formation samples microstructure, combined with mineralogy and its correlation with acoustic velocities measured in frequency domain of MHz, in order to determine and integrate better data input to geomechanical reservoir model was shown. Mercury intrusion, helium pycnometry, argon adsorption are commonly used analytical methods to determine the pore space characteristics in rock samples. Recently FIB/SEM technique, for pore space morphology and its 3D reconstruction, appeared as a method more and more frequently used by leading companies providing geological services. In this work we would like to show integration of nitrogen adsorption method with FIB/SEM data which seems to be powerful tool for shale rock extensive examination. All above mentioned methods should also lead to development of shale rock physical model explaining better well log data for example in terms of X-tended Range Micro Imager (XRMI) results interpretation.

## **INTRODUCTION**

During development of shale gas reservoir exploration there will be less and less parametric wells from which cores are available and extensive research program covering standard and special core analyses is performed. It is due to demand of oil and gas

companies to reduce drilling time and cost and their joyful faith in geophysical well logs, despite the cost of well logging is generally more expensive than laboratory analyses. Moreover, the possibility of control well trajectory during drilling horizontal wells, based on new sets of logs force to perform the same measurements in the vertical sections. Therefore, calibration of well logs, not only new ones but also classic ones from which we have data from 70's and 80's of 20<sup>th</sup> century, becomes very relevant. Finding the relationship between well logs and petrophysical, geomechanical and mineralogical parameters will improve interpretation of well logs.

Probably in the coming years laboratory analyses will be performed only on side-wall cores or cuttings. Conducting analyses on cuttings might be problematic because of novel polymeric and oil based muds which are hard to remove from rocks and small percentage of rock samples from PDP drill bits which are suitable for analysis. That is why most of the analysis will have to be performed on side-wall cores from interesting formations like sweet spots and caprocks.

To be ready for this challenge methodology of transferring analytical data, which gave comprehensive rock characteristic, to well logs parameters has to be developed in order to estimation geomechanical parameters of separated facies.

## **MATERIALS & METHODS**

### **Samples**

In this study a 450 m of core from Silurian and Ordovician shale formations of Baltic basin were examined. It was mainly claystones, mudstones and siltstones with very thin layers up to few mm.

### **Methods**

In order to characterize selected facies petrographical, petrophysical, mineralogical and geomechanical analyses were performed. Dynamic porosity was calculated from the measurement performed on an AutoPore IV mercury porosimeter. The samples have been analyzed by an adsorption analysis method (argon adsorption in temperature of liquid nitrogen) in Micromeritics Tristar 3020 pore space analyzer. Moreover, to verify mineral composition of the samples examined in Focused Ion Beam Scanning Electron Microscopy (FIB-SEM), Fourier Transformed Infrared Spectroscopy (FTIR) method was applied, and the results were correlated with X-ray diffraction measurements. FEI Helios 660 and mineral mapping were performed in the FEI Qemscan 650FEG microscopes. The mid infrared FTIR spectra in a range of 580 - 4000  $\text{cm}^{-1}$  were recorded on Thermo Nicolet 6700 FTIR spectrometer equipped with DLaTGS detector and XT-KBr beam splitter. The measurements were performed on a diamond ZeSe Specac ATR accessory. The sample was carefully ground before the measurement in an agate mortar for 3min, and 128 scans were recorded in a resolution of 4 $\text{cm}^{-1}$ . The measurement was independently repeated three times to verify the results.

Quantitative XRD analysis was conducted with the use of the internal standard method according to the procedure designed for clay-bearing rocks. Zinc oxide ZnO was used as a standard. Results were calculated with the use of the Siroquant programme, based on

the Rietveld method. Quantitative measurements were carried out using a Panalytical X'Pert Pro diffractometer with a modern ultra fast detector (real time ultra strip X'Celerator). A voltage of 40 kV, current of 40 mA, and step-width  $0.02^\circ 2\Theta$  were applied, and samples were scanned from  $5^\circ$  to  $65^\circ$  in  $2\Theta$ .

Representative samples for each facies were examined using triaxial compression tests and interpretation of the results was performed using procedures of the company MTS, which are based on the ASTM 2664-95a standard [1] as well as the recommendations of ISRM's „Suggested Method for Determining the Strength of Rock Materials in Triaxial Compression” [2]. The aim of the strength tests is first of all determination of the elasticity parameters: Young's static elasticity modulus (E) and static Poisson's ratio. Moreover, for all samples S and P wave propagation in frequency domain of MHz and reservoir temperature was performed and selected samples were examined in HTHP reservoir conditions. All measurements were performed on oriented core samples, what compared with XRMI allows to determine stress and fracture azimuths.

## RESULTS & DISCUSSION

Based on well logs in parametric well geophysical facies (electrofacies) might be distinguished. Electrofacies can be described as a rock type sediment exhibiting similar set of well log responses [3]. This classification does not require any artificial data subdivision of data population but follows naturally based on the unique data values reflecting minerals and lithofacies within the interval. Procedure of facies isolation is based on finding characteristic value ranges for microelectrical curve for analyzed interval. Calibration of electrofacies separation procedure was done based on quantitative laboratory analyses and its accuracy strongly depends on the available data statistic.

For analysed interval, based on the variation of resistivity registered by XRMI log, four basic electrofacies: tuffites, siltstones, shales and calcereous were distinguished.

Information from well logs and laboratory data is gathered at different scale which vary in resolution, spatial coverage and number of parameters measured. In order to correlate medium resolution well logging (resolution of several centimeters, coverage of several meters, and numerous parameters) with laboratory measurements from which numerous parameters at discrete depth of formation can be determined there is a need to scale up or down to increase reliability of prediction [4].

General idea of calibration and then prediction of geomechanical parameters was shown on Fig.1. For each separated based on XRMI and sedimentological analysis facies set of analysis covering petrography, mineralogy, petrophysics and geomechanics was performed. It allow unambiguously determine mineral composition, 3D mineral distribution and pore space and link it to geomechanical parameters of analysed facies. This procedure let us estimate geomechanical parameters in wells in which only well-side cores and cuttings are available, from which only more challenging information about rock formation might be obtained. The challenge in data analyzing might be caused by cutting inertia in the well and proper matching with depth and well logs which give us information in kHz frequency domain. In the case of analysis on cutting there is a need to verify mineral composition of the sample, in order to check if analyses are performed on

representative sample, which might be performed by quick FTIR-ATR (Fourier Transformed Infrared Spectroscopy - Attenuated Total Reflection) analysis or XRD. Scanning electron microscopy analysis (FIB-SEM, mineral mapping), petrophysical analysis and wave velocities ( $V_p$ ,  $V_s$ ) using Continuous Wave Technique can be easily obtained on the core or cutting samples. However, laboratory measurement of P and S wave velocities performed in the frequency domain of MHz provide shift of velocities observed in log response (Fig.2a, b), straight correlation between mineral composition can be obtained. In the case of HTHP measurements the shift is negligible in the case (Fig.2c).

In order to have coherent information, comprehensive understanding not only rock mineralogy but also pore space is needed. Commonly used laboratory methods do not cover pore ranges which are observed in shale rock samples and might be lower than 3 nm. Moreover, small amount of rock sample will indicate additional limitations and destructive measurement like mercury porosimetry might have to be avoided.

In this study, good correlation between FIB-SEM data and mercury porosimetry as well as SEM mineralogy and XRD was found. The main advantage of SEM microscopy is  $\mu\text{m}$  or even mm sample size which allow to estimate porosity and mineralogy on cuttings. However, this method have some limitations in case of pore space parameters determination like specific surface area which may be much affected by contribution of micropores (less than 2 nm), combined with isotherm adsorption analysis can give full information about pore space.

Pore space analysis of investigated rocks shows non-modal distribution of pore space. Generally, three groups of pores might be distinguished. First group are macropores with diameters over 8  $\mu\text{m}$ . Second group are pores with diameter from the range 100 - 1000 nm. Third group of pores, which was distinguish by isotherm adsorption method, have diameter lower than 10 nm and are connected to organic matter what was confirmed by FIB-SEM (Fig.3). Presence of micropores may explain discrepancy in  $V_p$ ,  $V_s$  velocities in formation rich in organic matter (Fig. 2a lower part)

Moreover using mineral mapping we can easily distinguish thin layers with different mineral composition which may also affect P and S wave propagation.

## CONCLUSION

XRMI data are highest achievable well log resolution and give possibility to correlate laboratory data. Result of this correlation should be used in a up scaling procedure from laboratory analyses to the standard logs and further for seismic and structural models. Precise work flow planning is essential in order to omit artefacts. Moreover, statistical representations of samples analyzed in especially in micro scale is crucial to obtain coherent results from different analysis.

In order to predict geomechanical parameters in wells from which there is no core material, but geological formation was characterized before in another parametrical well, extrapolation of petrophysical and geomechanical data is possible but should be verified on cuttings or side-wall cores by instrumental analysis and microscopic methods.

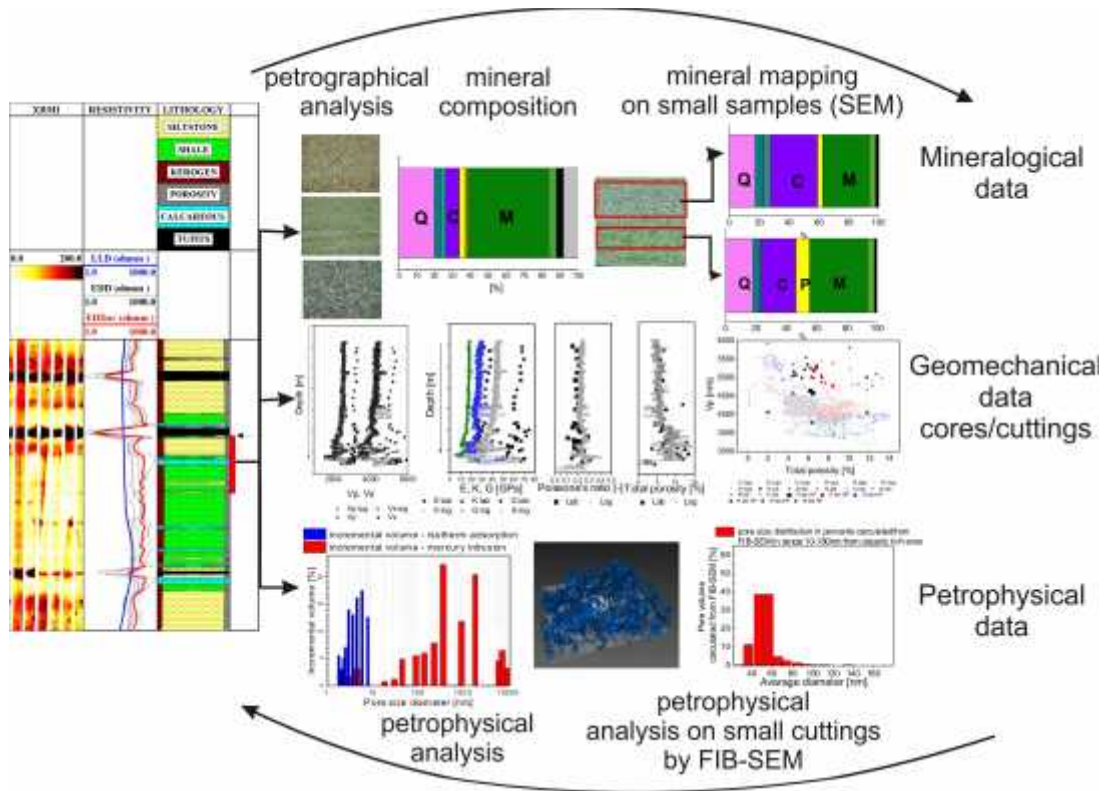


Figure 1 General idea of calibration of electrofacies separation procedure and prediction of geomechanical parameters

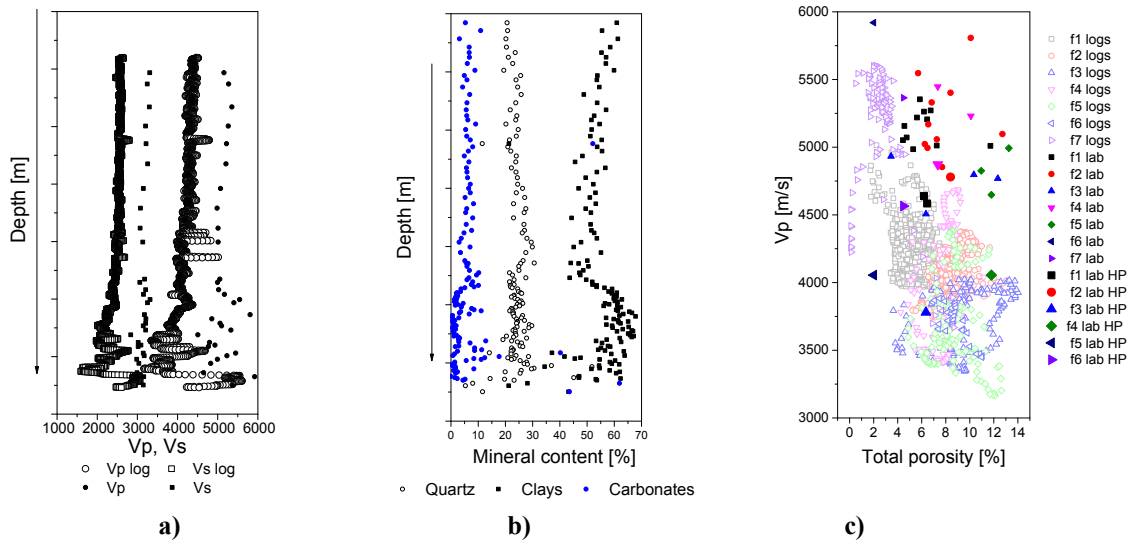
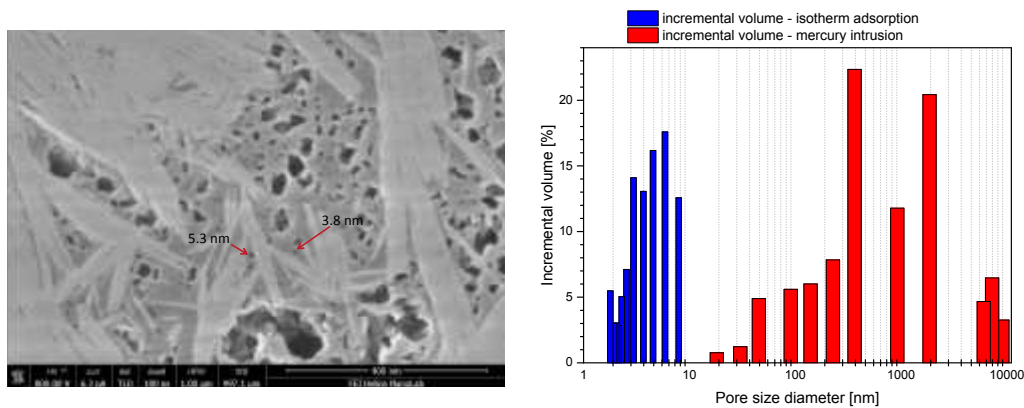


Figure 2 Shift of a) velocities from logs and laboratory data shown with b) mineral composition. The biggest discrepancy was observed for samples with high clay or carbonates content. An assembly of c) Vp vs porosity data for separated formations (f1-f7) for geophysical and laboratory data at reservoir temperature or HTHP conditions (HP) – observed trends are similar to literature data [5]



a) **Figure 3 Porosity on organic matter a) and pore size distribution b) by mercury intrusion and isotherm adsorption analysis for selected shale sample**

## ACKNOWLEDGEMENTS

The authors gratefully acknowledge the support of Polish-Norwegian Research Programme operated by the National Centre for Research and Development under the Norwegian Financial Mechanism 2009-2014 in the frame of Project Contract No Pol-Nor//196923/49/2013, Blue Gas Programme operated by the National Centre for Research and Development in the frame of Project Contract No BG1/MWSSSG/13 from which the research leading to these results was partially founded and Ministry of Science and Higher Education for financial support of research project archive number DK-4100/37/2014-01.

## REFERENCES

1. ASTM Designation: D 2664-95a, Standard Test Method for Triaxial compressive strength of Undrained Rock Core specimens without Pore Pressure Measurements.
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., p. 137-140
3. Kumar A., Sinha S. K. Total organic carbon prediction for shale gas exploration using statistical clustering, multiple regression analysis, 10<sup>th</sup> Biennial International Conference and Exposition, 2013
4. Prasad M. "Velocity-permeability relations within hydraulic units" *Geophysics*, vol.68 no.1 pp. 108-117
5. Jaeger J. C., Cook N. G. W., Zimmerman R. "Fundamentals of Rock Mechanics", 4th Edition, Wiley-Blackwell, ISBN: 978-0-632-05759-7, April 2007 pp. 323-367