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PETROPHYSICAL CHARACTERISTICS OF THE SHALE GAS FORMATIONS IN THE BALTIC BASIN, NORTHERN POLAND

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Abstract: Results of laboratory experiments combined with well logging to get information on density and porosity together with mineral composition were the basis for shale gas rock model construction. Density – bulk, skeletal and grain determined from various laboratory experiments and porosity – total, effective/dynamic and from dual liquid immersion measurement, well correlating with TOC and volume of kerogen, showed the place of organic matter and gas in pore space and the skeleton of claystone-mudstone gas formations. Results of pyrolysis (Rock-Eval), i.e. TOC (Total Organic Carbon) used to determine the hydrocarbon generation potential of rocks were also considered together with element composition from chemical analyses and mineral composition from XRD measurements. SCAL analysis, NMR experiments, Pressure Decay Permeability measurements together with water immersion porosimetry (WIP) and adsorption/desorption of nitrogen vapors (77K) method were realized and the comprehensive interpretation of the outcomes was done. Absolute permeability, pore diameters size, total surface area from Hg porosimetry were compared with outcomes of nitrogen physical adsorption/desorption method. It enabled extension of pore diameter range into extremely small values. The nitrogen adsorption/desorption method also contributed to porosity values, adding notable volume of the smallest pores. The results from NMR experiments confirmed a complicated porous space structure and showed that total surface area from adsorption/desorption of nitrogen method together with pore space area from Hg porosimetry correlate well with the irreducible water saturation. Laboratory results were combined with well logs (GR) and results of the comprehensive interpretation (PHI) and volumes of mineral components, for instance illite/smectite and kerogen. GEM logging results were also used to improve volume of carbonates in shale gas rocks matrix and organic/inorganic coal. Identification of Fe element was useful in pyrite recognition. Another important factor was including the sedimentological macroscopic description of cores before cutting plugs and geological homogenization of samples and adopting the proper sequence of laboratory experiments to be sure that all properties were determined on the same part of the rock material.

Keywords: *petrophysics, well logging, shale gas formations, mineral composition, reservoir properties*

INTRODUCTION

Unconventional shale gas formation has been the subject of intensive exploration in Poland since the late 1990s. The most promising regions were considered to be sedimentary basins placed along the Teisseyre-Tornquist zone from the north-west to south-east of Poland. The investigations were focused on the Silurian and Ordovician thick shale formation which spread in Poland along the western margin of the East European Platform in the Baltic, Lublin and Podlasie Basins (Kiersnowski 2013; Narkiewicz et al. 2014). Large archive core material and well log data indicating the presence of hydrocarbons after geological research conducted in the 1960s, ’70s and ’80s were also available and included for reinterpretation. New geological cores acquired from several wells drilled by POGC SA and LOTOS-Petrobaltic SA in recent years confirmed presence of gas in shale formations. New methods of laboratory experiments and methodology of well logging measurements and comprehensive interpretation were applied to possess more and more detailed information (Jarzyna–Wawrzyniak-Guz 2017b). Several scientific elaborations have conducted to recognize Polish shale gas formations and compare them to other world-wide shale plays known from the literature (see e.g. Raport PIG-PIB 2012).

GEOLOGICAL SETTING

The object of the analysis covers two formations, potential resources of unconventional hydrocarbons: the Ja Member of the Silurian Pa Formation and Ordovician Sa Formation. In these formations sedimentation of organic-rich black shales with graptolites as the main fossil started in the Late Llanvirnian reaching Wenlock (Podhalańska 2003). In the analyzed wells the Pa Formation is placed between Pr Fm (lower boundary) and Pe Fm (upper boundary). The lower boundary is marked at the point of clear lithological changes involving the replacement of limestone or marl deposits of the Ordovician series into clay sediments of the Pa Formation. Ja Mb belongs to the lower part of the Pa Fm and is built of black bituminous claystones. It is characterized by high organic matter content; however, it may not ensure large gas reserves since the thickness of this bed does not exceed 12 m (Modliński et al. 2006). The lower boundary of the Sa Formation runs on the limestone Ko Fm within Llanvirnian and Llandeilian series while the upper boundary is set by the marl and shale Pr Fm within Ashgillian series *(Table 1)*. The lithology is mainly comprised of black, dark grey and grey-greenish bituminous shales. Shales of Sa Fm and Ja Mb are rich in organic matter with the median values of TOC amounting to 3.1 and 3.0 wt.%, respectively. The highest TOC contents recorded in wells in the study area reached 8.5 wt.% in Sa Fm (Jarzyna et al. 2017a, b).

MATERIALS AND METHODS

Selected results of laboratory experiments were collected for the presented study. Results are related to formations, not to boreholes, to form a general description of the Silurian and Ordovician shale plays. Laboratory methods for density and porosity measurements were pointed out because bulk density and total porosity are values measured in standard well logging.

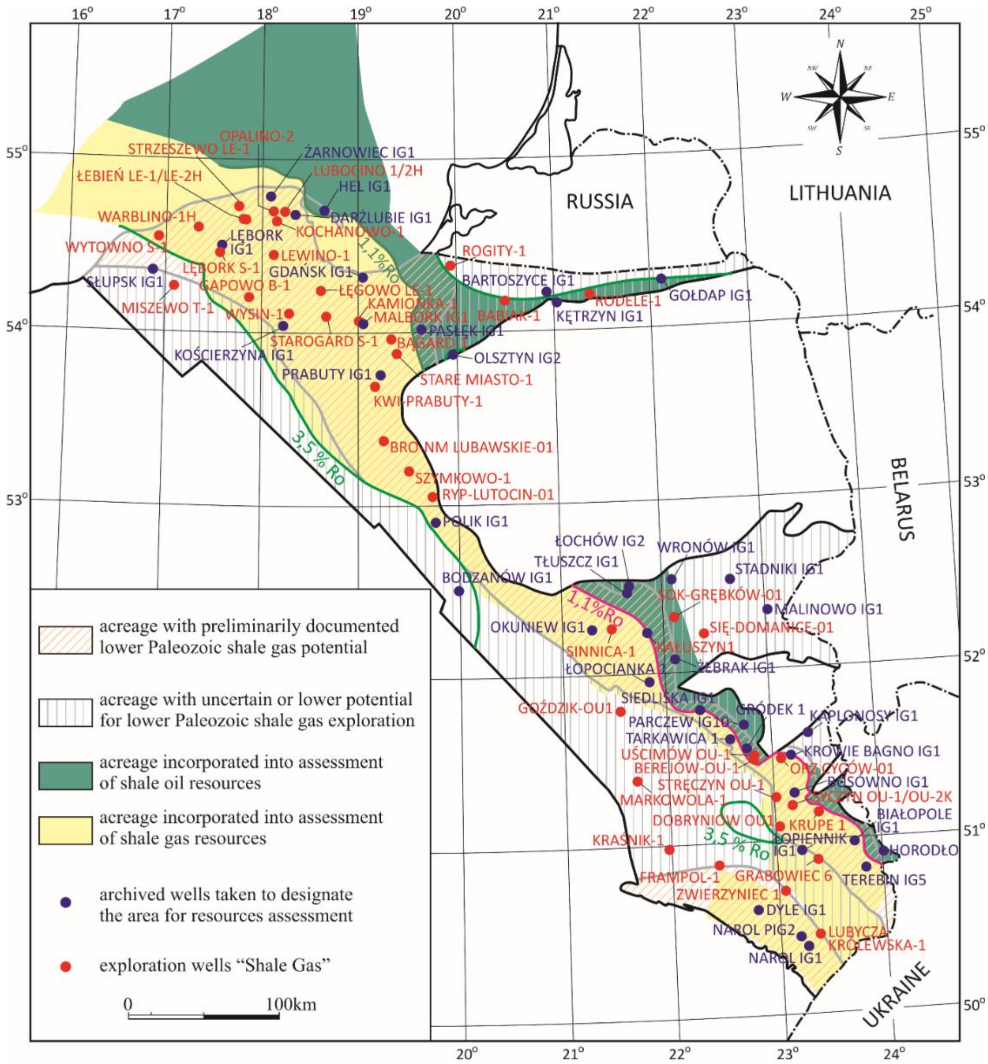


Figure 1. *Occurrence of the Lower Palaeozoic fine-grained rocks potentially accumulating shale gas*
(Kiersnowski 2013, modified)

A great variety of those parameters from the laboratory experiments provided the basis for studying mutual relationships. The list of laboratory measured parameters includes, among others, grain density (GD_{WIP}) and total porosity (Φ_{WIP}) determined by *Water Immersion Porosimetry (Dual-Liquid Porosimetry)* (Kuila et al. 2014;

Topor et al. 2016), bulk density (BD), skeletal density (SD), total pore area (TPA), average pore diameter (APD), effective porosity (Φ_eff) and physical permeability (K) from Hg porosimetry, permeability (K_PDP) and bulk density (BD_PDP) from Pressure Decay Permeability (PDP) method, density (D) from He pycnometry, total (Φ_NMR) and effective porosity (Φ_NMR_eff) together with irreducible water saturation (Sw irr) from NMR experiment. Results of physical adsorption of nitrogen (Rouquerol et al. 2014) were also included in the data set to extend the information from the Hg porosimetry into micropores. On the basis of this method specific surface (S_{BET}) was also determined together with pore volumes in several groups related to small pore diameters. Total organic carbon (TOC) and other characteristic parameters from Rock-Eval pyrolysis (Kotarba et al. 2014) and some petrophysical parameters calculated on the basis of mineralogy and chemical data like the photoelectric absorption index (PEF) or SIGMA were included. Specialist professional equipment was adopted for experiments, such as, AutoPore IV 9500 Micromeritics, PDP Terra Tek Schlumberger Reservoir Laboratory, Quantachrome Autosorb-1C automatic gas adsorption apparatus, Rock-Eval 8 and also some prototype sets for example for water immersion porosimetry.

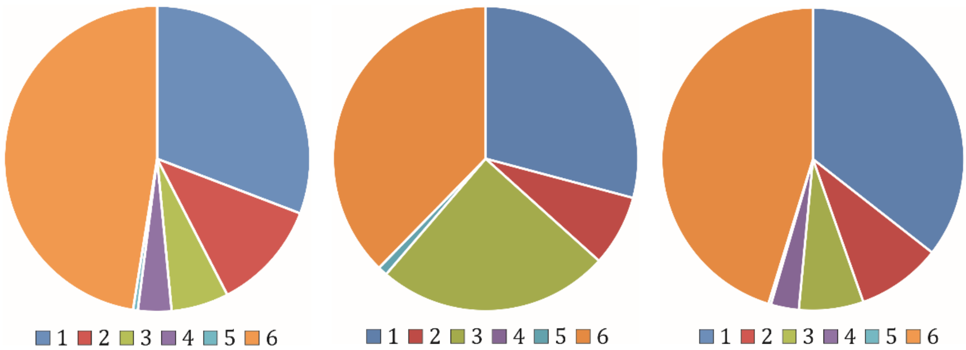
Table 1

Stratigraphic sequence and lithology of formations in the study area (Baltic Basin)

Geological formation	Stratigraphy	Lithology
Pe Formation	Silurian, Ludlow, Wenlock	Claystone
Pa Formation	Silurian, Llandovery	Claystone
Ja Member at the foot of Pa Formation	Silurian, Llandovery	Bituminous claystone
Pr Formation	Ordovician, Ashgillian	Marl, Limestone, Claystone
Sa Formation	Ordovician, Caradocian, Llanvirnian	Bituminous claystone, Claystone
Ko Formation	Ordovician, Llanvirnian, Llandeilian	Limestone

RESULTS

In data sets built for boreholes the number of measured quantities was limited and not the same, so comparison could be done only for selected parameters. Well logging results were considered in the full cored depth sections in each borehole. This paper presents results from the Pa formation including the Ja Member at the bottom, the Pr formation built of marls and claystones and the Sa formation. The mineral composition of formations is presented in pie charts *(Figure 2)* showing the percentages of minerals and groups of minerals: quartz, feldspars, dolomite and ankerite and clay minerals. Pyrite, ankerite, siderite and other minerals including iron element play an important role due their high density in contrast to kerogen and organic matter. The presence of such minerals is also considered from the aspect of shale formation brittleness. Differences in mineral composition between claystone-mudstone Pa formations and bituminous claystones in the Ja Member and Sa formations is distinctly visible.



Pa Formation with Ja Member at the bottom; 1–(29.4), 2–(10.0), 3– (5.6), 4–(2.4), 5–(0.7), 6–(52.3)

Pr Formation; 1–(29.15), 2–(7.54), 3–(24.62), 4–(0.00), 5–(1.01), 6–(37.69)

Sa Formation;1–(35.79), 2–(9.02), 3–(6.89), 4–(2.98), 5–(0.28), 6–(45.24)

Figure 2. *Histograms of the mineral composition in the representative depth sections on the basis of XRD mineralogy results, volume percentage: 1–quartz, 2–feldspar, 3–calcite, dolomite and ankerite, 4–pyrite, 5–others including anhydrite, siderite, hematite, halite, fluorite, bassanite, gypsum, galena, 6–sum of clay minerals*

In each of the formations *(Figure 2)* a relatively high quartz volume is observed. The volume of clay minerals is also high, but they are not further differentiated into illite, montmorillonite (smectite), kaolinite or chlorite.

Table 2

Ranges of the compared parameters

	Silurian, Pa Formation with Ja Member; depth: 2850.15–2906.7 m		**Ordovician, Pr Formation; depth: 2907-2914.9 m**		**Ordovician, Sa Formation; depth: 2915–2940.9 m**	
Numer of data	76		11		42	
	min	**max**	**min**	**max**	**min**	**max**
TOC [% wt.]	0.06	7.30	0.16	0.69	0.08	8.5
Bulk density [g/cm3]	2.38	2.70	2.64	2.72	2.38	2.69
Skeletal density [g/cm3]	2.51	2.81	2.76	2.79	2.46	2.81
Material density [g/cm3]	2.52	2.85	2.76	2.79	2.47	2.90
Kerogen volume [v/v]	0.00	0.12	0.00	0.04	0.00	0.18

Ranges of TOC, bulk, skeletal and material densities and kerogen volume (determined from the comprehensive interpretation of well logs) in the Silurian Pa Formation with Ja Member, Pr Formation and Ordovician Pr and Sa Formations are presented in *Table 2* to illustrate variability among stratigraphic formations. A distinct relation is visible between lithology and density. There are also visible changes in bulk density and skeletal density with depth. The decrease of bulk density with increase of kerogen volume and TOC percentage can be observed.

Relationships between skeletal and material density vs TOC in the Sa Formation are presented in *Figure 3*. Dispersion of both densities is visible and from these plots it is difficult to decide which density better describes shale gas formations. The slope of correlation lines is similar but the bulk density in the Sa Formation of the highest TOC content reveals higher values, which means that kerogen is located in pore space.

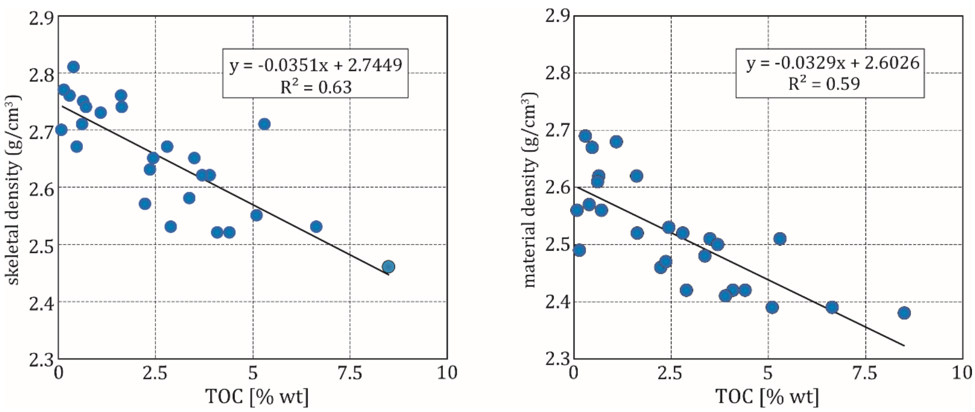


Figure 3

Relationships between skeletal and material density vs TOC in the Sa Formation

Relationships between petrophysical parameters from well logs and results of the comprehensive interpretation: GR anomaly and total porosity – PHI vs. kerogen volume *(Figure 4)* presented separately for the Ja Member and Sa Formation sweet spots show that the intensity of natural radioactivity correlates similarly in both shale gas formations *(Figure 4a)*. Thus, high anomalies in GR may be primary indicators of sweet spots, i.e. places with a higher volume of kerogen. Comparison between PHI and the volume of kerogen shows a higher dispersion of points than previous relationships. PHI presents a similar range of values for both sweet spots. In both formations data are grouped into two parts; in both groups there is a visible increase of kerogen volume with porosity. This indicates kerogen presence in the pore space of the shale formation. For the selected porosity a higher kerogen volume is observed in the Ja Member in comparison to the Sa Formation. The distinct division of Ja Member data into two groups shows that for the same porosity two different volumes of kerogen can be detected. This means that kerogen in the Ja Member is present in two forms, probably filling partially pore space and as organic matter in the skeleton *(Figure 4b)*.

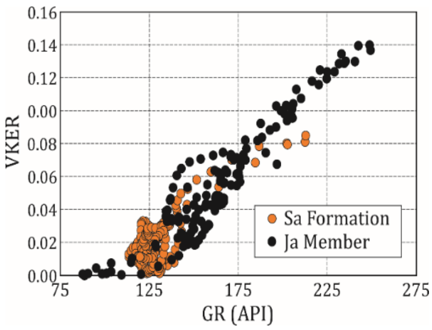


Figure 4a
Relationship between kerogen volume vs. natural radioactivity GR anomaly

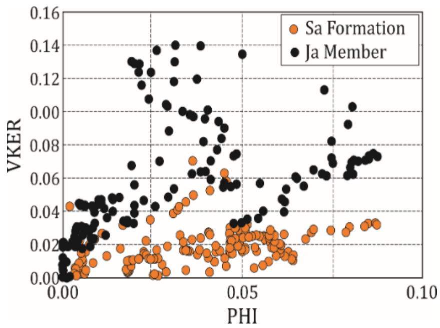


Figure 4b
Relationship between kerogen volume vs. total porosity PHI

Measurements of the low temperature isotherms of nitrogen adsorption (77K) are the basis to determine porous texture parameters of rocks, i.e. specific surface, volume of pores, average pore diameter and pore distributions (Thommes et al. 2015). The low pressure part of the isotherm provides data for microstructure characterization – micropores are filled with porous media at relatively low pressure, based on the Dubinin and Raduszkiewicz theory. In mesopores adsorption is multilayered and at the end capillary condensation takes place (gas is adsorbed in pores as fluid under pressure). Pressure increase causes an increase in the adsorbed medium volume. The method elaborated by Barrett, Joyner and Halenda (BJH) is the mostly used to describe mesopores distribution. Micro- and mesopores influence specific surface which is frequently calculated using Brunauer, Emmet, Teller (BET) equation. Micro- and mespores were also characterized using the Quenched Solid Density Functional method (DFT). Macropores are not very important in specific surface calculations but they are the path for media transport to smaller pores. Pore volume can be simply calculated from the nitrogen adsorption isotherms but the pore size and specific surface determination is based on adopted models. Three examples of adsorption/desorption isotherms and results of their processing are presented *(Figure 5)*. They represent different mudstone-claystone rocks from the Ja Member (a, b, depth: 2896.93 m, BD = 2.39 g/cm^3, Φ_eff = 2.18%, TOC = 5.93%wt), and Sa Formation (c, d, depth: 2920.47 m, BD = 2.97 g/cm^3, Φ_eff = 0.29%, TOC = 2.40%wt and e, f, depth: 2936.85 m, BD = 2.23, Φ_eff = 2.65%, TOC = 2.27%wt). Differences in plots in Figure 5 are distinctly visible. They are partially caused by different mineral composition *(*see *Figure 2)* and variability of bulk density and TOC. Comparison of BJH and DFT curves illustrate the influence of the adopted model for nitrogen adsorption data processing. The most details are revealed in the DFT approach. Hg porosimetry results (poroz.) showed the important role of micro- and mesoporosity in the rocks in study, especially in specific surface determination.

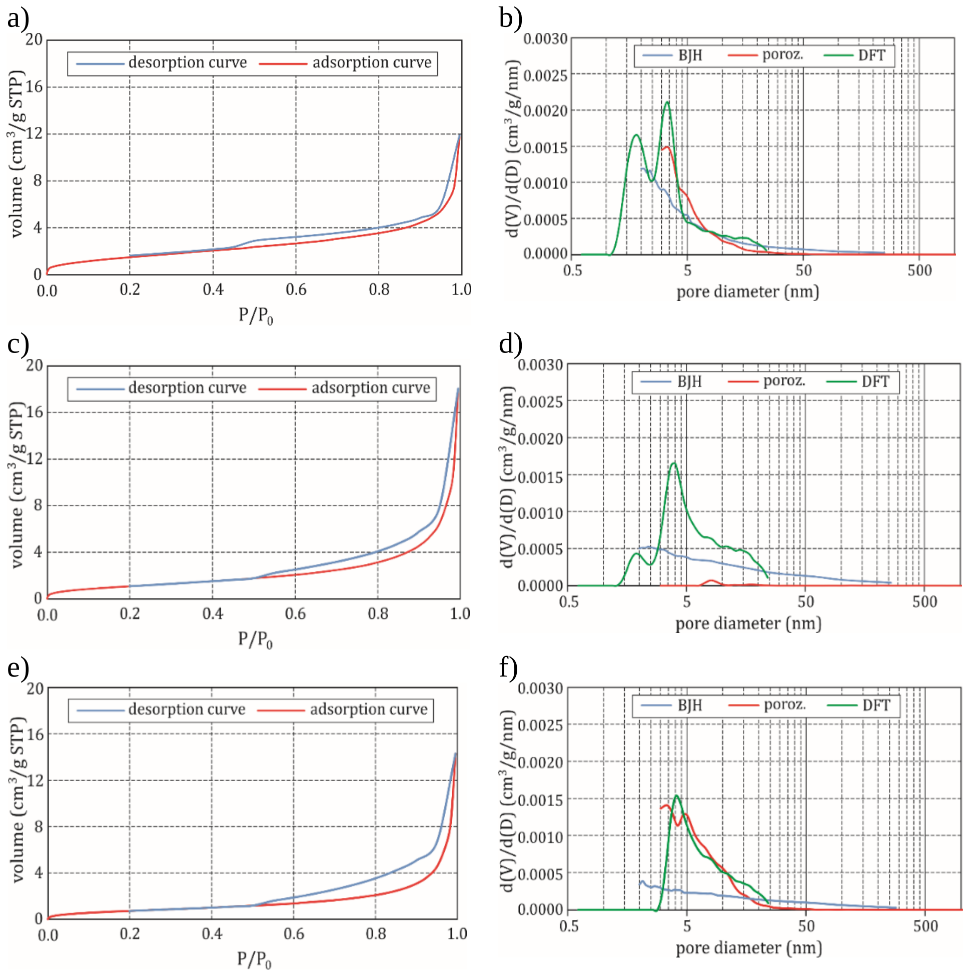


Figure 5

Results of physical adsorption of nitrogen; left plots – adsorption (red) and desorption (blue) curves, right plots – distribution curves, results of processing using Barrett, Joyner and Halenda (BJH) method and DFT method compared to Hg porosimetry result (poroz.)

SUMMARY

As many as possible laboratory techniques and well logging measurements were done to identify mutual relationships between quantities representing petrophysical parameters of shale gas formations. Mineral composition, reservoir properties and geochemical parameters were the basis for sweet spot characterization. Combined results from the complementary methods revealed additional features. Laboratory methods and specific equipment dedicated to shale gas formations investigations were listed along with relevant literature. The same approach was adopted for well logging without including formulas and plots illustrating all mutual relationships.

Examples and references provided the idea of a comprehensive approach to shale gas deposits. A short geological description and depth information was included to provide information on Polish shale gas plays.

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