



GeoShale 2014

**Recent Advances
in Geology
of Fine-Grained
Sediments**

**POLISH GEOLOGICAL INSTITUTE
NATIONAL RESEARCH INSTITUTE**

24–26 September 2014 | Warsaw, Poland

THE 2nd
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Book of Abstracts

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oral presentations

GAS SHALE FORMATIONS IN CARPATHO-DANUBIAN AREA – AN OVERVIEW

.....
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Key words:

shale, gas, Carpathians, organic matter

1

In Romania, potential gas bearing shale formations of *gas shale* type are located in Orogen units, in folded structures (*Fold Belt Basin*) and in platform units (in Carpathian *Foreland Basin*), at depths exceeding 2500–3000 m (Moesian Platform with its extension in South Dobrogea, Scythian Platform (Bârlad Depression), south of Moldavian Platform (a part of East European Platform), and Getic Depression, too.

Objectives. The **age** of formations of interest covers a time interval ranging from Early Paleozoic (Silurian – 425 mln. years) to Cenozoic (Paleogene – 30 mln. years). The Paleozoic ones indicate parameters with optimal values, close to the international standards.

The thickness of formations is variable (100–2000 m), and its values are influenced by the tectonic framework, the paleorelief of the basin during their accumulation and the frequency of drillings which have intercepted such formations; the shale formations have bed like, tabular geometries, with side, interrupted, frequent thinning of a system of major faults.

Procedures. The **thermal gas generating potential** of the argillitic (slate) formations was tested in the Carpathians and extra-Carpathians by grain size analysis, SEM,

Rock-Eval (TOC-wt%, an R_o %), mineralogical and xRay analysis. The main lithological types are represented by shales, dysodiles, menilites, marls alternating with thin levels of sandstones, and the dilution degree of shales and argillites (slate) may reach 50% or may exceed this value.

Results. The **criteria and standards** for the assessment of the gas bearing potential of shales with a content of organic matter are described. The content of Total Organic Carbon (TOC-wt%) may have values between 0 and 12 as the maturity (maturity degree); mature systems have R_o values between 0.6 and 1.35.

Conclusions: The analysis results indicate a **high potential** for the Silurian formations of Moesian Platform, Scythian Platform and Moldavian Platform.

The Eastern Carpathians and the Getic Depression, the Oligocen formations have an **average potential**.

Formations with proper potential are considered those in which the TOC values are higher than 2–4%, the vitrinite reflectance – R_o : 1.5%, with a kerogen of type II–III and which has a higher than 430°C REv-maturation temperature. These formations may be explored.

SHALE GAS POTENTIAL IN CARBONIFEROUS UNITS OF THE SOUTH PORTUGUESE ZONE: PRELIMINARY ASSESSMENT

.....

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Key words:

shale gas, maturation, gamma-ray, South Portuguese Zone, total organic carbon

2

This work reports preliminary results on the potentiality and future perspectives regarding the exploration of shale gas deposits in the South Portuguese Zone (SPZ), based on the analysis of total organic carbon (TOC), maturation (both organic and inorganic) and gamma-ray data.

The SPZ is located in the most meridional sector of the pre-Mesozoic Iberian Variscan Chain which is composed by highly deformed upper Paleozoic rocks of low grade metamorphism. Previous works on maturation analysis for the SPZ are controversial. Results based on organic maturation support the view that the SPZ Carboniferous units are mostly over-matured (framed in the epimetamorphic zone), thus discarding any hydrocarbon accumulation potential. However, results based on inorganic maturation consider that they are mostly located within the gas window (diagenetic and anquimetamorphic zones).

New samples were collected in Mértola, Mira and Brejeira Formations, in potential non over-matured zones, which were identified by using a krigging statistical interpola-

tion approach of previous organic maturation data. Thirty nine samples were processed at Weatherford Laboratories in order to obtain TOC results (39 samples of the Mértola, Mira and Brejeira Formations) and equivalent vitrine reflectance data (5 samples of the Brejeira Formation). The organic maturation of the other collected samples was obtained using the correlation between the Th/K ratio and the reflectance average power.

For the SPZ Carboniferous units studied in this work, the obtained TOC results mostly range between 1.0 wt% and 4.0 wt%, i.e., they can be considered as good to very good quality, regarding its hydrocarbon generation potential. Moreover, according to Weatherford's technical report, the organic matter of the analysed samples was classified as pyrobitumen, thus inducing that the sampled units had probably generated hydrocarbon. As a consequence, previous negative perspectives regarding the exploration of shale gas deposits in the SPZ Carboniferous units based on organic maturation, which discarded any hydrocarbon generation potential, must be reconsidered.

HYDROCARBON ANOMALIES IN THE CARBONIFEROUS UNITS OF THE SOUTH PORTUGUESE ZONE USING THE THORIUM-NORMALIZED METHOD (GAMMA RADIATION)

.....

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Key words:

shale gas, thorium-normalized method, gamma-ray, South Portuguese Zone, Carboniferous

3

This work aims to assess the exploration potential of shale gas concentrations, based on the interpretation of hydrocarbon anomalies recorded in Mértola, Mira and Brejeira Formations. These formations, mostly composed by shaly and sandy turbidites, outcrop in the South Portuguese Zone (SPZ).

The SPZ is located in the southernmost sector of the pre-Mesozoic Iberian Variscan Chain, which is composed by highly deformed Upper Paleozoic rocks of low grade metamorphism. However, in some areas, it is possible to observe the same rocks practically undeformed of the same age.

In order to identify and explain the radiometric anomalies related with hydrocarbon emanations, several models have been developed by different authors. One of the most successful models is the one where the relationship is based on the fact that, in oil fields context, the potassium (^{40}K) decreases and uranium (^{238}U) tends to increase. Thorium (^{232}Th) is considered to be highly retained in local rocks and soils and not to be affected by hydrocarbon ascension or seepages. Based on these assumptions, the ideal potassium and the ideal uranium has been defined in literature as:

$$K_i = (K_{av}/Th_{av}) \times Th_s$$
$$U_i = (U_{av}/Th_{av}) \times Th_s$$

Where "s" refers to the measured or sampled value; "i" is the ideal value; and "av" is the average value for the stud-

ied area, normally, at least five times bigger than the predicted anomaly. The difference between the average and the ideal values is then calculated as follows:

$$KD = (K_s - K_i) / K_s$$
$$UD = (U_s - U_i) / U_s$$

When hydrocarbons are present, KD decreases and UD increases. In order to capitalize these two relationships, a new variable called DRAD has been defined as:

$$DRAD = UD - KD \text{ or}$$
$$DRAD = ((U_s/U_{av}) - (K_s/K_{av})) / (Th_s/Th_{av})$$

Therefore, hydrocarbon anomalies are characterized by positive values of DRAD.

The radiometric data collected in this work, using a portable gamma-ray detector, have been compared to TOC and organic maturation values in order to find and understand the behaviour patterns of these three main elements (^{40}K , ^{238}U and ^{232}Th) and the important radiometric anomalies (DRAD).

In the active oil fields that applied this method as an exploration technique, positive anomalies were also found in 70% to 80% of actual production oil fields. Therefore, the obtained results in the present work are very encouraging, as positive anomalies were achieved in more than half of the studied levels in the SPZ.

OIL SHALE FORMATION IN THE UPPER CRETACEOUS NENJIANG FORMATION OF THE SONGLIAO BASIN (NE CHINA)

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Key words:
Songliao Basin, oil shale, biomarker, bioproductivity, preservation

4

Two oil shale layers were deposited in the Upper Cretaceous Nenjiang Formation of the Songliao Basin, representing excellent hydrocarbon source rocks. Their biomarker compositions provide evidence for a major contribution of aquatic organism (dinoflagellates, green algae, botryococcus) within the photic zone of the water columns. Phytoplankton blooms were promoted by warm-humid climate, and lake eutrophication. Microbial communities included heterotrophic bacteria, cyanobacteria and chemoautotrophic bacteria, as well as purple and green sulfur bacteria. The presence of methanotrophic bacteria is indicated by ¹³C-depleted methylhopane. The sediments were deposited in a eutrophic, alkaline paleolake. Salinity stratification and anoxic bottom water conditions are indicated in the lower oil shale layer, whereas decreased salinity and partial oxygenation of the water column are evident in the upper oil shale layer. A freshwater environment and suboxic conditions in the deep water prevailed during this period. Higher input of terrigenous OM occurred during deposition of the upper

Nenjiang Formation. Therefore, a stratified water column with high salinity and anoxic bottom water conditions contributes to OM preservation in the lower oil shale layer. In contrast, high bioproductivity in combination with OM preservation, favoured by enhanced algae sizes and adsorption of OM on clay minerals, are suggested as the OM enrichment mechanisms in the upper oil shale layer. In addition, factors such as microbial activity, and terrigenous detrital matter input cannot be ignored for OM enrichment. In this study, a preservation model within the lower oil shale layer and a productivity model within the upper oil shale layer are established. The models imply that excellent preservation is the major controlling factor for OM enrichment in the lower oil shale layer, whereas the high bioproductivity is the major controlling factor for OM enrichment in the upper oil shale layer. However, the combination of both factors for oil shale depositions must be considered.

SILURIAN BLACK SHALES IN RUSSIA

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Key words:
shales, Silurian, Russia

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The Silurian interval is characterised by a wide distribution of oil-prone rocks. Together, they generate ~9% of the world's conventional petroleum reserves. These rocks are also an important source of unconventional oil and gas. However, despite their global importance there is still much basic information lacking in many parts of the world, nowhere more so than in Russia. As part of the CASP ongoing research on Silurian organic-rich source rocks, a database of Silurian black shales in Russia is being compiled from the literature with a complementary data set provided by original fieldwork and associated samples.

Silurian black graptolitic shales are well developed in Russia. They are recorded in the Kaliningrad area, the Caucasus, along the Urals Mountains including Vaigach and Pai-Khoi, Novaya Zemlya, Severnaya Zemlya, East Siberia including Taimyr, Altai-Sayan, New Siberian Islands, Kolyma and Chukotka. In some areas as much as 1500 m of black graptolitic shales are reported. In north-western parts of East Siberia, the source rock quality of the basal Silurian black shale is so good that it was mined and used as a fuel, since it generally burns without any processing. For all of the regions reviewed,

we present stratigraphic charts from Llandovery to Pridoli with information on sediment thickness, biostratigraphy and depositional settings.

Our field studies cover Severnaya Zemlya, Taimyr, northern East Siberia and Kotel'ny Island of the New Siberian islands, where the Silurian sequences contain black graptolitic shale and bituminous limestone intervals. Field and analytical studies have been undertaken to map the rocks, document their stratigraphy, identify the fossils, establish and describe facies, reconstruct the palaeogeography, determine palaeoenvironmental conditions, to obtain independent parameters on organic matter source, composition, thermal history and to investigate their possible oil-oil and oil-source correlation. Combined Rock-Eval and other geochemistry analyses on the Silurian samples from Severnaya Zemlya reveal the presence of early Silurian source rocks. The Llandovery Sredny Formation yields organically rich black shale with good source potential and a Type II oil and gas prone bulk source rock quality. The Rock-Eval T_{max} value suggests that the organic matter is early mature for oil generation.

CLAY MINERALOGY AND MAJOR ELEMENT GEOCHEMISTRY OF THE UPPER TRIASSIC TO LOWERMOST JURASSIC CONTINENTAL MUDROCKS IN POLAND – THE IMPLICATIONS FOR CLIMATE, PROVENANCE AND TECTONICS

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Key words:

clay minerals, major elements, palaeoclimate vs. provenance, upper Norian–lower Hettangian, Polish Basin

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The bulk-rock mineralogy, clay mineralogy and geochemistry of many continental mudrock samples collected from four borehole cores and few clay pits yield information on late Norian–early Hettangian palaeoclimatic and palaeoenvironmental variations in the Polish Basin. Changes in detrital clay mineral composition and in the major element contents were mostly controlled by weathering regime and climate. After semi-dry climate conditions in the Norian times (common domination of illite in the Zbąszynek Beds), smectite predominance in most part of early–middle Rhaetian Wielichowo Beds points to some increase in precipitation and its distinct seasonality. Subsequently, smectite preponderance was replaced by kaolinite and illite domination. This fundamental mineralogical shift (supported by major element data) indicates very significant change in climate humidity. In the late Rhaetian part of Zagaje Formation, the kaolinite mostly prevails and it signifies the predominance of warm climate with high round-year rainfall. However, the imprint of provenance is also observed. The clay fraction shows differences from one section to another. In Pomerania mudrocks, significant amounts of berthierine are observed, due to erosion of berthierine-containing

palaeosols and lateritic covers in the northern source areas. Moreover, the time span of the Rhaetian smectite enrichment is not uniform and suggests the local weathering of mafic rocks that overlaps with the climate record. In the early Hettangian part of Zagaje Formation, a generally stable illite-kaolinite-chlorite assemblage is observed, reflecting increasing delivery of less mature material, because of tectonic rejuvenation at the very beginning of Jurassic. Nevertheless, the relative abundance of kaolinite supported by palynofloral data suggests still significant hydrolysis that considerably masked tectonic signal. Importantly, some beds in the Rhaetian and in the lowermost Hettangian are particularly rich in kaolinite indicating extreme chemical weathering in the aftermath of super-greenhouse events that are correlated with C-isotope negative shifts and floral changes recorded in Poland and many sections worldwide. In addition, abrupt and episodic shifts in the kaolinite-illite ratio point to profound climate destabilisation and a sequence of frequent, catastrophic climatic reversals at the end Triassic and at Triassic–Jurassic boundary. These rapid reversals led to multi-phase extinction.

IMPORTANCE OF MUDROCK (SHALE) GEOLOGIC PARAMETERS TO IMPROVED PRODUCTIVITY ACROSS THE NORTHERN APPALACHIAN BASIN, USA



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Key words:

unconventional gas, liquids, mineralogy, fracability

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In the last ten years, tremendous progress has been made in the exploration and development of unconventional gas and liquids worldwide, especially in North America. The application of horizontal drilling and multi-stage hydrological fracture stimulation technologies provide economic gas flow in extremely low porosity and permeability reservoirs. Since 2005, the organic-rich shale units the Ordovician Point Pleasant-Utica and Middle Devonian Marcellus Shale of the northern Appalachia basin in the eastern United States of America have been one of the most active shale gas/oil plays in the world.

To date, successful mudrock plays have been primarily a function of drilling intensity, cost reductions and application of new technology (e.g., steerable rotary bits). Understanding of key reservoir engineering and geoscience parameters represents an important area that can contribute to improved well architecture and optimal lateral placement. Changes in mudrock reservoir properties that strongly influence productivity have been mapped across the Appalachian basin and define regions of higher and lower productivity. Important mu-

drock reservoir properties include: the distribution of organic content, the “fracability” of the unit (mineralogy and containment), structural discontinuities (faults), present and past stress regimes, and thermal maturity. In addition to dictating type of hydrocarbon fluid or gas present, thermal maturity has a critical influence on the development of the reservoir porosity and permeability system.

Established classification systems of pore size and structure are compared considering types of gas storage, flow and production of reservoirs. The goal is to understand pore structure in mudrock reservoirs in order to evaluate the reservoir and predict the storage capacity and productivity. In addition, techniques are proposed to predict detailed mineralogy and geomechanical properties using commonly available well-logs calibrated to core and/or advanced pulsed neutron spectroscopy logs. Migration of hydrocarbons from the matrix to the wellbore is investigated from a geologic perspective and can have significant impact on productivity in numerous mudrock reservoirs.

COMPLETE GEOMECHANICAL DESCRIPTION OF AN UGS CAPROCK FROM DRILL CORE MATERIAL: AN INTEGRATIVE LABORATORY STUDY



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Key words:

geomechanics, RACOS®, caprock, threshold pressure, 3D stress field

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A well constrained geomechanical model is the base for successful hydraulic fracturing of an unconventional reservoir, but also necessary for any safety prediction for underground gas storage. In this study, we present a complete geomechanical description of a marlstone, which serves as caprock for a pore-space store in south-eastern Europe. Our approach combines:

- brine permeability measurements and threshold pressure determination in a climate chamber under in situ conditions;
- measurement and calculation of the geomechanical characteristics of the caprock by rock strength testing (Brazilian tests, UCS testing, multi-stage compression and extension experiments);
- identification of the 3D rock anisotropy and in situ stresses from ultrasonic measurements on samples from the drill core with RACOS® analyses (Rock Anisotropy Characterization On Samples).

The core samples come from a grey marlstone taken at a vertical depth of about 2000 m and with a porosity of about 4% and a density of 2.6%. The brine permeability and threshold pressure measurements show that the marlstone is fairly tight to act as caprock.

The investigated rock material has a (for marlstone typical) low uniaxial strength. The axial compressive load-

carrying capacity at failure only increases slightly with increasing confining pressure. The strength analysis also indicates brittle behaviour at low confining pressure, which transforms relatively quickly into a ductile state.

From the measured direction-dependent wave velocities a horizontal bedding and N-S and E-W striking in situ structures are derived. Moreover, the spatial pore pressure effectiveness (Biot's coefficient in 3D) was determined, which is a significant factor for the evaluation of rock properties, as well as for the tensorial linking of effective and total stresses. Values of about 0.45 are relatively small as is typical of rocks with low porosity and permeability. The least component can also be an indication of the preferred flow direction. However, in this case only a very small spatial anisotropy was found.

The *in situ* stress state at the time of coring can be described as a normal faulting regime ($\sigma_v > \sigma_{hmax} > \sigma_{hmin}$). There are only small differences in the magnitudes of the horizontal stresses. The larger horizontal component (which is significant for the propagation of hydraulic fractures) is oriented NNE-SSW. The determined stress data correspond with data from the world stress map in this region.

TECTONIC DEFORMATION: GOOD OR BAD FOR SHALE GAS EXPLORATION? THE CASE OF THE SW IBERIAN VARISCAN CHAIN

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Key words:

shale gas, exploration, deformation, Iberian Variscan Belt, SW Portugal

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High volumes and low deformations are some of the peculiar features responsible for the increasing success of shale gas exploration activities, mainly in North America. In fact, the most important explored shale gas deposits (about 50% or more of the total gas), which are located at depths between 1 and 3 kilometres, show significant spatial continuity (of several tens of kilometres) and thickness (about 300 meters), display low angle to horizontal deep and are undisturbed in terms of fragile and ductile deformation. These exceptional conditions can promote relatively high exploration rates. However, they can only be found at wide continents with large cratonic regions, not affected by orogenies and/or if affected by important tectono-metamorphic events deformation, folding patterns show high amplitude and low frequency wavelengths.

In Iberia, the high deformation associated with the three main stages of the Variscan Orogeny, was used as an argument for not proceeding with prospection/exploratory projects of promising lithotypes. In fact, during the Variscan Orogeny, the Iberia was affected by several deformation stages, but there is no reason to exclude that some of the long flanks of the resulting folding patterns

may have important volumes of rocks bearing high quality indexes for the exploration of shale gas.

Recent studies developed in Portugal (SW Iberian Variscan Chain, South Portuguese Zone and Iberian Pyrite Belt) – based on the analysis of total organic carbon, maturation (both organic and inorganic) and gamma-ray data, revealed the occurrence of potential lithotypes and favourable conditions for the existence of shale gas. At the South Portuguese Zone (SPZ), it is possible to identify some units (Mértola, Mira and Brejeira Formations) which seem to be able to accommodate interesting volumes of gas, but they are associated to important deformation. In this case, the values of porosity and the microfracturation seem to be more important than the deformation style and consequent regional fracturation. Moreover, the thickness of those formations can be also very favourable to the existence of shale gas, as they frequently overpass one or two hundreds of meters, considered as appropriate for gas accumulation.

As so, the SPZ should not be excluded as a potential unconventional petroleum system, thus displaying great potential for shale gas exploration.

INTEGRATED PETROPHYSICAL PROPERTIES AND MULTI-SCALED SEM MICROSTRUCTURAL CHARACTERIZATION



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Key words:

petrophysical properties, microstructural characterization, organic content

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The microstructure and mineralogy of any system define its chemical and physical properties. The heterogeneity of shale exists across multiple scales with respect to these primary variables, making accurate characterization difficult. In addition, heterogeneity has made linking variance in measured physical properties to the observed microstructural characteristics equally problematic. Because of the nanoscale components of shale, scanning electron microscopy (SEM) has proven invaluable as a tool for understanding microstructural and physical properties such as storage, storage partitioning between organic and inorganic phases, and fluid flow dynamics of shale. However, until recently, the restricted field of view of the SEM has limited its utility in yielding representative analysis of these complex rocks. The emergence of automated high-resolution imaging and stitching software permits bridging 7 orders of magnitude of scale using a single instrument. The collection of large (~ 1 cm²)

images with nanometer-scale resolution is utilized to image and quantify microstructural characteristics from several different shale plays. Image analysis on the stitched mosaic provides quantitative measurements of key microstructural elements. These features include, organic content, organic and inorganic porosity, pore aspect ratio and size distributions among others. In addition, mineralogy will be quantified using FTIR as well as a new EDS-based approach for automated identification of the spatial mineralogy of each sample. The results of this detailed microstructural and mineralogical analysis are presented in the context of petrophysical measurements made on the same samples. Key petrophysical measurements include helium porosity, mercury injection capillary pressure, and NMR for porosity, TOC and pyrolysis for organics and organic maturity as well as seismic velocity and nanoindentation for defining mechanical properties of the samples.

MAGNETIC FABRIC IN SEDIMENTARY ROCKS OF ACCRETIONARY PRISMS AND ITS MODIFICATION BY PROGRESSIVE DUCTILE DEFORMATION

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Key words:

magnetic fabric, deposition, ductile deformation

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The magnetic fabric sensitively reflects depositional conditions of clastic sedimentary rocks, such as shales and sandstones, as well as progressive tectonic modification of their sedimentary fabric. Thanks to its efficiency and rapidity it can be easily applied to sediments of shale basins on regional scale. The depositional magnetic fabrics are observed in sediments of the Intracarpathian Palaeogene and Flysch Belt of the Western Carpathians and their modification due to progressive deformation can be illustrated on flysch-like sediments of the Rhenohercynian Zone of the Variscan orogen of the Bohemian Massif.

After deposition of sedimentary rock, the intensity of magnetic fabric is relatively low indicating relatively weak efficiency of deposition in orienting minerals, the magnetic fabric is predominantly planar, the magnetic foliation is approximately parallel to the bedding and the magnetic lineation is either parallel (in most sediments) or perpendicular (in lowermost A member of Bouma cycle) to the near-bottom water currents. During

vertical compaction, the intensity and the planarity of magnetic fabric increase, and the orientations of magnetic foliations and lineations remain. During bedding-parallel lateral compaction, the intensity and the planarity of magnetic fabric on the contrary decrease, the magnetic foliation may remain parallel to the bedding or may create an embryonic girdle in their poles, and the magnetic lineation reorients to become perpendicular to the shortening.

During continuing deformation when spaced cleavage develops, the magnetic fabric is on the transition between planar and linear, the magnetic foliation may remain parallel to the bedding and the magnetic lineation is parallel to the bedding/cleavage intersection lines. If the deformation is strong giving rise to flow cleavage, the fabric intensity is high, the magnetic fabric is again strongly planar, the magnetic lineations remain parallel to the bedding/cleavage intersection lines, but the magnetic foliations re-orient to the parallelism with the cleavage.

ARCHIVES OF DEPOSITIONAL AND DIAGENETIC EVENTS IN MUDROCKS, EXTRACTED USING MINERAL MAGNETIC AND PALAEOMAGNETIC METHODS

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Key words:

magnetic, palaeomagnetism, palaeocurrents, diagenesis

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Magnetic methods provide a range of tools which can be used for extracting, chronological, depositional and diagenetic events from shale and mudrock successions. This is because mudrocks contain small amounts of various magnetic minerals, both Fe-oxides, Fe-bearing silicates and sometime magnetic sulphides, which act as mineralogical archives of geologic events recorded in mudrocks. During mudrock deposition, the geomagnetic field can be recorded, along with depositional grain alignment, the former recording changes in geomagnetic behaviour and the later reflecting preferred grain alignment and palaeocurrent events in successions. These 'recordings' are carried by different magnetic particle populations, reflecting either Fe-oxide grain size differences, or the Fe-oxide versus Fe-silicate sources of the magnetic susceptibility (MS) – where the MS is a reflection of the abundance of all magnetic particle populations. The geomagnetic field is typically recorded by Fe-oxide particle populations.

We examine the diagenetic pathways that can give rise to the various magnetic particle populations and mineralogy's common in non-metamorphosed Palaeozoic mudrocks.

Anisotropy of magnetic susceptibility (AMS), which measures the magnetic grain alignment, is typically carried by Fe-silicates (various Fe-rich clays) in unmetamorphosed Palaeozoic sediments, and reflects the grain alignment and palaeoflow conditions in mudrocks. Examples of the use of AMS are illustrated. Also outlined in more detail are the geomagnetic field characteristics during the Ordovician and Silurian, based on existing and new data, which have clear implications for a better chronological sub-division of parts of the Palaeozoic. Lastly, we examine how the production of new mineral phases during diagenesis may help determine the age of the smectite to illite transition during diagenesis in mudrocks.

NEW INSIGHTS TO THE HYDROCARBON POTENTIAL OF EARLY PALAEOZOIC SHALES FROM POLAND AND CORRELATIONS TO POTENTIAL PALAEOZOIC SHALE PLAYS IN EUROPE AND THE USA



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Key words:

Silurian, Poland, hydrocarbon potential, optical kerogen analysis, organic maturation

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The early Palaeozoic shale belt of Poland has become a major target of unconventional shale gas exploration in Europe, leading to intense studies of these shales all along this zone. While in the north valuable plays are discovered, in several other places, particularly the SE, the initial expectations could not be proven by exploration results. This indicates significant regional heterogeneities within the shale belt, which is also typical for other European basins, considered as potential unconventional hydrocarbon plays. The significantly higher regional variations within European basins compared to the classical shale plays of North America are mainly caused by a smaller basin size, higher tectonical complexity and complex palaeogeographic settings. This shows the need for enhanced workflows for unconventional hydrocarbon system exploration in such complex basins.

A very promising approach for enhanced exploration and risk minimization is Optical Kerogen Analysis, based on optical analysis of the composition, preservation and maturation of the kerogen, combined with basin analysis. Kerogen composition quantifies each kerogen type separately, enhancing significantly the resolution and reliability of kerogen analysis compared to bulk-rock

geochemical analysis (Rock-Eval). Kerogen preservation gives information on hydrocarbon generation and on the gas storage capacity of the shales. Integrated optical organic maturation analysis provides highly reliable information on the maturity of the play.

For better understanding of regional differences in the Polish shale belt, samples from the early Palaeozoic of the Baltic- and Podlasie-Lublin Basin were studied, focused on Optical Kerogen Analysis. The aim was to quantify each kerogen type within each sample, the productive versus unproductive proportions of the total kerogen and the oil-prone versus gas-prone kerogen within the productive kerogen. Beside this, the gas storage capacity was analysed based on degradation (microporosity) of the organic matter just as the organic maturation. This is coupled with *in situ* analysis of micro-porosity and distribution of organic matter in shales by thin sections and SEM, also providing *in situ* analysis of rock texture, mineral composition and distribution. Results from Poland are correlated to organic rich Palaeozoic shales from Europe and USA, comparing the prospectivity of early Palaeozoic shales from Poland to other prospective Palaeozoic shale plays.

LOWER PALEOZOIC SHALES OF THE SW EDGE OF THE EAST EUROPEAN CRATON IN SE POLAND AND W UKRAINE, THEIR STRUCTURE AND TECTONIC EVOLUTION – REGIONAL COMPARISON



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Key words:

Lower Paleozoic, Poland, Ukraine, Teisseyre – Tornquist Zone

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The Lower Paleozoic basin of Poland has recently been the focus of intensive exploration for shale gas; its continuation in W Ukraine also attracted several companies that applied for exploration concessions. This sedimentary basin developed above the SW edge of the East European Craton (EEC) and could be regarded, at least in general regional framework, as a distal part of the Caledonian foredeep basin. The EEC edge in Poland and Ukraine is limited by the Teisseyre – Tornquist Zone (TTZ), crustal-scale fault zone characterized by a very complex tectonic structure formed during the entire Phanerozoic. In order to better understand Polish and Ukrainian counterparts of this sedimentary basin, an attempt towards comparative analysis of selected geological and geophysical data has been made. In Poland, analysis of relatively rich database of archive and recently acquired seismic data calibrated by numerous deep research and exploration wells and supported by analysis of gravity and magnetic data provided new information on regional structure of the cratonic edge including: Neoproterozoic extensional structures and associated volcano-sedimentary cover possibly related to the Orsha–Volhyn Aulacogen, deeply rooted strike-slip fault zones located at boundaries of the Malopolska gravity high, SW-NE –

trending system of mostly reverse faults related to the Bretonian (Early Carboniferous) reactivation of the Precambrian structures, thin-skinned Variscan (Late Carboniferous) compressional structures detached above top of the Precambrian basement or at the base of the Silurian shales, and regional reverse faulting related to the Late Cretaceous – Paleogene inversion of the MPT. In Ukraine, reprocessed archive seismic data calibrated by several deep wells was used to determine main structural elements of the cratonic edge. Identified structural elements in general conform to their counterparts known from Poland, main difference being Neogene structural overprint related to the emplacement of the Outer Carpathian thrust belt and formation of the Carpathian foredeep basin. Within the Paleozoic cover significant thickness variations across major fault zones have been mapped, possibly associated with syn-tectonic deposition. Additionally, major compressional structures (reverse faults, thrusts) have been identified that could be associated with the Variscan (Late Carboniferous) tectonic phase. Some amount of the Mesozoic and Neogene (Miocene) tectonic activity could have been also documented.

SALT TECTONICS WITHIN THE CENTRAL MID-POLISH TROUGH AND ITS CONTROL ON UNCONVENTIONAL PETROLEUM SYSTEM

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Key words:

Mid-Polish Trough, unconventional hydrocarbons, salt tectonics, Mesozoic

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The study area is located in central Poland, within the Kuiavian segment of the Mid-Polish Swell (MPS). The MPS was formed as a result of regional uplift and inversion of the Mid-Polish Trough: Permo-Mesozoic basin that evolved above the Teisseyre-Tornquist Zone. Following deposition of the Rotliegend siliciclastics and Zechstein evaporites, the Mid-Polish Trough was filled by several kilometers of the Triassic–Cretaceous sediments, mostly shallow water siliciclastics and carbonates. The Mid-Polish Trough was completely inverted in Late Cretaceous – Paleogene. Regional development of the Triassic–Cretaceous sedimentary cover was primarily governed by a regional subsidence related to deeper crustal processes such as basement faulting and thermal subsidence with secondary role of salt tectonics. Salt structures started to develop in Triassic times, salt movements have been at least partly triggered by regional basement faulting. In Late Triassic some of the salt pillows reached diapiric stage. After their further growth in Jurassic to Early Cretaceous times, salt structures were compressionally reactivated during Late Cretaceous–Paleogene inversion of the Mid-Polish Trough. Continuous growth of salt structures controlled Mesozoic depositional systems, with

thinner sedimentary cover characterized by generally shallower facies developed above these structures, and larger thickness and deeper facies located within the synclines between salt structures. Most complex salt structures are known from the Kuiavian segment of the Mid-Polish Trough, where the large Klodawa salt diapir is located. In this area, several targets for shale gas exploration have been identified: middle Jurassic mudstones, upper Jurassic mudstones and marls and Kimmeridgian fractured carbonate. Conventional reservoirs in Triassic through middle Jurassic sandstones offer attractive additional targets. Thermal modeling and Ro data indicate that the shales are in the oil window in the syncline adjacent to the Klodawa diapir. Core data indicate excellent quality reservoirs within 500 m thick Jurassic Dogger sand packages. Oil in open fractures has been frequently noted in the Tithonian carbonate (35–116 m thick) that lies directly above the organic rich Kimmeridgian shale. Seismic data calibrated by deep wells allowed to better constrain timing of growth of salt structures and, as consequence, to reconstruct evolution of the source rocks and better understand Mesozoic petroleum system.

STRUCTURE AND EVOLUTION OF THE LOWER PALEOZOIC BASIN DEVELOPED ABOVE THE EAST EUROPEAN CRATON IN POLAND: NEW INSIGHT FROM REGIONAL HIGH-EFFORT SEISMIC REFLECTION DATA

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Key words:

Lower Paleozoic, unconventional hydrocarbons, reflection seismic

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Recently the Lower Paleozoic basin located on the western edge of the East European Craton in Poland has become the focus of very intense exploration for unconventional hydrocarbons. Results of early exploration wells clearly demonstrate that there are still many unknowns regarding various aspects of the unconventional petroleum system, including structure and depositional architecture of the Lower Paleozoic succession. Seismic data from a recently acquired high-effort regional deep reflection survey, PolandSPAN project, has allowed for a better understanding of the complex tectono-sedimentary history of the prospective basins. The results of this regional seismic study will be reviewed and the impact on exploration objectives discussed. The Cambrian–Lower Ordovician succession, deposited on the Baltica passive margin, is covered by the Upper Ordovician–Silu-

rian succession of the Caledonide foredeep basin. Base of the foredeep succession is marked by “hot shale” interval. Large-scale seismically defined geometry of the foredeep infill reflects its progressive progradation towards the east-southeast. Late Paleozoic and Mesozoic tectonics have resulted in compartmentalization of the Lower Paleozoic basin into the Baltic, Podlasie and Lublin sub-basins. Tectonic deformations documented using new seismic data include Late Triassic thick-skinned normal faulting in the Baltic Basin, Late Devonian reverse/strike-slip faulting in the Podlasie and Lublin basins, and thick and thin-skinned Late Carboniferous thrusting and folding in the Lublin Basin. Further consideration and a better understanding of these complex geologic issues should benefit exploration efforts for unconventional hydrocarbons in Poland.

SHALE OIL AND GAS POTENTIAL IN THE LOWER PALAEOZOIC SUCCESSION IN THE CENTRAL PART OF THE BALTIC BASIN

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Key words:

Baltic sedimentary basin, shale oil and gas potential, the Lower Palaeozoic succession, reservoir properties of shales, maturity of organic matter

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The Baltic Basin is an intracratonic Phanerozoic sedimentary basin, situated on the western margin of the East European Craton. The unconventional hydrocarbon potential is related to the Early Palaeozoic organic-rich source rocks: the Cambrian (Alum shales and Middle Cambrian) in the south-eastern part, Ordovician Caradocian and Silurian Llandovery black shales that are considered as the major potential for shale oil/gas plays.

The analytic studies were performed on ~500 shale samples from 20 boreholes in SW Lithuania – reflectance of vitrinite-like macerals, Rock-Eval screening pyrolysis, total organic carbon content (TOC) have been determined. The organic matter of the source rocks is predominantly oil-producing type II and oil-gas liquids producing type II–III. Pyrolysis yields (32–76(~100) kg HC/ton rock) suggest a good hydrocarbon generation potential. TOC varies from 0.2–3 to 8–11% (up to 17%) and generally decreases up the section. New data show considerable variations of the maturity increasing southwestwards from 0.6–1.4(1.94)% (R_o). Locally, the anomalously high maturity of organic matter, indicating the lower part of the wet gas/condensate window have been recorded, probably being related to the locally-increased paleotemperatures.

Llandovery shales are clay-rich – values range 28–59 vol.% (mean – 47%); dominated by chlorite, illite and mica and illite-smectite. Quartz content ranges from 18–34 vol.% (mean – 30 vol.%), calcite and dolomite – 0.3–38 vol.% (mean – 10%); pyrite – 0.3–3.7 vol.%. Brittleness factor of 0.3–0.7 (mean – 0.46) suggests moderately brittle to moderately ductile rheological properties of shales. Interparticle pores dominate; interparticle pores in organic matter are rare. Total interconnected porosity – 1.4(5,4)–10.5%. Permeability varies in a range of 0.013–0.19 mD. Llandovery–Late Ordovician shales are the most prospective for unconventional hydrocarbons potential in the central part of the basin. New organic geochemistry data, incorporated with well log and core data were used to reveal the potential for shale oil/gas. Despite high amount of TOC, vitrinite reflectance data imply the thermal maturities for liquid, rather than gaseous unconventional hydrocarbons generation. The Volume of generated unconventional hydrocarbons in the central part of basin for the Late Ordovician–Early Silurian section of 30 m thick comprises: shale oil in place – 1.15–5.75 bill. m³; shale gas in place – 0.32–1.61 tril. m³.

MICROSTRUCTURE CHARACTERISTICS OF TIGHT SANDSTONE AND SHALE GAS RESERVOIR IN THE UPPER TRIASSIC SICHUAN BASIN, WESTERN CHINA

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Key words:

tight gas, microstructure, sandstone and shale reservoir, Nano-CT imaging

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The western Sichuan Basin is a foreland basin formed in the Late Triassic at the front of the Longmen Mountain in the western Sichuan Province of China. The Upper Triassic Xujiahe Formation in the basin is an ultralow-permeability and low-porosity tight sandstone and shale gas reservoir.

Tight gas reservoirs are often defined as gas-bearing sandstones or carbonates having *in situ* permeabilities to gas less than 0.1 mD (Holditch, 2006; Smith *et al.*, 2009). This paper offers an integrated approach to describe microstructure characteristics of tight sandstone and shale gas reservoir. In particular, the primary and secondary porosity of a tight gas sandstone are identified and quantified in three dimensions using X-ray Nano-CT imaging and visualization of core material at the pore scale.

3D images allow one to map in detail the pore and grain structure and interconnectivity of primary and secondary porosity. Once the tomographic images are combined with SEM images from a single plane within the cubic data set, the nature of the secondary porosity can be determined and quantified. In-situ mineral maps measured on the same polished plane are used to identify different microporous phases contributing to the secondary porosity. Once these data sets are combined, the contribution of individual clay minerals to the microporosity, pore connectivity, and petrophysical response can be determined. Insight into the producibility may also be gained. This illustrates the role 3D imaging technology can play in a comprehensive reservoir characterization program for tight gas.

SEQUENCE STRATIGRAPHIC FRAMEWORK OF THE LOWER PALEOZOIC SUCCESSION: IMPLICATIONS FOR UNCONVENTIONAL TARGETS



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Key words:

sequence stratigraphy, shale, hydrocarbons, unconventional, Paleozoic

19

Polish part of the East European Craton (EEC) remains the object of intense exploration for unconventional hydrocarbons. Based on the present data, the most promising area seems to be the Baltic sub-basin, but taking into account a number of drilled wells, the final definition of a hydrocarbon province is still open.

Lower Paleozoic organic-rich mudstone was deposited on the western margin of the EEC. Targets for unconventional exploration are defined as the Upper Cambrian – Piaśnica Fm (Alum shale analog), the Caradocian – Sasin Fm (Baltic sub-basin)/Udal Fm (Podlasie, Lublin sub-basin) and the Llandoveryan – Jantar Member. The vertical profile is characterized by thickening towards the west, reaching >3000 m Silurian deposits, e.g. Słupsk IG 1 well. High lithofacies diversity occurs in vertical profile and horizontal extent. The main goal of the study was sequence stratigraphy framework constructing.

Based on detailed lithofacies analysis (well cores), sequence stratigraphy framework was prepared. Well logs were useful only in the lower part of the profile (Cambrian–Llandovery). The majority of the profile in relation to the sequence stratigraphy analysis, because of subtle changes in rock properties, may be constructing mostly on the core samples. Characterization of bioturbation intensity and biogenic structures as well as grain size, diagenetic features, lamination and sedimentary structures define flooding surfaces – parasequences.

Lower part of the profile (Cambrian–Lower Silurian) was deposited during Passive Margin Stage (PMS) of the EEC basin, whereas upper part (Middle/Upper Silurian) during Flexural Bending Stage (FBS) of the EEC basin. Diachronism causes migration of the boundary between PMS and FBS in the vertical profile. Thickness of the 5th order parasequences in the PMS deposits is about 1X – 3XX cm, whereas in FBS from X00 cm to X000 cm. Deposition rate was about 1cm/12500 years and 1cm/435 years, respectively. Basin architecture during PMS was driven by eustasy. Sea level changes recorded in the upper part of the profile, related to the FBS, were mostly driven by tectonic and sediment supply, i.e. relative sea level changes. General stacking pattern is defined as aggradational to progradational in sequences from the 5th to 2nd order.

The main targets (Piaśnica Fm, Sasin/Udal Fm, Jantar M) are defined as TST deposits. Those targets are characterized by elevated TOC (up to 20%) and limited thickness – thin or very thin (pinching out to 0 m), about few meters on average. The upper part of the profile (Middle/Upper Silurian), because of organic matter dilution, was not considered as a target so far. Intervals related to the maximum flooding surfaces in this section (Middle/Upper Silurian) are characterized by elevated TOC, which in relation to the expedient mineralogy and reasonable thickness makes those targets promising.

CHARACTERISTICS OF CARBON DIOXIDE SORPTION IN COAL AND GAS SHALE – THE EFFECT OF PARTICLE SIZE

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Key words:

sorption, carbon dioxide, gas shales, coal

20

Gas retention mechanisms in shales are to some extent similar to that of coal. The gas is adsorbed in organic matter (mostly organic carbon) and clay minerals whereas transport of gas occurs in fractures. In coal, the gas is adsorbed in coal matrix and diffuses into fractures where it moves according to Darcy's law. It was assumed that fractured, depleted gas shale reservoirs in Poland could be potential places for CO₂ geological storage and contribute to the reduction of CO₂ atmospheric emissions. In the study two materials were analyzed – coal from the Upper Silesia Coal Basin and shale sample from Baltic Basin. The shale sample was characterized by a rather low TOC (0.12%) but high clay minerals content. The purpose of the study was to compare the high pressure CO₂ sorption characteristics of coal and gas shale and relate it to the particle size of samples subjected to tests. The par-

ticle size in every sorption related experiment must compromise the time for attaining sorption equilibrium, and reflect natural pore distribution and access to the nano- and micropores where sorption occurs. For that purpose both sorbents were crushed and sieved into three particle size fractions and the CO₂ sorption was measured. The measurements were performed at the constant temperature of 55°C and up to the pressure of 15–16 MPa. To model the sorption behavior, a three parameter Langmuir model was fitted to experimental values. Results of the sorption tests show that the particle size of the sorbent has an impact on the obtained sorption isotherm. In case of coal it could be related to the ash content which is higher in fine particle size (<0.1 mm), whereas in shale it might depend on the area of exposed surface.

UNDERSTANDING GEOPHYSICAL RESPONSES OF ORGANIC-RICH HEKKINGEN SHALES IN THE HAMMERFEST BASIN, THE NORWEGIAN BARENTS SEA

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Key words:

geophysics, petroleum exploration, Hekkingen Shales, Barents Sea

21

The Hammerfest Basin is the most important petroleum exploration area in the Norwegian Barents Sea. Several discoveries have been reported with almost all the accumulations being dominated by natural gas (e.g., Snøhvit, Askeladd, Albatross, Alka, Tornerose, etc.). Although the Hekkingen Formation is the main source rock in the area, the detail understanding of complex geophysical responses has not been investigated. This study focuses on understanding the complex geophysical responses of organic-rich Hekkingen shales in the Hammerfest Basin using 38 exploration wells drilled in the area. The organic-rich shales consist of brownish-grey to very dark grey shales and claystones with occasional thin interbeds of limestones, dolomites, siltstones and sandstones deposited in open to restricted shelf environments during late Oxfordian/early Kimmeridgian to Ryazanian time. The organic-rich shales are very heterogeneous in composition and rock physical properties at macroscopic to microscopic scales. The base is defined by the transition from carbonate cemented and pyritic mudstones to poorly consolidated shales, producing a sudden increase in gamma ray and interval transit time and an abrupt decrease in bulk density. To understand the geophysical responses, this study uses several rock physics relation-

ships, which are constrained with geology and formation-evaluation analysis, to calculate effective properties such as impedance, V_p/V_s , $\lambda\rho$ and $\mu\rho$. Results demonstrate that the geophysical responses are strongly influenced by mineralogy, TOC, porosity and spatial distributions of shales. Analysis also suggests that the diagenetic processes are quite different due to different exhumation histories in different parts of the basin adding further complexities to interpret the geophysical responses. Mineralogy (clay, calcite, silica, etc.) strongly affects the strength and/or fracability of the rock. Organic matters and their maturation have significant influence on resistivity, bulk density and sonic velocities, which suggests the necessity of proper handling TOC effects in the analysis of geophysical data (e.g., seismic and well log). The porosity-sensitivity of V_p at different porosity range is significantly influenced by TOC variations. The changes in bulk densities and effective velocities due to TOC and mineralogy also influence the AVO behavior. Due to significant lateral and vertical variability of rock physical properties, no universal relationship between TOC and mineral composition is expected for the organic-rich Hekkingen shales.

HYDROCARBON SOURCE ROCKS OF THE MARAGH LOW, EASTERN SIRT BASIN, LIBYA



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Key words:

geochemical analysis, pyrolysis results, hydrocarbon facies, Triassic Shale

22

Biostratigraphical analyses of well sections from the Maragh Low in the Eastern Sirt Basin have allowed high resolution correlations to be undertaken. Full integration of this data with available palaeoenvironmental, lithological, gravity, seismic, aeromagnetic, igneous, radiometric and wireline log information and a geochemical analysis of source rock quality and distribution has led to a more detailed understanding of the geological and the structural history of this area.

An assessment of pyrolysis results and a palynofacies analysis has allowed hydrocarbon source facies and quality to be determined. There are a number of hydrocarbon source rock horizons in the Maragh Low, these are sometimes vertically stacked and they are of fair to excellent quality.

The oldest identified source rock is the Triassic Shale. This unit is unconformably overlain by sandstones belonging to the Sarir Group and conformably overlies a Triassic Siltstone unit. Palynological dating of the Triassic Shale unit indicates a Middle Triassic, Anisian age. The Triassic Shale is interpreted to have been deposited in a lacustrine palaeoenvironment. Geochemical analysis of the Triassic Shale indicates total organic carbon varying between

1.37 and 3.53. S₂ pyrolysate yields vary between 2.15 mg/g and 6.61 mg/g and hydrogen indices vary between 156.91 and 278.91. The source quality of the Triassic Shale varies from being of fair to very good/rich. Linked to thermal maturity it is now a very good source for light oil and gas. It was once a very good to rich oil source.

High resolution biostratigraphical interpretations have been integrated and calibrated with thermal maturity determinations (Vitrinite Reflectance (%Ro), Spore Colour Index (SCI) and T_{max} (°C) and the determined present day geothermal gradient of 25°C/km for the Maragh Low. For the Triassic Shale the early phase of oil generation was in the Late Palaeocene/Early to Middle Eocene and the main phase of oil generation was in the Middle to Late Eocene. The Early Barremian Shale reached the main phase of oil generation in the Early Oligocene with late generation being reached in the Middle Miocene. For the Rakk Group section (Rachmat Formation, Tagrifet Limestone and Sirt Shale Formation) the early phase of oil generation started in the Late Eocene with the main phase of generation being between the Early Oligocene and the Early Miocene.

CATHODOLUMINESCENCE INVESTIGATIONS ON QUARTZ CEMENT IN THE SANDSTONES OF KHABOUR FORMATION FROM IRAQI KURDISTAN REGION, NORTHERN IRAQ

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Key words:

Paleozoic, Ordovician, Khabour Formation, Northern Iraq, quartz cement

23

The Ordovician deltaic to shallow marine Khabour Formation in Northern Iraq consists mainly of sandstone with minor siltstone and interbedded shale. The sandstones are pervasively cemented by quartz that resulted in very little preserved primary porosity. Cathodoluminescence and petrographic studies showed that the silica cementation occurred in five successive phases which can be distinguished by their luminescence pattern. The precipitations of two phases have predated the major compaction process while the other phases are younger. The successive phases represent a sequence of changes

in silica supply which were classified as very early and early, derived from dissolved biogenic silica that precipitated as opal/microquartz, possibly pre-compactional and of non-luminescent quartz overgrowth type. This was followed by phases whose silica supply derived from pressure solution of quartz, dissolution of feldspar, and hydrothermal fluids related to major thrust fault event. These successive quartz cement phases showed an increase in luminescence and the development of complicated zonation pattern in late-stage quartz cementation.

EARLY JURASSIC SUPER-GREENHOUSE EVENT IN ATMOSPHERIC/TERRESTRIAL SYSTEM – A UNIQUE RECORD FROM TOARCIAN MARGINAL-MARINE SHALES IN POLAND

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Key words:

shales, organic matter, climate, decomposition

24

The climatic impact on vegetation and the atmosphere system in extant world is characterized by complex patterns and processes that act at various time and space scales. Much less is known about similar processes which took place in deep geological past. The formation of black mudstone, rich in total organic carbon (TOC) is associated with Oceanic Anoxic Events (OAEs). One of the most severe of these events is the Toarcian OAE (Early Toarcian, approx. 183 Ma ago). The T-OAE is associated with a high rate of organic burial, coincident with a prominent negative carbon isotope excursion (CIE), and extinction of many groups of marine organisms. In the Polish basin, strata coeval to marine black shales of the T-OAE are represented by poorly consolidated green/grey mudstones with sandstone intercalations (Ciechocinek Formation) deposited in a large embayment/lagoon. The organic matter in all the samples studied is strongly dominated by kerogen type III of terrestrial origin, showing very low thermal maturity. Carbon-isotope data from the woody organic matter in Early Toarcian successions of the Polish Basin (phytoclast separates) show a negative CIE, which occurred in major steps, reproducing observations on CIE in marine successions, where they have been attributed to astronomical forcing of climate. While a perfect

correlation between CIE steps in marine and marginal-marine/non-marine settings can be drawn, trends in TOC through the Early Toarcian of marine and marginal marine successions diverge strongly. Geochemical analyses contradict anoxic conditions in the whole Lower Toarcian in Poland. Palynodiagrams (palynomorph composition) of selected borehole sections reflect the composition of standing vegetation and show the plants' response to the Early Toarcian environmental perturbation. Also clay mineral composition (kaolinite-illite ratio, reflecting climate and weathering conditions) follows – although not so exactly, the palynomorph composition. Also palynofacies reflect climate changes. Each CIE cycle during the T-OAE time was characterized by increasingly severe palaeoclimatic conditions, culminating in extremely hot and humid conditions. Low TOC content, strongly correlated with most negative C-isotope values, was caused by enhanced decomposition of organic matter, associated with high temperature.

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GEOLOGICAL SETTING AND RESERVOIR HETEROGENEITY OF LOWER PALEOZOIC SHALES IN LUBLIN BASIN (POLAND) AND ITS IMPACT ON EXPLORATION



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Key words:

Lublin Basin, Lower Paleozoic, shale gas, unconventional play

25

Lower Paleozoic, Ordovician and Silurian shale succession represent key, prospective horizons in the Lublin basin, defined as unconventional resources.

For almost three years E&P companies have conducted research to evaluate HC potential, estimate resources and confirm reserves.

During this time, in the area of nearly 20,000 km², which covers the Lublin Basin and partly Podlasie Basin, 22 new boreholes were drilled, 10 of which by ORLEN Upstream (7 vertical and 3 horizontal wells). Almost 1,800 m of core samples were recovered from the wells when performing coring operations and brought to the surface for study and detailed analysis. In addition to the drilling operations, ORLEN Upstream has conducted about 1,250 km of new 2D seismic surveys, the reprocessing and interpretation of more than 5,000 km of 2D seismic profiles and also more than 150 km² of 3D seismic data.

Most relevant was to understand the structural evolution of the Lower Paleozoic succession, detailed definition of the main reservoir parameters of Ordovician and Silurian shales and their extension.

The analysis of parameters variability was performed (heterogeneity rock analysis). It involved wellbore, inter-well and fieldwide scale heterogeneities and used cores, logs, seismic, structural and facies analyzes.

The preliminary results from ten wells drilled by ORLEN Upstream in the Lublin Basin confirmed the general known trends of shale succession and high tectonic complexity within the research area. However, a noticeable variation of main reservoir and geochemical parameters and geomechanical properties of rocks occurred both along and across the Lublin Basin.

There is a notable correlation between the level of transformation of organic matter and the depth of the horizons, no evidence of reservoir overpressure, and a relatively high level of anisotropy of the stress.

The amount of drilled wells and production tests is for now insufficient to determine the relationship between the results obtained during the analysis and for estimation of the productive potential of Lower Paleozoic shale succession.

THE LOWER PALAEOZOIC SHALE GAS PLAY IN SOUTHERN SCANDINAVIAN: ROCKS, RISKS AND RESOURCES

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Key words:

Alum Shale, facies, play evaluation, resource assessment

26

In Denmark, shale gas exploration targets Lower Palaeozoic shales and two licences have been awarded to Total E&P in northern Jutland and northern Zealand, respectively. The first Danish shale gas exploration well is scheduled to be drilled in northern Jutland Winter 2014, targeting deeply buried gas-mature shale, notably the so-called Alum Shale which is unusually rich in organic matter, typically 5–10% and locally up to 25%. The formation is up to 180 m thick in offshore areas of Denmark.

GEUS and the University of Copenhagen have since 2009 conducted a wide range of shale gas evaluation programmes including shallow coring in areas where the shales are accessible immediately beneath a thin Quaternary cover. Key questions addressed include the facies types, gas storage capacity, mineralogy, and how the rock responds to hydraulic fracturing.

The prospective areas for shale gas in Denmark are the rims of the Norwegian-Danish Basin where the Palaeozoic shales are buried between 2 and 4 km. The Palaeozoic shales were matured to gas stage within a Caledonian foreland basin in Late Silurian time. In the Carboniferous and Early Permian the shales were faulted and the fault blocks were tilted and uplifted and locally

subjected to intensive erosion. Because of the complicated burial history it is currently uncertain whether significant amounts of gas are still trapped in the shales. The gas may have leaked out through millions of years of uplift and progressive erosion since the gas formed more than 400 million years ago. A study conducted by Shell on the Alum Shale in southern Sweden indicated that Alum Shale now located at 700–800 m depth did not contain gas in economical quantities and gas retention in the shale poses a major technical risk for the play.

Working with GEUS, the U.S. Geological Survey (USGS) recently assessed the gas resources in the Lower Palaeozoic shales of Denmark. Assuming best practice technology, recoverable resources of 0 to 4.8 TCFG were estimated onshore (mean = 2.5 TCFG) and 0 to 8.5 TCFG were estimated offshore (mean = 4.4 TCFG), i.e. a total estimated mean of 6.9 TCFG. The wide range of estimates reflects the sparse data and geological uncertainty inherent in this untested play. While significant, this estimate stands in stark contrast to the 2013 EIA/ARI estimate of 31.7 TCF. The USGS estimates probably reflect a recovery efficiency of about ten percent of original gas in place.

SHALE RESERVOIRS AND IP WORKFLOWS



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Key words:

shale, IP, senergy, reservoir, workflow

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The presentation makes an introduction to Shales Reservoirs and Petrophysics workflows, bringing up in detail the Clay Volume calculations vs the Shale Volume calculations. The Total Organic Content (TOC) and Kerogen concepts are explained, all culminating with the advantages of utilising a non-deterministic, error-minimising solver.

Audience benefits:

- Shale Gas Reservoirs characterisation.
- Clay Volume vs Shale Volume.

- With simple triple/quad combo data a deterministic workflow can be used to provide a robust determination of TOC, porosity and gas saturation.
- More detailed and complex non-deterministic models can be developed (such as IP Mineral Solver) utilising the TOC and heavy mineral volume from deterministic models.

ELECTRICAL AND ELECTROMAGNETIC LOG DATA INTERPRETATION IN SHALE AND ARGILLITE BEDS OF WEST SIBERIA

.....
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Key words:
dielectric permittivity, electromagnetic logging

28

This paper analyzes some results of the numerical interpretation of Russian lateral logging (BKZ, direct current arrays) data and high-frequency electromagnetic logging data measured in shale and argillite beds.

Frequencies of an exciting electromagnetic field are chosen in the range from 875 kHz for a 2 m long probe to 14 MHz for a 0.5 m long probe. BKZ array lengths are 0.4, 1, 2, 4, 8 m. A sounding curve (combination of all probes signals at one depth point) demonstrates resistivity distribution from borehole to the uninvaded formation.

As has been observed, every so often the estimates of electrical resistivity from electromagnetic sounding data and the estimates from direct current sounding disagree with each other. The signals measured by various techniques were compared at intervals of thick homogeneous argillaceous deposits in marker beds. Signals from electromagnetic probes in such formations tend to be consistent with model containing low resistance invasion zone, whereas BKZ signals are attributed to models either with missing invasion zone, or with narrow high-resistivity zone. The said apparent contradiction is eliminated by introduction of dielectric permittivity allowing to exclude invasion zone from electromagnetic data interpretation in the intervals with impermeable rocks.

In the course of inversion a certain effective permittivity value was determined. Consequently, with such an approach employed, estimate is often higher than values for each individual component composing rocks. Different researchers suggest different estimates for relative permittivity as high as several hundreds and thousands of relative units, based on the results of samples investigation (sandstone, loams, clay; Talalov, Daev, 1996), and of LWD data interpretation (frequencies 0.4 and 2 MHz, pyritized shales; Anderson *et al.*, 2007), with permittivity frequency dispersion also being observed therewith.

According to electromagnetic logs, frequency dispersion of permittivity in some clayey formations is determined when the signal measured at each operating frequency corresponds to its own permittivity value. Estimates of permittivity vs. frequency, obtained in several West-Siberian shale or argillite formations, are consistent with the values obtained on shale samples, and take a values 30–50 relative units vs. 14 MHz, 100–500 vs. 3.5 MHz, 300–1000 vs. 875 kHz. The estimated permittivity values differ for different argillaceous layers of bazhen oil-generating formation.

MUDSTONE STRATIGRAPHIC LOCATION AS A CONTROL ON FLUID PATHWAYS IN A FLUVIAL SEQUENCE



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Key words:

fluid pathways, mudstones, fluvial sandstones, hematite, whitening

29

Mudstones within the Pennsylvanian-Permian Fountain sandstone, a dominantly fluvial sequence exposed in northern Colorado, appear to have served as baffles that controlled the specific pathways of fluid migrating through the sandstone beds. Paleofluid flow included at least one episode of hydrocarbon migration, an episode that appears to be responsible for stripping hematite from the sandstones, transforming the fluid flow pathways from red to white. The white fluid pathways, which account for about 15% of the total rock volume, are also distinguished from the remaining red sandstone by the presence of fluorescent cements and greater alteration of feldspars.

The repetitious fining-upward fluvial sequences exposed in the study area are mainly coarse- to fine-grained sandstones capped by thin paleosol mudstones. Coarse-grained channel fill sandstone beds at the base of a se-

quence are somewhat more likely to be whitened than fine-grained sandstone beds, but the whitened flowpaths preferentially hug both the upper and lower surfaces of the mudstones regardless of sandstone grain size. Sandstone beds that appear equally likely to have transmitted fluids are more likely to be red if separated from the mudstones and white if in contact with the mudstones. Individual sandstone beds in contact with the mudstones show greater whitening immediately adjacent to the mudstones, than they do a few tens of cm removed from the mudstones.

The association observed in the Fountain Formation between tightly focused fluid pathways and the boundaries of low-permeability mudstones suggests that low permeability mudstone beds may affect the specific fluid pathways through more permeable parts of sedimentary successions more generally.

ILLITE + SMECTITE AS COMPONENTS OF SHALES

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Key words:

CEC, diagenesis, illite-smectite, K-Ar dating, maximum paleotemperatures

30

Illite+smectite accounts for 30–50 wt.% of shale mineral composition and for ca. 1/3 of the mass of all sedimentary rocks. Both due to this large quantity and unique properties illite+smectite control numerous properties of the bulk shale.

Smectite is a low-temperature mineral: the product of weathering and low-temperature diagenesis. In shales it can be then both detrital and authigenic. In surface temperatures and close to neutral pH it is very stable: known from the Ediacaran.

Illite may also form at surface temperatures in rare environments of elevated pH, but it is the dominant product of typical silicate rock alteration at elevated temperatures. Illite survives temperatures up to 300°C, when it recrystallizes into coarse-grained mica, and is also quite resistant to weathering. Thus, in shales illite most often is both detrital and authigenic. If the substrate of illitization is smectite, mixed-layer illite-smectite minerals appear, and their composition evolves gradually towards end-member illite with increasing temperature. This process terminates when the maximum paleotemperatures are reached. An admixture of illite-smectite is present in almost every shale sample, and the layer ratio can be quantified by XRD, offering the most universal proxy for the maximum paleotemperatures.

All clay crystals are plates, with high aspect ratio. Smectite and thin illite crystals are flexible and they have electric charge on the surfaces, which makes them form quasi-parallel aggregates. This property creates microporosity and anisotropy of the shale fabric, the properties evolving with the increasing overburden.

Smectitic interlayers are the only components of common shales with high surfaces, which are charged, thus capable of exchanging cations, adsorbing water, and organic molecules. Thus, illite+smectite control electric properties of shales (their CEC) and shale behavior on contact with fluids.

A great majority of shale boron is contained in illite layers. This property offers an opportunity of quantifying illite+smectite component of shales via borehole geophysics (Σ_{cl}).

In common shales a few to more than 50% of potassium in illite is substituted by ammonium. The level of substitution is controlled by the supply of ammonium. The ammonium fixation in shale illite is the key process of the global nitrogen cycle.

Authigenic illite is the only common K-bearing diagenetic product, thus containing information about the age of the maximum paleotemperatures (K-Ar).

UPPER ORDOVICIAN AND SILURIAN MUDROCK FACIES FROM THE HOLY CROSS MOUNTAINS AND THEIR RELATION TO PALAEOZOIC SEA-LEVEL AND CLIMATE

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Key words:

Ordovician, Silurian, black shales, mudstones, sea-level changes, oceanic circulation

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Upper Ordovician and Silurian mudrocks in the Holy Cross Mountains (HCM) form a relatively thick succession (200–350 m) deposited in the Caledonian foredeep basin developed along the SW margin of Baltica. They consist of grey/green claystones and mudstones with recurrent black shales reflecting major changes of paleoceanographic conditions. It is widely accepted that black shales are prominent horizons recording significant sediment starvation, oxygen deficiency and organic matter preservation during transgressive events. However, sedimentological and geochemical data clearly show that oxygen deficiency during deposition of Ordovician and Silurian black shales in the HCM was intermitted by seasonal increase of benthic oxygen level.

The Upper Ordovician mudrock facies in the HCM reflect an overall change of redox conditions triggered by the major overturn in oceanic circulation consistent with shift from greenhouse to icehouse climate (Trotter *et al.*, 2008). Two dark shale horizons within the *gracilis* and *clingani* graptolite zones are accordingly coeval to the early Sandbian and early Katian transgressions. The latest Ordovician glacio-eustatic regressive event in sedimentary record of the HCM is recorded by a rapid increase of coarse-grained clastic material yielding the upper Cambrian to Middle Ordovician acritarchs within the Hirnantian palynomorphs.

The Silurian mudrock succession in the HCM shows profound variation in the benthic oxygen level reflecting changes of accommodation space driven by eustatic fluctuations as well as delivery of fine-grained clastic and carbonate material. The graptolite biostratigraphy shows that transgressive black shales in the Silurian succession are restricted to the *acuminatus*, *triangulatus?*, *convolutus*, *turriculatus*, *crenulata*, *spiralis*, *centrifugus/murchisoni* and *dubius?* zones. However, the predominating lithofacies are grey or grey/green mudstones indicating deep-water ventilation and permanent benthic oxygenation coeval to regressions on the Silurian sea-level curve (Page *et al.*, 2007).

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UNCONVENTIONAL HYDROCARBON RESOURCES OF GREECE



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Key words:

shale gas, methane hydrates, coal bed gas, Greece

32

Greece has important oil and gas reservoirs and a significant potential of unconventional hydrocarbon resources. However, the probable and proven reserves of these hydrocarbons are unknown as long, as the detailed investigations are lacking. Nowadays, the intensive exploration of probable conventional hydrocarbon reservoirs, through the interpretation of seismic profiles and the abundant surface geologic data, will provide the subsurface geometric characteristics of the unconventional ones. The most important Cretaceous and Paleogene formations which may contain shale gas are found in the basins of Orestias-Alexandroupolis, Kavala-Prinos, Axios-Thermaikos, Grevena, and Island Limnos. In addition, Western Greece (Ionian and pre-Apulian zones) contains significant immature, or close to the early maturation stage, source rocks with black shale composition. The subsurface geometry of these fine-grained layers is unknown

and may be revealed only by detailed seismic exploration. Methane hydrates have been detected in the Eastern Mediterranean, in the submarine Anaximander Mountains east of Rhodes Island. The region has about 250 million m³ hydrates containing 96.5% methane and 3% ethane. Exploiting the hydrates with the current technology is economically unprofitable. Greece, with approximately 43 active coalfields, holds the second position in the EU and 11th worldwide in the production of coal (especially lignite). However, its coal bed methane potential is insignificant. The low content in gaseous hydrocarbons and the extensive tectonics in the whole Hellenic Peninsula are the main factors which prevented such accumulations. The indicated reserves of conventional gas in Greece are approximately 3.5 trillion m³, while the reserves of unconventional hydrocarbons are unknown.

INFLUENCE OF SEDIMENTOLOGICAL VARIATION ON RESERVOIR AND SOURCE ROCK CHARACTERISTICS IN SHALE DOMINATED CYCLOTHEMS (CAMPINE BASIN, NE BELGIUM)

.....

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Key words:

Campine Basin, cyclothems, source rock, reservoir, mineralogy

33

The KB186 well (GSB 047W0264, Lommel-Kerkhoven) is situated in the northern part of the Campine Basin (NE Belgium) to the west of the Donderslag fault and has been studied and sampled in detail from a depth of 1182.72 meters to 1197.75 meters. The objective is to examine the sedimentological, petrographical, mineralogical and petrophysical characteristics and their variations within and between two cyclothems. 17 shale samples were taken throughout the entire section, varying from roof shales, over dark organic-rich shales with siderite enrichments, to bluish-grey shales without no visible organic content.

Two sedimentary cycles have been recognised. Macroscopically, they show some similarities and differences in facies and sedimentological trends. Both are approximately 6.5 meters thick and contain sections of mainly shale deposits (with or without siderite enrichments). Fining-upwards sequences of fine sand and clayey silt and larger sandstone bodies occur between these shale-rich sections.

A petrographical assessment by means of optical light and electron microscopy is conducted for a qualitative analysis of the mineralogical composition and diagenetic

history, while a quantitative determination is made by means of X-ray diffractometry. Important mineralogical reservoir parameters are the relative amounts of silica and quartz, and the clay mineralogy with special attention to smectite and illite.

Variations within and between the sedimentary cycles in reservoir and source rock characteristics are further examined by means of geochemical and petrophysical techniques. The Rock-Eval method yields information on source rock quality, such as hydrocarbon generation potential and thermal maturity. The reservoir potential of the deposit depends also on the amount of natural gas that can be stored in the rock as free gas in the pore spaces or as sorbed gas on the surface of clay and organic particles. Low-pressure sorption of CO₂ and N₂, helium pycnometry and mercury injection porosimetry (MIP) provide detailed information on pore volume and pore structure. High-pressure methane sorption isotherms are measured to assess the sorption capacity.

We will demonstrate to which degree reservoir properties relate to sedimentological and/or diagenetic characteristics, a relation that when extrapolated can be used to make basin scale projections.

LONG-TERM EXPERIMENTS TO EVALUATE INTERACTIONS BETWEEN GAS SHALES AND STIMULATION FLUID DURING UNCONVENTIONAL GAS PRODUCTION



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Key words:

flowback, composition, stimulation fluid, Posidonia, Alum

34

The release of organic and inorganic compounds from 2 different black shales into a lab-made stimulation fluid was studied over time to provide an understanding of the main factors influencing the composition of flowback water to give first indications for water treatment and remediation solutions. Two long-term experiments at 100°C/100 bar were performed with samples from Alum and Posidonia shale lasting 6 and 2 months, respectively, using a stimulation fluid containing commercially-available biocide, surfactant, friction reducer and clay stabilizer.

Our results show that the amount of dissolved constituents at the end of the experiment is independent of the pH of the stimulation fluid but highly dependent on the composition of the black shale and the buffering capacity of specific components, namely pyrite and carbonates. The amount of elements released into the fluid is also dependent on the residence time in as much as half of the measured 23 elements show highest concentrations within four days.

We clearly demonstrate that the composition of the flowback water is effected by the natural organic matter composition and maturity of the shale and not just by the selected chemicals in the stimulation fluid. Alum and Posidonia shale extracts tend to have their specific com-

position of DOC. These patterns in DOC composition are also released in presence of DOC-rich stimulation fluids. Due to this observation it can be assumed that even with the application of DOC-rich stimulation fluids, the resulting flowback waters tend to have very shale-specific compositions of DOC and these compositions also may change over time. The compositions of the stimulating fluids have the potential to change the mobilization of organic compounds from shales. The high yields of formate from Posidonia samples differ from earlier studies evaluating the potential of shales to release formate as being low (Olsson *et al.*, 2013). It is not clear whether the formate released from the natural organic matter of the shale may have been enhanced by the chemical additives or whether the organic polymers within the stimulation fluids may have been the subject of decarboxylation and generation of formate at the environmental conditions of the shale formations. These results may also complicate the application of formate and acetate concentration as tracers for stimulation fluids.

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FRACTURE PROPAGATION DURING HYDRAULIC STIMULATION IN AN ANISOTROPIC STRESS FIELD APPLYING THE DISCRETE ELEMENT METHOD

.....

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Key words:

hydraulic stimulation, fracture propagation, anisotropic stress field, DEM, 3D model

35

Key to a successful exploitation of unconventional reservoirs is the hydraulic stimulation of the host rock to increase permeability. The current study investigates the fracture propagation induced by hydraulic stimulation in a highly anisotropic stress field, where the major principal stress σ_1 is approximately two times the minor principal stress σ_3 . The hydraulic stimulation is simulated applying 3DEC (Itasca™), a software tool based on the discrete element method. The three-dimensional 3DEC models consist of blocks and the contacts between those blocks. Latter provide possible pathways for fracture propagation. Both blocks and contacts are calibrated to exhibit host rock analogue properties.

The fracturing fluid is injected into “predefined” fracture planes, which are oriented perpendicular to σ_3 . After

the stimulation of the first fracture, a significantly altered stress field can be observed in the vicinity of that fracture. Especially the increase of σ_3 affects the subsequent hydraulic stimulation process. The fracture interaction due to the altered stress field is investigated by modifying (1) the pauses between stages, (2) the spacing's between adjacent stages, and (3) the stage alignment.

The simulation results indicate that fractures tend to propagate in random directions away from the stress shadows of existing fractures, even for a stage spacing of 200 m. This fracture deflection may result in undesirable fracture propagation directions or produce shortcuts between fracture stages. A careful planning of the stage alignment seems the most promising procedure to counteract/control fracture deflection.

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GENETIC STUDY OF THE GRAIN SIZE DISTRIBUTION OF THE SEDIMENTARY FILLING OF THE CLAY PAN OF MHABEUL, SOUTHEAST TUNISIA

.....

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Key words:

lay pan, genetic approach, diagram, climatic phases, Sebkha Mhabeul, southeast Tunisia

1

A 93 cm core was carried out at the clay pan of Mhabeul southeast Tunisia. Sampling was each 0.5 cm. Samples underwent the grain size analysis through FRITSCH Laser granulometer. The mean of the grain size distribution shows that the sedimentary filling of Sebkha Mhabeul is fine. On the one hand, the genetic approach shows the interplay of eolian and hydraulic fraction due to alternation of arid and wet phases. On the other hand, the plot of the grain size parameters such as skewness

and standard deviation along well-known diagrams (e.g., $SK = f(\sigma)$ and Passega diagram) confirms this interplay of climatic phases. The fine sedimentary filling of Tunisian clay pans is homogenous in such a way that we have a difficulty to distinguish between samples. Nonetheless, the high technology of FRITSCH on the one hand and the use of the appropriate methods and diagrams on the other hand allow distinction between similar samples and finding out of different climatic stages.

THERMAL EVALUATION OF LOWER PALEOZOIC SEDIMENTARY SUCCESSIONS BY NEW MULTI-METHOD APPROACH: THE CASE HISTORY OF POLISH BALTIC BASIN

.....

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Key words:

thermal evolution, Lower Paleozoic, Polish Baltic Basin

2

Sometimes, the evaluation of thermal maximum exposure by means of traditional indicators may be insufficient to define thermal maturity levels (especially in sedimentary deposits, as Lower Paleozoic succession) that are devoid of vitrinite-huminite group macerals. We propose a successfully adopted multi-method approach to assess thermal maturity, applied to Lower Paleozoic sections from three deep wells, recently drilled in the Polish Baltic Basin by Eni.

This strategy consists of: (i) measurement of organoclasts reflectance; (ii) FT-IR; (iii) Raman spectroscopic analysis; (iv) pyrolysis on dispersed organic matter; and (v) X-ray diffraction on fine grained sedimentary rocks. This approach guaranteed a robust cross check among thermal indicators as an input to burial and thermal modeling. We have correlated common adopted parameters

(eg., vitrinite reflectance, T_{max} etc) to selected indexes from spectroscopic analyses (both Raman and FT-IR), sensitive to temperature increase. This integration among optical and spectroscopic analyses, in the analyzed successions, provide a well constrained assessment of thermal maturity that ranges from mid-late mature to over-mature. Furthermore, in agreement with results obtained from organic matter analyses, X-ray diffraction identifies the late diagenetic zone.

In conclusion, this multi-method approach provided very encouraging results that make it a powerful tool for the assessment of thermal maturity of problematic organic facies. This could drastically reduce uncertainties concerning thermal modeling and positively influence decisions on the development of prospects, especially when aimed at exploring shale gas targets.

TEXTURES AND SECONDARY FEATURES OF ORE MINERALS IN THE CENTRAL PART OF SIEROSZOWICE MINING DISTRICT



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Keywords:

Kupferschiefer, Zechstein, iron and base metal sulphides, ore mineral textures, Sieroszowice

3

The Kupferschiefer is a black shale, representing the lowermost unit of the marine Upper Permian Zechstein Group of the Central European Basin. This paper presents petrographic characteristics of ore minerals, as well as their vertical distribution in the Kupferschiefer profiles. Samples for the study come from archival boreholes located in central part of Sieroszowice mining district. This area is characterized by the widespread presence of shales, where the Kupferschiefer is underlying the Zechstein Limestone, and overlying the Weissliegend sandstones. Generally, the Kupferschiefer in the studied area is about half meter thick and consists of alternating carbonate and clay-organic microlaminae, locally with calcite grains and terrigenous quartz. Sulphide grains and aggregates are dispersed throughout organic and carbonate material and arranged semi-conformably with lamination. The most abundant ore minerals in analyzed profiles are: chalcocite, digenite, covellite, bornite and chalcopyrite accompanied by galena, sphalerite and pyrite. Sporadically, minor arsenic sulfosalt, cobaltite, gersdorffite, stromeyerite and bismuth are identified. Microscopic examination revealed many ore mineral textures: disseminations, replacements, streaks, impregnations,

lenses and veinlets. Most ore minerals are xenomorphic, however subordinate Ni-Co minerals display idiomorphic textures. Pyrite commonly forms idiomorphic crystals and framboidal grains. Rare framboids of chalcocite and bornite also occur. The Kupferschiefer mineralization within the discussed region is zoned vertically in successive ore layers, which are enriched in: a) Cu-S type sulphides, b) Cu-Fe-S type sulphides and, c) pyrite and marcasite with minor galena and sphalerite; from the bottom to the top of profiles, respectively. Iron sulphides are almost absent in ore-volume copper concentration, but they predominate Cu-sulphides beyond copper-bearing zone. Ore mineral textures and microprobe analysis imply successive transformation of pre-ore mineralization dominated by pyrite-marcasite phases into high-grade copper mineralization, indicating that syndiagenetic iron sulphides have been replaced by later base metal sulphides. Replacements of iron sulphides and distribution of Cu-sulphides in a form of layers that cut across the lamination indicate that the base metal sulphides were formed as a result of flow of ascending metal-bearing solutions into reduced sediments.

BASIN ANALYSIS OF POTENTIALLY GAS-BEARING SHALES OF LOWER CARBONIFEROUS SUCCESSION IN THE AREA OF FORE-SUDETIC MONOCLINE

.....

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Key words:

Silesian Basin, Lower Carboniferous, flysch, hydrocarbon potential, shale gas

4

The Silesian Basin of Poland constitutes a part of the foreland basins system of the Variscan orogen. The Silesian Basin comprises Lower Carboniferous sediments which were analyzed as potential unconventional hydrocarbons source rocks.

The data used in the research had been collected during sedimentological profiling of archival cores. The western part of studied area comprises rocks which were described as proximal flysch facies. Facies distalization was observed in the eastern direction of the study area. Distal facies are represented by black shales intercalated with very fine-grained sandstones. In the south-eastern direction a transition of flysch facies into carbonate facies was observed.

The analyzed sediments are represented by arcose wackes, arcose-lithic wackes, silty mudstones and mudstones. There is an increase in mineral maturity of the Lower Carboniferous succession, what was proved by decreasing content of lithoclasts and increasing content of

quartz and feldspar grains towards the topmost part of the succession.

High organic-matter and pyrite contents were stated in the Lower Carboniferous flysch succession. Scanning electron microscope analysis also revealed the presence of bastnasite, what is the evidence that the rocks have not undergone high-temperature processes.

The measured values of TOC range from 0.7 to 1.65% and are sufficient for hydrocarbon generation. According to data from the southern edge of the study area vitrinite reflectance values reach from 2.26 to 4.59% (dry gas window to overmature stage). The organic matter of the Lower Carboniferous flysch sediments was stated to comprise the gas-prone, type-III kerogen.

The area located between the Kraków-Lubliniec (Hamburg) fault zone (northern boundary) and parallel of latitude of Kędzierzyn-Koźle (southern boundary) was described to comprise the optimal factors for unconventional hydrocarbons (shale gas) forming.

STRESS-DEPENDENCE OF POROSITY AND PERMEABILITY OF SHALES: IMPLICATIONS FOR GAS IN PLACE CALCULATIONS

.....

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Key words:

in-situ porosity, gas permeability, Bossier Shale, pore compressibility, effective stress

5

Information on porosity and permeability at realistic sub-surface (*in-situ*) stress conditions is a prerequisite for successful exploration and production of shale gas. In order to study the effects of elastic pore compressibility on these parameters, porosity and permeability coefficients of Upper Jurassic Bossier Shale were determined at stress levels up to 40 MPa.

Pore volume compressibility α was measured using a gas expansion technique by helium (He) expansion from a calibrated volume into the pore system of the confined sample. The recorded decrease in specific pore volume (V_p) with increasing effective stress was fitted by an exponential function:

$$V_p = V_{p,0} e^{-\alpha \sigma'} \text{ with } V_{p,0} = 0.0175 \text{ cm}^3/\text{g} \text{ and } \alpha = 0.00201 \text{ MPa}^{-1}$$

Unstressed specific pore volume $V_{p,0}$ corresponds to an unstressed porosity (φ_0) of 4.5%. At the *in-situ* effective stress value (σ') of ~60 MPa, V_p had decreased by approximately 12%.

Steady-state permeability tests were performed with six different gases and external stress levels up to 40 MPa. Apparent gas permeability coefficients (k_{gas}) increase with decreasing mean pore pressure (p_m) due to slip flow (Klinkenberg-effect):

$$k_{gas} = k_{\infty} (1 + b/p_m)$$

Klinkenberg-corrected (intrinsic) permeability coefficients (k_{∞}) decrease with increasing effective stress while slip factors (b) increase. The experimental results were fitted by exponential expressions:

$$k_{\infty} = k_{\infty,0} e^{-\alpha_k \sigma'} \text{ with } k_{\infty,0} = 5.9 \text{ } \mu\text{D} \text{ and } \alpha_k = 0.06 \text{ MPa}^{-1} \text{ for CH}_4$$

$$b = b_0 e^{-\alpha_b \sigma'} \text{ with } b_0 = 0.289 \text{ MPa} \text{ and } \alpha_b = 0.022 \text{ MPa}^{-1} \text{ for CH}_4$$

Increasing slip factors indicate that the average effective pore diameters of the shale sample are significantly reduced with increasing effective stress.

During production of a shale gas reservoir the pore pressure is reduced. Apparent permeability coefficients will increase due to slip flow whereas poro-elastic deformation will lead to a decrease in permeability during production. Based on the parameters derived from the experimental data the permeability coefficients for CH₄ were tentatively modelled for a hypothetical production history of a Bossier shale reservoir. Reduction of pore pressure results in a decrease of permeability throughout most stages of production following the exponential poro-elastic relationship. At a pore pressure of 6.5 MPa, apparent permeability reaches a minimum and then, with further decrease of pore pressure, the (apparent) gas permeability coefficient increases due to slip flow.

LIMITATIONS OF LANGMUIR MODEL FOR MODELLING CO₂ SORPTION ON GAS SHALES

.....

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Key words:

sorption, Langmuir model, gas shales

6

Depleted shale gas reservoirs can act as geological traps for CO₂. The CO₂ in shale is either adsorbed on organic matter (organic carbon) and clay minerals or is stored as a free gas in pores and fractures. In order to measure the CO₂ storage potential, adsorption measurements at the temperature of 45°C were performed in 5 different samples from the Baltic–Podlasie–Lublin Basin with a wide TOC range (0.12% to 2.04%). Three parameters Langmuir was used to model the adsorption isotherms. Different fitting results were obtained, varying the regression values between 0.99 and 0.85 due to the different behaviours of the different samples when supercriti-

cal CO₂ adsorption was performed. The sorption isotherms reach their plateau at approximately 3–6 MPa and the sudden drop in sorption is observed in the supercritical region. This could be explained by the fact that at higher pressure the density of the free phase is getting closer to the adsorbed phase density. Since some of the samples have low TOC content, it might be explained by low attraction forces of the adsorbate (CO₂) to the sorbent (shale). Therefore, in some of the cases the Langmuir model does not give a good fitting, as it was developed for the type I isotherm.

LITHOGENIC INPUT VARIATIONS IN THE BERRIASIAN OF THE KRIŽNA SUCCESSION (TATRA MTS, WESTERN CARPATHIANS): CORRELATIONS WITH CLIMATE AND SEA-LEVEL CHANGES



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Key words:

magnetic susceptibility, detrital supply, Berriasian

7

Magnetic susceptibility (MS) in sedimentary rocks is a measure of para- and ferromagnetic minerals content, distributed in diamagnetic matrix (e.g. SiO_2 or CaCO_3). It has been used in recent years as a proxy of fine lithogenic material input to sedimentary basins (e.g. Ellwood *et al.*, 2000). The MS variations in stratigraphical section, like all other indicators of lithogenic supply, might be interpreted as a result of sea-level variations or climatic changes (e.g. Duchamp-Alphonse *et al.*, 2011). We present integrated MS and geochemical record from five Berriasian, clay rich hemipelagic sections from the lower Sub-Tatric (Križna) succession from the Tatra Mts (Western Carpathians, Poland). All sections were precisely dated, by means of calpionellid stratigraphy (Pszczółkowski, 1999; Grabowski, Pszczółkowski, 2006); two of them were also calibrated, using magnetostratigraphy. The interval studied embraces the Calpionella and Calpionellopsis zones, which correspond to magnetozones from M19n to M16n. Excellent correlation is observed between lithogenic elements (Al, Ti, Rb, etc.) and MS, which indicates that MS reflects the content of fine clay minerals. The most important MS event is an increase that starts just below the Lower/Upper Berriasian boundary, i.e. Remaniella cadischiana/Calpionellopsis simplex subzonal boundary. The MS increases in the Cadischiana and lower part of Simplex subzones (M17n and M16r), but more pronounced and sudden rise is observed in the up-

per part of Simplex Subzone (M16n), which continues into the Oblonga Subzone. The latter corresponds to the Be7 sequence boundary and a major sea-level fall in the Tethyan realm (Hardenbol *et al.*, 1998; Grabowski *et al.*, 2013). Stepwise MS increase in magnetozones M17n and M16r correlates with well documented climatic change, related to humidity increase and intensified detrital supply in the Western Tethys and surrounding areas (Morales *et al.*, 2013).

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SHALE GAS EXPLOITATION AND PUBLIC ANGST



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Key words:

environmental impact, communication, public concerns

8

Whereas other energy resources experienced relatively minor changes during the last decade, unconventional resources, especially shale gas and oil, unexpectedly contributed a considerable part to the energy market. The prospect of the USA becoming energy self-sufficient illustrates the geopolitical impact unconventional resources might have in other regions. But even though there are significant economical benefits, this new type of resource is heavily criticized. Globally, there is a strong public opposition, driven by the angst of lasting environmental pollution and reduced life quality. To ease the emotional discussion, public concerns need to be heard and arising questions must be properly addressed.

In an ongoing study the Federal Institute for Geosciences and Natural Resources (BGR) is evaluating not just Germany's shale gas potential. A prime subject is the evaluation of possible environmental impacts (NIKO-Project).

In studying reports of incidents and possible environmental impacts from areas where shale gas and oil is pro-

duced, the BGR delivers science-based evaluations of potential contaminations by the exploitation of shale gas. The scientific evaluation of past and current studies will help to answer major public concerns. It is essential to filter emotionally driven flamm and myths from realistic environmental threats.

Therefore, one area of special interest is the Marcellus Shale Play in the eastern USA. Besides the economic importance, this area received a significant attention in scientific studies on environmental issues, e.g. potential groundwater contamination, and is probably the birthplace of public resistance against shale gas.

Proper addressing of these public concerns will be an important future task for local authorities, governments and operators to conduct European shale gas exploitation in a socially agreeable way.

STRUCTURAL FEATURES OF THE PHANEROZOIC FORMATION IN THE EXPLORATION BOREHOLES LOCATED IN THE BORDER ZONE BETWEEN THE EAST EUROPEAN CRATON (BALTICA PROTO-PLATE) AND THE EUROPEAN PALEOZOIC PLATFORM (MAŁOPOLSKA BLOCK)



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Key words:

tectonics, sediments, boreholes

9

Shale basins exhibit diverse tectonics, stratigraphy and lithology. The main source providing geological information, is the data from the borehole. When analyzing shale basins, it is crucial to use and correlate the knowledge from the different branches of geology. The understanding of tectonics and structural geology in a rock deformation processes and structures formation helps in the geological interpretation and in the hydrocarbon exploration works.

The boreholes Narol IG 1, Dyle IG 1, Książpol-12 and Dzików-17 are located in the marginal zone of the East European Craton and the NE marginal zone of the Małopolska Block. It is an area located near the Holy Cross Fault Zone (HCFZ). The Cambrian rocks from the mentioned boreholes were mezostructurally analyzed. In particular the morphological, orientation, superposition and kinematic analyzes were performed.

The Cambrian sediments in these boreholes are both poorly and strongly involved tectonically. Sedimentary layers are placed horizontally, vertically and subvertically. The angle between the sedimentary surfaces and a horizontal plane (dip) is extremely variable, and ranges

from 0 to 90°. In the Książpol-12 borehole, the dip oscillates within the bounds of 5 to 65°. In the Dzików-17 borehole the dip oscillates within the bounds of 30 to 90°. In the Narol IG 1 borehole, sedimentary surfaces have generally subhorizontal orientation, they are inclined at the angles not exceeding 10–15°. In the Dyle IG 1 borehole, clastic Cambrian formations have been very intense, multi-phase folded. They are characterized by well-marked sedimentary surfaces. The observed large variation of inclination of layers, is closely associated with a multi-stage tectonic evolution of these rocks.

The formations in the analyzed boreholes show highly complicated geological structures. The rocks represented by the drilling cores are involved tectonically in many different ways, and a lot of structures like faults and folds of a different origin can be observed. Folding processes and activity of thrust, normal and slip faults have the greatest impact on the rocks, leading to the rip-off of layers and the formation of the fault folds. We may state that the analyzed Cambrian rocks can be the reservoir rocks for hydrocarbons in this area.

CHARACTERIZATION OF GAS-BEARING SHALE ON THE EXAMPLE OF THE CORE FROM THE BOREHOLE LUBOCINO-1

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Key words:

gas-bearing shale, borehole Lubocino-1, properties characterization, Baltic basin

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In the framework of this study were characterized physical and mechanical properties as well as mineralogical and chemical composition of gas-bearing shale from the Baltic basin. Samples of the core taken from the well Lubocino-1 (PGNiG SA concession area), dated from the interval of late Ordovician and early Silurian were selected for the study. The results of the physical properties study let us know about the distribution of the particle size in relation to their densities. Thanks to the use of computer microtomography non-destructive analysis of the internal structure of the shale was possible. The analysis of the mechanical properties of shale rock performed by uniaxial compression test was performed using DIC (Digital Image Correlation), which allowed to obtain high accuracy. Compressive strength of low-carbonate shale

was estimated at 125 ± 25 MPa with the Young's modulus of 16 ± 2 GPa. On the other hand, high carbon shale are characterized by compressive strength of 50 ± 30 MPa and a Young's modulus of 4.5 ± 0.5 GPa. The results of chemical analysis shows that the main element in the analyzed samples is silicon. Significant content (more than 3%) of aluminum, iron and potassium was also found. It was also determined that the examined sample consists of seven phases. The main phases by Semi-Quantitative analysis are quartz (50.57% of weight), muscovite-mica potassium (27.31%), chloryt (11.25%) and albite (8.25%). The total content of calcite, hematite and dolomite is 2.61%. Thanks to these research wide characterization database of shale rocks from Lubocino-1 borehole has been developed.

ORDOVICIAN–SILURIAN SOURCE ROCKS IN THE DARŻLUBIE IG 1 BOREHOLE: THEIR THICKNESS, DISTRIBUTION AND BASIC CHARACTERISTICS



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Key words:

Baltic Basin, Darżlubie, TOC, hydrocarbons

11

Darżlubie IG 1 borehole is located in the north-western part of the Baltic Basin, Poland. Potentially prospective for hydrocarbon generation intervals of Wenlock, Llandovery, Ashgill, Caradoc and Llanvirn are covered by 2738 m overburden composed of sedimentary rocks, including Paleozoic, Mesozoic and Cenozoic strata. Zone of the potential hydrocarbon generation is located at the depth interval from the top of Wenlock to the bottom of Llanvirn (2978 m). This 240 m thick interval is built by unevenly distributed rock sections of non-source rocks, poor and very good source rocks. Wenlock is composed of 139 m thick interval of dark-gray, shaly claystones. In turn, Llandovery forms dark gray claystones with a thickness of 66 m. Ashgill builds clayey siltstones and marls with a thickness of about 8 m. Caradoc is dominated by claystones gray and black in color, with a total thickness of 22 m. Stratigraphically oldest, the Llanvirn interval is formed by marly limestone and grey claystones with thickness of 10 m. Hydrocarbon potential (HI) of the perspective Wenlock-Llanvirn section ranges between 90–376 mg HC/gTOC and thermal maturity of organic matter (T_{max}) is in the range of 441–474°C. The TOC content oscillates from 0.03 to 5.92 wt%, and the volume

of the S2 parameter vary in the range of 0.13–11.37 mg HC/gRock. Contribution of the residual carbon in the volume of TOC is variable and fall in range between 50–90%, which in conjunction with T_{max} and HI parameters allowed to distinguish 3 sections of very good source rocks, 4 sections of poor source rocks and 3 non-source rock sections. The degree of thermal alteration of examined samples indicates that the majority of them fall in the range of an oil window (80%), and only a small number in the gas window. Access to the core material has allowed to complete sampling of the stratigraphic interval of Llanvirn, lower and upper Caradoc, Ashgill, and the lowermost section of Llandovery. Stratigraphically oldest, very good source rocks section of the Llanvirn strata is dominated by the type III kerogen and TOC 1.9–2.4 wt%. A very good source rocks of Caradoc and Ashgill are characterized by kerogen type III/II and TOC 1.0–5.7 wt% and kerogen III with TOC 2.1–5.9 wt%, respectively. To determine the continuous profile of the TOC content, appropriate archive Russian-style logs have to be environmentally corrected, standardized, mathematically transformed and calibrated with properly depth-shifted, core-derived TOC data.

ORDOVICIAN–SILURIAN SHALE GAS SWEET SPOTS – LOST, HIDDEN OR NON-EXISTENT?



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Key words:

sweet spots, shale gas, Ordovician-Silurian, Exploration and Production, Poland

12

After the development of many potential shale gas/oil plays in the US, it has been realized that not all shales can provide the economic production. For national and international E&P companies Polish Ordovician-Silurian shale gas exploration works have been a so called “greenfield” projects and the good targets for the hydrocarbon production, which can be defined as the sweet spots are not yet found.

It was planned to locate the most perspective areas for the shale gas production after drilling more than 100 of wells as a stipulated number. With only 64 exploration wells today (June, 2014), the question whether these sweet spots will be found or they will be announced non-existent remains unanswered.

The sweet spot is defined as follows: “Shale reservoir sweet spots are areas that produce noticeable better than other areas” (Gading *et al.*, 2013). The more detail expression is: “sweet spot – Colloquial expression for a target location or area within a play or a reservoir that represents the best production or potential production. Geoscientists and engineers attempt to map sweet spots enable wellbores to be placed in the most productive areas of the reservoir. Sweet spots in shale reservoirs may be defined by source-rock richness or thickness, by natural fractures, or by other factors, using geological data such as core analysis, well log data, or seismic data” (Slb: Oilfield Glossary).

The parameters that influence shale reservoirs quality and therefore determine the sweet spots characterization include: organic content, kerogen type, thermal maturity, formation thickness, porosity, permeability, hydrocarbon saturation, mineralogy, rock hardness, natural fracture orientation and density, in situ stresses, formation pressure and more.

The sweet spots location can be achieved by many methodologies starting from the analyses of the cored perspective shale intervals, through the analysis of a well logging data, determination of a proper seismic attributes, and finally ending at the testing of a reservoir empirically by hydraulically fracturing the vertical and horizontal section of a borehole that was drilled in a target interval.

Till today, in a case of the Polish Ordovician–Silurian basin the “sweet spot” is defined as an area of the reservoir that represents potential production.

The reasons why a sweet spot is located in the certain place can be explained by the sedimentological and paleogeographic facies analysis of a basin. Polish Ordovician-Silurian basin differs from most of the US basins as it constitutes the Caledonian foreland basin with facing margin controlled by the tectonic structures of the edge of East-European crystalline platform. The basin unique structural framework determined lithofacies and organic matter distribution where “sweet spot” (if any ?) can actually be formed in the shape of a “sweet sedimentary belt”,

“dispersed sweet mini-spots” or even “sweet erosional channels”. Furthermore, late erosion of the Ordovician-Silurian shales and related deposits on vast Mazury Suwałki uplift area, may caused the preservation of remnants of the former sweet spots just on the present erosional basin margins.

The poster presents our considerations on search of an expected Ordovician-Silurian sweet spots and may guide the way to answer the question in the title: Ordovician-Silurian shale gas sweet spots – lost, hidden or non-existent?

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AUTHIGENIC SILICATES ASSOCIATED WITH MICROBIAL ORGANIC MATTER IN EARLY SILURIAN SILICEOUS ROCKS FROM POLAND



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Key words:

cyanobacteria, silicates, black cherts, silicification, Silurian

13

Silicate minerals are known as authigenic precipitates in marine environments. Some of them (clay minerals) have been recognized as crystallizing in association with living organic matter, in soils, biofilms and microbial mats, particularly in specific environments such as streams and salt lakes. Some of these silicates were apparently formed as a result of metabolic activity of microorganisms or are products of interactions of microbial metabolic processes with the surrounding environment. Experiments showed that microbes were able to influence and, to some extent, control the microenvironment around their cells, and in that way mediate precipitation of a variety of silicates. Such silicates can be considered as biominerals. The authors recognized the occurrence of tiny silicate flakes and grains (interpreted as biominerals) attached to fossilized remnants of coccoid cyanobacteria in the marine Early Silurian black cherts and siliceous shales (TOC 0.43–8.11%) from the Sudety and the Holy Cross Mts., Poland. The cyanobacteria and associated organic matter form layered structures which represent remnants of the former microbial mats. These mats underwent early diagenetic silicification in a peculiar environment highly enriched in silica due to a “rain” of opaline tests of dead

radiolaria that settled on the mats overgrowing the sea bottom. It is assumed that both the silicification of the microbial mats, and the precipitation of the authigenic silicates were primarily driven by pH changes within the degraded parts of the mat. Since silica solubility increases with the rise of pH, it is expected that early diagenetic massive dissolution of the radiolarian opaline skeletons took place during earliest stages of the mat biomass degradation where significant pH increase could have occurred due to photosynthetic mat activity (up to pH 8.82 in a modern marine mat). The dissolved silica diffused downward where mat bacteriolysis generated lower pH, hence decreasing silica solubility and causing silica precipitation. Precipitation of silicates in the mats could take place almost simultaneously with the overall silicification of the mat. Both processes were related to almost simultaneous excess of silica and to availability of various ions (Al^{3+} , Mg^{2+} , K^+ , Ca^{2+}) which during the cyanobacterial life time were bound into cyanobacterial mucus and released after mat degradation.

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LITHOLOGICAL FRACTIONS AND FLOTATION EFFICIENCY OF COPPER ORE IN KGHM POLSKA MIEDŹ S.A.

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Key words:

copper ore, shale, ore enrichment, geochemistry, concentrate grade

14

The copper ore deposit exploited by KGHM Polska Miedź S.A. is one of the world's largest deposits, and is located within a large structural unit named Fore-Sudetic Monocline. The rock strata mined by KGHM are of sedimentary origin, with later copper deposition. The ore is characterized by three types of lithological fractions, formed by the same basic minerals, mainly chalcocite, bornite, covellite and chalcopyrite, however in different proportions. A layer of sandstone is separated from the carbonate rock by a layer of shale containing organic matter, which turned into the carbon over time. The cause of difficulties related with an enrichment of copper is a systematic increase in the amount of shale, the most important source of organic carbon in the ore, and consequently, in the concentrate. The shale is characterized by the most difficult beneficiation in comparison to other components of lithological fractions. It is a mixture of clay minerals and carbonate, quartz with a small amount of addi-

tionally bonded organic substance, however the higher is the amount of sandstone and carbonate, the better are the grinding requirements fulfilled. The shale proved to be a significant factor in flotation behavior and appears to carry a significant proportion of the copper. Copper is spread in the shale, what increases the mass recovered to the concentrate and thus lowers the concentrate grade. The aim of the investigation was identification of changes in the ore enrichment depending on the lithological fractions and their quality. The enrichment curves of the lithological fractions were designed, basing on the ingredients characteristic curves, what will enable the prediction of ore enrichment results. Preliminary analysis of the ore samples showed that for different regions, the enrichment curves for lithological types are arranged above the curves obtained for the mixtures, and below the curves obtained in the laboratory analysis.

EVIDENCE OF WILDFIRES DURING DEPOSITION OF THE UPPER SILESIAN KEUPER SUCCESSION. MOLECULAR COMPOSITION OF FOSSIL CHARCOAL AND RELATIONSHIP WITH INCOMPLETE COMBUSTION OF WOOD

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Key words:

charcoals, fusinite reflectance, wood taxonomy, biomarkers, Upper Triassic

15

Charcoals from the Upper Triassic vertebrate-bearing clays of the Zawiercie area were analyzed using petrographic methods, to characterize their taxonomic affiliation and to reconstruct burning temperatures as well as taphonomic processes. SEM and reflected light microscopy show excellent preservation of charcoals most probable connected with early diagenetic permineralization by calcite. The charcoal were assigned to three morphotypes, probably corresponding to three different fossil taxa: *Agathoxylon (Dadoxylon) keuperianum*, *Agathoxylon* Hartig and *Brachyoxylon* Hollick et Jeffrey. Fusinite reflectance data suggest that the highest temperature reached above 600°C, corresponding to average reflectance of 3.59%, what counterparts to the lower limit crown fire temperature. The values for most of the samples are lower (c.a. 1% to 2.5%), what correspond to surface fires. In many cases fusinite reflectance values depend on the measured zone within the sample, which implies that calculation of fire temperatures based on average fusinite reflectance values might be too far-reaching simplification. Occurrence of fungal hyphae within the charcoal supports the interpretation of a predomination of surface fire, consuming dead twigs and stems. Charcoal extracts were also studied using gas chromatography – mass spectrometry (GC-MS) to recognize

their molecular composition. Extractable compounds were divided into biomarkers, i.e. diagenetically changed primary wood components and products of combustion. Major compounds in the first group were: 1,2,5-trimethylnaphthalene and 1,2,5,6-tetramethylnaphthalene, cadalene, dehydroabietane, simonellite and retene. All of these are derived from resins. Moreover, propyl phenols, butyl acetophenones and pentyl acetophenones, as products of lignin breakdown, as well as fatty acids with a predominance of palmitic acid, typical constituents of wood, were also detected. Polycyclic aromatic hydrocarbons (PAHs), as well as ketones and aryl phenols, considered as high temperature combustion products, occurred at relatively low concentration in the samples due to their enhanced solubility in gelified, non-charred wood fragments, and vaporization of the major part of the burning products. Despite the low PAH concentrations, their distribution, with a significant contribution from typical pyrolytic compounds such as anthracene, 4H-cyclopenta[def]phenanthrene, benz[a]anthracene and benzo[a]pyrene was typical for rapid combustion. There was no direct relationship between the PAH concentration and wildfire intensity.

DETERMINATION OF GEOMECHANICAL PARAMETERS OF SILURIAN AND ORDOVICIAN ROCKS IN THE BALTIC BASIN

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Key words:

Baltic Basin, geomechanics, Young's modulus, Poisson's ratio, brittleness

16

Introduction

Shale gas – great geological discovery of last decades has or may have unusual impact on some countries' economies around the world (Gąsiewicz, 2013). Geomechanical parameters belong to the most important aspects of exploration as they determine success of hydraulic fracturing of the source rock. The main objective of this study was showing the determination way of specific parameters such as Young's modulus, Poisson's ratio and Brittleness of Silurian and Ordovician rocks in the Baltic Basin.

Methodology

Geomechanical parameters define the material response to applied stress. The most important elastic parameters are Young's modulus and Poisson's ratio. Young's modulus (E) represents the material stiffness, while Poisson's ratio (ν) is a ratio of the relative change of transverse strain to the relative change of the axial strain of the material (Dadlez, Jaroszewski, 1994). In most cases, to estimate this parameters the well log data are used, especially P and S wave velocities and bulk density, as those attributes are dependent on elastic parameters (Plewa, Plewa, 1992). All the data used in calculations in this particular study come from public sources, mostly from literature. Additional physical property of rocks is Brittleness Index. It describes how rocks are rupturing during the stress

applying (Perez, 2013). Brittleness in unconventional reservoirs is controlled by mineralogy. The presence of quartz and carbonates makes shales more brittle, while the presence of more clay makes shales more ductile. Shale with higher Young's modulus and lower Poisson's ratio tend to be more brittle. Ductile rocks exhibit low Young's modulus and high Poisson's ratio.

Conclusions

Finding the Sweet Spot is a broad issue, for as much is not enough to find a zone characterized by a very high content of TOC. Effective productivity of the continuous gas accumulation is strongly dependent on geomechanical properties of rock and brittleness factor.

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RELEASE OF CONSTITUENTS FROM SHALE DRILL CUTTINGS UNDER BATCH TESTING



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Key words:

shale cuttings, leaching, constituents mobility

17

Shale rocks are formed from sediments deposited in stagnant aquatic environments and as such are composed mainly of inorganic minerals enriched in organic compounds. However, these formations are also known to contain many potentially toxic elements, especially heavy metals. Constituents of potential concern present in the materials may be subject to leaching during hydraulic fracturing, storage, and reuse applications of both drill cuttings and flowback water. Runoff and water percolating through the materials may produce drainage water rich in metals and organics, carrying them to the ground, and eventually into the aquifers or surface streams. Treatment and recycling of flowback water from shale gas production may pose risks to aquatic biota if directly discharged to the environment, and create problems in reusing of the fluid. Therefore, leaching tests are common tools that provide a fundamental basis to estimate constituent release under different conditions.

In this study, the constituent leaching from shale drilling cuttings extracted from marine deposits in Baltic Basin formed in Lower Silurian Llandovery age was evaluated.

Batch test as a simple tool for compliance or quality control reasons was used to represent solubility and release different pH conditions. The composition of shale rock used in the experiments was determined by X-ray fluorescence spectroscopy, and CHNSO elemental analysis, whereas the released constituents in aqueous phase by atomic absorption spectrophotometry (AAS), and UV/VIS spectroscopy.

The total analysis of the substrate indicated that the elemental composition is dominated by Si > Al > Fe > Ba > Ca > K > Mg > S > Na. The mobility of most elements contained in shale drill cuttings was observed to be markedly pH sensitive. Maximum heavy metal extraction (Cu, Pb and Zn) was obtained at acidic conditions. Na, Ca, Mg, Fe, K, Mn are elements that can, in the long term, be leached in considerable amounts from the shale material especially at lower pH. Minimum solubility of metals was attained for the pH 7 and 12, whereas for pH 10 relative attenuation of the metals release was observed. Determination of the TOC content in leachates revealed leachability of organic matter from shales of (0.50–0.65 g/L) regardless of pH.

SORPTION CAPACITIES OF BOSSIER AND HAYNESVILLE SHALE AS A FUNCTION OF PRE-ADSORBED WATER



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Key words:

sorption, shale, high pressure, moisture

18

The sorption behaviour of CH₄ on Bossier and Haynesville shales with varying moisture conditions has been studied at 318 K and pressures up to 30 MPa. The aim of this study was to assess the influence of pre-adsorbed water on the maximum sorption capacity.

Measurements were conducted using a manometric set-up with continuous pressure and temperature recording. Sorption tests on each sample were conducted at four different moisture states resulting in a total of eight isotherms. The samples were moisture-equilibrated with three different salt solutions establishing relative humidity (RH) levels of 33, 55 and 97%. The water uptakes in weight percent were 1.3, 1.8, 7.1% for the Bossier and 1.0, 1.3, and 4.7% for the Haynesville shale, respectively.

The maximum CH₄ excess sorption capacities of the Bossier shale decreased from 0.11 mmol/g at dry conditions to 0.02 mmol/g at 97% RH moisturisation. The Haynesville shale sample showed a similar decrease in excess sorption capacity from 0.095 mmol/g in the dry state to 0.02 mmol/g at 97% RH. All isotherms have a maximum

between 8 and 15 MPa. Moisturisation of both samples at 33% RH resulted in a significant decrease in excess sorption capacity compared to the dry state (Haynesville: -37%; Bossier: -64%). Subsequent increase in moisture content led to only a relatively small decrease in sorption capacity compared to the effect of the initial moisturisation step (33% RH). This holds also for the final moisturisation step (55–97% RH), where a significant water uptake results in only a relatively small decrease in sorption capacity. Except for the dry state the Haynesville shale shows significantly higher sorption capacities than the Bossier shales at all moisture levels. The results of this systematic study show that already small amounts of pre-adsorbed water have a significant negative effect on the sorption capacity of carbonaceous shales. This effect is strong at small amounts of pre-adsorbed water far below water saturation. The sorption capacity will not be as strongly influenced upon further increase of moisture content.

VOLUMINOUS STREAMS OF WATER IN EXTRACTION OF HYDROCARBONS



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Key words:

water, flowback fluid, recycling, extraction, hydrocarbons

19

Water is of crucial importance while considering the energy sector, especially in extraction of oil and gas. Oil and gas companies have to face various aspects pertaining to water, collection of water for e.g. reservoir stimulation, wastewater recycling from almost every process.

In the case of extraction of oil or gas from unconventional deposits, water of appropriate quality is substantial in the hydraulic fracturing process. Hydraulic fracturing uses water with chemical additives to crack the shale. It consists of around 95% fluid and is injected into the shale formation to increase permeability of the rock and to unblock the movement of gas or oil residing within the structure. After completion, part of injected water returns to the surface as the flowback fluid, which contains pollutants, requiring removal before disposal.

Oil and gas companies view water as a strategic component of the value chain. Managing water resources requires a strategic approach, since it is essential to production growth. Over the last years, the water demand for oil and gas recovery increases, which induces the holistic approach to water management in the upstream oil and gas industry. In many locations, such as Pomerania, the amount of water required by the oil and gas industry is small comparing the overall availability of water resources and water consumption by other industrial sectors. The development of new techniques, which will lead to improved production processes and water treatment operations, is needed.

FACTOR LIMITING SHALE GAS EXPLORATION IN POMORSKIE VOIVODESHIP



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Key words:

shale gas exploration, water resources, groundwater resources, spatial planning, environmental policy

20

Ground and surface water availability, impact of legally protected areas, and the management of oil and gas production areas in Pomorskie Voivodeship are considered. Environmental requirements are similar for conventional and unconventional deposits excluding differences in water demand for completion and stimulation of shale gas reservoir. For hydraulic fracturing from 8,000 to 20,000 m³ water per 1000 m of borehole length is needed. The groundwater resources in Pomorskie Voivodeship are discussed with respect to stratigraphy, area, depth

and available resources. Spatial planning related to the protected area, agricultural land and coniferous and deciduous forests as well as woody and bushy land is addressed. Population density in Pomerania (125 person/km²) and protected areas (32.7%) may influence the drilling pad site location. Access to water in Pomorskie Voivodeship is very conducive due to the presence of 17 major groundwater reservoirs. Also surface waters, including surface saline waters of the Baltic Sea, are being considered for hydraulic fracturing.

ENVIRONMENTAL RADIOACTIVITY OF DRILLING WASTES PRODUCED FROM SHALE GAS EXPLORATION

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Key words:

TENORM, gamma spectrometry, radiological hazard, drilling wastes, shale gas

21

Exploration and exploitation of oil and gas reservoirs may cause migration of naturally occurring radioactive materials (NORM) and lead to the concentration in drilling wastes. The aim of the research was to evaluate radiological hazard of drilling wastes produced from shale gas exploration in the Baltic Basin. 7 NORM can appear in drilling cuttings, flow back fluid, scale and sludge in facilities such as pumps, pipelines and separators. Equivalent absorbed dose from the gamma ionizing radiation was determined by portable NaI scintillation spectrometer

and was similar to the background radiation in the area. The global annual effective dose from background radiation is 2.4 mSv/a, and in Poland the average dose is 3.3 mSv/a. In examined materials, the most active radionuclide is potassium K-40. The average absorbed doses did not exceed accepted levels of natural radioactivity for wastes. The drilling cuttings and flow back fluid from shale gas exploration in the Baltic Basin did not contribute to workers exposition to the gamma radiation.

LATE CAMPANIAN–EARLY MAASTRICHTIAN CALCAREOUS NANNOFOSSILS BIOSTRATIGRAPHY AND PALEOECOLOGY OF THE GURPI FORMATION (GURPI ANTICLINE – SW OF IRAN)

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Key words:

calcareous nannofossils, biostratigraphy, paleoecology, Zagros, Iran

22

Gurpi Formation consists of dark grey to grey marly shales, marls and marly limestones which has a good outcrop and extensive exposure in Zagros Basin in Iran. The present study focuses on the lower part of the Gurpi Formation in the central Zagros basin of Iran at the Dezful embayment. The aim of the present study is to exactly date the studied interval with regard to the calcareous nannofossils and to determine the calcareous nannofossil biozonation and paleoecology.

A total of 129 samples with a sampling resolution of about 2 m were collected and were processed using the gravity settling technique. The slides were studied with an Olympus BH2 microscope at 1250x magnification.

Calcareous nannofossils have been investigated at the lower part of Gurpi Formation at Gurpi Anticline in the south west of Iran. The lower part of Gurpi Formation consists of grey to yellow marls, marly limestones, shales and limestones. According to the calcareous nannofossils data, the studied interval spans from Late Campanian to Early Maastrichtian and from the middle part of CC22/UC15e^{TP} to CC24/UC18. Eight bio-horizons are recorded at the studied interval, which are as follows from the bottom to top of the section respectively: LO of *Reinhardtites anthophorus*, LO of *Eiffellithus eximius*, LO of *Uniplanarius trifidus* (Long Ray), LO of *Aspidolithus parvus parvus*, LO of *Aspidolithus parvus constrictus*, LO of *Uniplanarius trifidus*

(Short Ray), LO of *Uniplanarius gothicus*, LO of *Tranolithus orionatus*. The studied interval encompasses the Campanian–Maastrichtian boundary (CMB). At the Late Cretaceous (Campanian–Maastrichtian) a cooling event is recorded with some oscillations, which is associated with changes in the calcareous nannofossil assemblages and is recorded by the expansion of cold water taxa towards low latitude (such as *Ahmuellerella octoradiata*, *Gartnera-go segmentatum*, *Kamptnerius magnificus* and *Nephrolithus frequens*). At the studied interval, the abundance of the above mentioned cold water taxa is below 0.5%, but other taxa that are considered as cool water (e.g., *Discorhabdus ignotus*, *Biscutum constans*, and *Tranolithus orionatus*) are present along with warm water taxa (e.g., *Watznaueria barnesae* and *Ceratolithoides* spp.), although the number of warm water taxa is higher than cool water taxa. It must be mentioned that the oligotroph taxa (such as *Watznaueria barnesae*, *Eiffellithus* spp., *Lithraphidites* spp., *Staurolithites* spp. and *Prediscosphaera* spp.) are observed along with the eutroph and mesotroph taxa (e.g., *Discorhabdus ignotus*, *Biscutum constans* and *Zeugrhabdus* spp.), but the number of oligotroph taxa is higher than eutroph and mesotroph taxa, which is similar to DSDP Hole 390A (Black Nose). The increase in the size of *Arkhangelskiella cymbiformis* is also observed above the CMB, which is similar to the other parts of the world.

EVALUATION OF BLOATING PROPERTIES OF SHALE CUTTINGS

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Key words:

shale cuttings, lightweight aggregate, mineralogical composition, area of bloating

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Lightweight aggregates (LWAs) constitute granular material with the loose bulk density not exceeding 1.20 g/cm^3 or with a particle density not exceeding 2.00 g/cm^3 that is formed by rapid, high temperature sintering of substrates that have the ability to expand.

The objective of this investigation was to utilize shale drill cuttings as an additive to lightweight aggregate production. The chemical and mineralogical composition of shale cuttings was determined by X-ray fluorescence spectroscopy and X-ray diffractometry. The natural radioactivity of the material was measured by potassium, radium and thorium isotopes content (K_{40} , Ra_{226} , Th_{228}) by the scintillation method.

The results indicated that shale cuttings can be classified as low plastic clay and the chemical and mineralogical composition is located within the Riley's 'area of bloating'. Content of fluxing elements ($\Sigma F - Na_2O, K_2O, CaO, MgO, Fe_2O_3, FeO$) and melting behavior expressed as Al_2O_3/SiO_2 and $(CaO+MgO)/(SiO_2+Al_2O_3)$ ratios follow the composition requirements for the application of shale cuttings as an acceptable material for LWA manufacture. The $SiO_2/\Sigma F$ ratio was calculated to be above 2, which is the minimum value required to maintain suitable viscosity during sintering.

INDEX GRAPTOLITES FOR SILURIAN BIOZONES IN POLAND – THE STUDY OF ARCHIVAL AND NEW CORE DATA



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Key words:

biostratigraphy, graptolites, Silurian, East European Craton

24

The shale-dominated succession in the western part of the East European Craton (EEC) represents the most complete Silurian graptolite record. The graptolites occurred there, being the extremely fast evolving group of plankton organisms. Therefore, they are an excellent tool for the biostratigraphic dating of the Ordovician and Silurian organic rich fine-grained sediments which are considered as the most prospective rocks for shale gas exploration.

The sedimentary cover of the Polish part of the EEC was penetrated by hundreds or so deep boreholes mainly in the 50–80s of the 20th century. The history of the Polish paleontological studies of the Silurian graptolite fauna has began in the early 20th century which resulted with the detailed stratigraphic scheme for the Silurian system in Poland.

Recently, numerous new boreholes have been drilled in Poland due to prospection of zones with the unconventional hydrocarbon resources. Some new fully cored and continuous profiles provided rich faunas of beautifully preserved, both flattened and uncompressed graptolites and the crucial data on the development of graptoloid faunas and the Silurian graptolite biostratigraphy in Poland. The new core data as well as the archival paleontological material proved the great taxonomic composition of the graptolite assemblage including index and characteristic taxons for Silurian graptolite biozones and were the base for stratigraphic subdivision and regional correlation of gas-bearing shale rocks in Poland.

GEOCHEMICAL PECULIARITIES OF ORGANIC MATTER SHALE STRATA OF JURASSIC AGE

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Key words:

organic matter, shale strata, Jurassic age, suite

25

Within the frames of this work we carried out comprehensive geochemical study of high-carbon rocks samples taken from the three segments of the Jurassic system – from the lower (Kotuhtinskaya suite), from the medium (Tyumenskaya suite) and from the upper (Vasyuganskaya, Georgievskaya and the Bazhenovskaya suites).

The complex study of the organic matter (OM) of these strata included the following: chloroform extraction of bitumen, the determination of the group and element composition, gas chromatography (GC) and gas chromatomass-spectrometry (GC/MS).

As a result of this work we identified various biomarkers that allow characterizing each oil and gas source strata under the study in the open-cast of the Jurassic system:

Kotuhtinskaya suite. The build-up of this suite took place in the coastal marine weakly reducing conditions. In their composition these deposits contain some highly transformed humus organic matter (gradation of catagenesis MK3).

Georgievskaya suite. Accumulation of OM in this strata occurred in shallow marine strongly reducing conditions. According to its type, this matter is sapropel, and plankton-algal composition was the source material for it. Its

accumulation occurred in terrigenous clay sediments. Organic matter of Georgievskaya suite is situated in the area of moderate maturity. Also the traces of oxidation and of some contribution of ground vegetation were revealed in the OM, all that somewhat reduces the oil and gas generating potential of this strata.

Bazhenovskaya suite. The organic matter under the study accumulated mainly in shallow marine reducing conditions. Some small residue of ground vegetation is detected in the deeper layers. The type of the Bazhenovskaya suite organic matter is defined as sapropel. The source matter for this organics is a mixed – bacterial and algal type. OM is characterized of sufficiently high maturity.

Thus, as a result of the complex geochemical studies several strata that might participate in the formation of oil and gas deposits were revealed. These strata include Tyumenskaya suite of the middle segment, Georgievskaya the Bazhenovskaya suites the upper segment of the Jurassic system. Composition of the OM of these strata, the conditions of accumulation and the degree of their catagenetic transformation allows us to label them as having great potential, and as probably effective oil source strata.

NEW DATA ON THE DOM (DISPERSED ORGANIC MATTER) SHALE ROCKS OF SOUTHERN KAZAKHSTAN SECTOR OF THE CASPIAN SEA (GEOCHEMICAL METHODS)



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Key words:

geochemical study, shale rocks, sector of the Caspian Sea

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In this paper, we want to highlight the results of geochemical study of the sedimentary cover of the southern part of Kazakhstan sector of the Caspian Sea. In recent years, several wells have been drilled here and so we obtained core material for further studies. In particular, in the arching of "Rakushechnoye" structure, well R1, and "Nursultan" structure, well N1, Triassic, Jurassic and Cretaceous deposits were revealed.

The DOM from the strata within the continental zone was mostly accumulated in the coastal marine reducing conditions; the composition of organic matter is mixed – humus-sapropel – and is situated in the "zone of the oil window".

The results of geochemical studies of DOM of the rocks that N1 well revealed show that it is characteristic of different values for biomarker ratios in the section. Perhaps this is due to the presence of the oil-and-gas source rocks that are now in the zones of oil-and-gas formation, as well as to the presence of syngenetic OM and migrated HC.

The research showed no potential oil-and-gas source rocks among the studied samples. The rocks of the Lower Cretaceous and Upper Jurassic contain only migrated HC

and according to the results of Rock-Eval, possibly they are productive in the context of a given well. Only a few samples from the Middle Jurassic (TOC = 2.23%) can be defined as "poor" gas source series (HI = 77mgUV/g TOC), which has not yet reached the required level of maturity for gas HC generation ($T_{max} = 431^{\circ}C$). However, we should mention that the studied samples were mainly represented by carbonate rocks with small amounts of clay material and only a few of them (Middle Jurassic samples) were represented by clay variations. Therefore, it is too early to draw conclusions regarding the oil-and-gas generation potential of Mesozoic rocks.

Thus, the geodynamic evolution of the region was a complicated process, during which there were conditions for the accumulation of oil-and-gas source series and for transformation of organic matter, both in the Triassic and Jurassic deposits. Therefore, further study of the generation properties of the rocks in the section is needed; and we would recommend that future samples be taken from clay intervals according to the data from GIS wells that were drilled in the shelf zone.

VARIABILITY OF SHALES IN THE SWEET SPOTS OF THE POLISH SHALE GAS PLAYS ON THE BASIS OF LABORATORY AND WELL LOGGING DATA

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Key words:

shale gas plays, well logging, petrophysics, statistics, cluster analysis

27

Introduction

The goal of the study was to enhance and improve information on the Polish shales with gas saturation. Multivariate statistical studies were performed on the available data. Two sweet spots and surrounding formations were analysed in the well located in the land part of the Baltic Basin (Poland). First group of data represented Llandovery (Silurian) formation – Jantar Member in Pasłek Formation when the second one, Sasino Formation, belonged to the Caradoc and Llanvirn (Ordovician) formation.

Working material

Laboratory results included bulk and skeletal density, total and effective porosity, clay bound water, capillary and free water saturation, mineral recognition and TOC. Standard well logging curves included transit time interval, neutron porosity, bulk density, index of photoelectric absorption, natural radioactivity, volumes of potassium, uranium and thorium, apparent resistivity in flushed and uninvaded zone.

Combination of laboratory and well logging data

Different vertical resolution of well logging devices and point laboratory data were taken into account to make the best ties between data. TOC point data and various gamma logs (continuous curves) were the basis for depth matching. Precise analysis of well logs revealed diverse anomalies in sweet spots sections.

Statistical methods

Basic statistics were calculated for all, lab and logs, parameters. Correlations showed diversification between the formations. Based on cluster analysis, groups corresponding to sweet spots zones were found and diversification of formation was confirmed.

Results and conclusions

Preliminary statistical analysis of the lab data and well logging data from single well in two sweet spots revealed visible diversity of shales. Cluster analysis was used for the classification and grouping of data according to natural petrophysical features of the analysed rocks. Groups corresponding to the sweet spots were found. Results were compared to standard logs interpretation. Complex analysis showed diversification: firstly, between sweet spots and surrounding, secondly, between the two sweet spots formations, thirdly, internal diversification in each sweet spot. It is concluded that multivariate statistical analysis occurred to be a useful and quick tool for preliminary classification of members and gas-saturated zones identification. Analysis improved characteristics of shales with gas saturation.

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ADVANCED PERFORATING SYSTEMS FOR SHALE GAS RECOVERY



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Key words:

perforation, pre-completion, shale gas, explosive, propellant

28

Hydraulic fracturing (HF) is main method of shale gas well completion. However, it should be remembered that before each HF operation, well pre-completion is carried out and the perforation of a well has to be performed. Explosive charges are used to create a perforation tunnel through the pipe, the casing and a small length of the reservoir. It is an important operation in the shale gas recovery and has a significant impact on production rate. There are many methods of perforation performing. The simplest and also the least effective technology of a well pre-completion is called a perforating gun. It consists of directional detonations of high explosive charges in order to give a short perforation channels. There are also few combined, perforating-fracturing systems based on low and high energy explosive materials which already exist on the market and are used in the pre-completion stage of unconventional oil and gas recovery. The best known and the most promising ones are StimGun and GasGun technologies. They both combine perforating and fracturing with propellant stimulation technology. They have much greater range of perforation than other systems and also these two methods

are best for the rock formation with high content of water sensitive illite and smectite. Characteristics and the comparison of strengths and weaknesses of each of these methods have been shown. In addition, detailed analysis of commonly used propellant parameters has been done.

There are numerous problems which need to be solved in perforation techniques, such as: preventing shale formation failure, damage of perforating guns, deposition of drill cuttings, providing security and high efficiency of perforation treatment. To face it, numerous innovations in modern perforators were introduced: advanced electronic detonation systems, new mechanical solutions of device, novel geometry and composition of cumulative charges. Improvements, which enable creation of clear perforations channels are also very important, as they result in increased wellbore productivity. For this purpose, there have been developed techniques, which use high pressure differences existing between the perforating, drilling and fluid systems during a few hundred milliseconds after the detonation. All mentioned solutions are presented in this paper.

DIFFERENTIAL SCANNING CALORIMETRY ANALYSIS OF BALTIC BASIN SHALES

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Key words:

DSC, shale gas, kerogen, organic matter maturity

29

Although methane contributes to increase of the greenhouse effect, the U.S. Energy Information Agency (EIA) reports that increase of natural gas recovery from shales caused the largest reduction of CO₂ emissions into atmosphere. According to the EIA report (Monthly Energy Review...) CO₂ emissions in 2013 in the United States reached the lowest level since 1992. This is critical information that could have a significant impact on CO₂ emissions especially in countries like Poland where energy is based on coal combustion. Methane, depending on geology and the location of the reservoir, constitutes usually more than 90% of the natural gas (Arthur, 2008). Other components are ethane, propane, liquid hydrocarbons, carbon dioxide, nitrogen, hydrogen, hydrogen sulfide and noble gases (argon, helium). Gas composition depends on the place of occurrence, geology of reservoir, depth, temperature, formation of kerogen and organic carbon (Ciechanowska, 2012). In order to evaluate a reservoir for the economic exploitation of shales, drilling cores are subjected to detailed petrophysical analysis which include:

- reservoir properties of rocks,
- sealing properties of rocks,
- thermal properties,

- mechanical properties,
- natural radioactivity.

Full analysis of cores is complex and time consuming. Therefore, most representative and simple analytical techniques have been proposed in recent years for the approximate characterization of gas-bearing shales. This poster presents results of differential scanning calorimetry analysis of Polish shales that attempts to assess the content of organic compounds and type of kerogen. The possibility of application of differential scanning calorimetry to a preliminary determination of combustible content in shale rock is considered.

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SHALE GEOCHEMISTRY OF SOURCE ROCKS IN LOKICHAR AND CHALBI BASINS, NORTHWESTERN KENYA RIFT SYSTEM

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Key words:

Lokichar, sedimentary rock, hydrocarbons, exploration, source rocks

30

The hydrocarbon potential of the subsurface sedimentary rock sequences of Lokichar (Tertiary) and Chalbi (Cretaceous), which has been interpreted using some selective geochemical analysis of rock samples in Lope-rot-1 Well (denoted as LT-1 Well) for their total organic carbon (TOC) and other sedimentological parameters in order to understand the essential structural features of the source-reservoir-seal rocks petroleum targets, form the premise of this paper. Understanding the subsurface structures and depositional environments conducive for hydrocarbon generation and trapping is essential as it

forms the basis for exploration rationale. The organic matter richness, facies type and degree of thermal alteration and/or maturation are factors useful in evaluating the potential source rocks. Rock-Eval pyrolysis has been employed on selected rock samples with high TOC for quantification of data, and this would help in precise identification of phases of hydrocarbon generation. Some lithologs from C1, C2 and C3 wells in the Chalbi basin were also examined using the seismic and gamma ray characteristics in order to characterise the shale-rich strata in which there was oil/gas shows.

APPLICATION OF SPECTROMETRIC METHODS IN CORE SAMPLING FOR LABORATORY TESTS



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Key words:

spectroscopy, gamma, XRF, sampling, core

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Typically, sampling is based on macroscopic observations of cores, which may lead to undifferentiated collection of samples. The poster presents two portable spectrometric devices that allow for immediate quantitative evaluation of the chemical composition and organic matter content. This specialized equipment can assist in the selection of appropriate samples for testing at the initial stage of sampling.

Gamma Logger uses the gamma ray spectroscopy method, which measures natural formation radioactivity. It allows for fast analysis of the concentration of the radioactive elements: Potassium (K^{40}), Uranium (U^{238}) and Thorium (Th^{232}); and helps to assess the volume of organic matter and clay content in the drilled rocks. To achieve high efficiency, the device uses bismuth germanate (BGO) detector ($Bi_4Ge_3O_{12}$), due to high atomic number of bismuth and its high density. The results of spectrometric gamma measurements are presented as four sets of graphs (logs)-total [CPS], potassium [%], uranium and thorium [ppm].

Energy Dispersive X-Ray Fluorescence Spectroscopy (EDXRF) is used to measure chemical composition of

the rocks. Portable XRF (Bruker co.) allows for instant and accurate surveys, which results are displayed in real time. Analysis gives information about: clay content, rock-fragmentability, as well as calcium and silica concentration. This method is also useful with the probe calibration. The range of analysis device is from Magnesium to Uranium, with the possible measurements of 45 elements. Portable XRF operates by analyzing the fundamental parameters, thus it does not need special standards of calibration. Data from device can be presented as graphs (logs) to compare with other analyses.

Presented methods can be used both on the rig site and core repository. The advantages are: rapid data collection, high accuracy, mobility and uncomplicated comparison with other measurements. Furthermore, presented methods are non-destructive and tests are executed directly on the drill core. To summarize, information obtained from Gamma Logger and Portable XRF are helpful during core sampling for laboratory testing. It allows for preliminary assessment of cores content and facilitates the choice of the most appropriate samples.

RAPID CHANGES IN REDOX CONDITIONS DURING THE EARLY SILURIAN IREVIKEN EVENT – AN EXAMPLE FROM THE DEEP SHELF SUCCESSION OF THE HOLY CROSS MOUNTAINS



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Key words:

Ireviken event, redox conditions, inorganic proxies, pyrite framboids, Silurian

32

The redox history of the Early Silurian, including the little-known Ireviken biotic crisis in the lowermost Wenlock (Scheinwoodian Stage), has been constrained through a multiproxy study of sedimentology, total organic carbon (TOC), total sulphur, inorganic proxies, and pyrite framboid distributions from the Wilków IG1 borehole from Poland. Sedimentological observations of the Llandovery/Wenlock (Telychian to Scheinwoodian) boundary reveal that hemipelagic sedimentation under an anoxic/euxinic water column was interrupted by low density bottom currents or detached diluted turbid layers that resulted in intermittent seafloor oxygenation. TOC values throughout the borehole section are in general accordance with the inorganic proxies, and reflect variable redox conditions. U/Th, V/Cr and V/(V+Ni) ratios, as well as Uauthig and Mo concentrations suggest that during the Ireviken event, bottom-water conditions deteriorated from oxic during the Telychian to mostly suboxic/anoxic immediately prior to the Llandovery/Wenlock boundary, before a brief reoxygenation at the end of the Ireviken event in the Scheinwoodian Stage. Follow-

ing the Ireviken event conditions once again became oxygen deficient, with the development of a euxinic zone in the water column.

Lower in the sequence, elevations in TOC content near the Aeronian/Telychian boundary, together with increased U/Th and V/(V+Ni) ratios and the occurrence of small pyrite framboids, are all consistent with the development of dysoxic/anoxic conditions. U/Mo ratios > 1 throughout much of the Aeronian and Telychian, together with an absence of pyrite framboids, suggests oxygenated conditions. Rapid fluctuations in U/Mo during the Ireviken event are characteristic of fluctuating redox conditions that culminated in an anoxic/euxinic seafloor in the Scheinwoodian stage. Pyrite framboid diameter results are in agreement with bulk and inorganic proxies, but the greatest correlation was noted between pyrite diameter results and the V/(V+Ni) ratio. Rapid fluctuations of the chemocline across the Llandovery/Wenlock boundary is likely to have been a major cause of the Ireviken extinctions, which affected mainly pelagic and hemipelagic fauna.

ACRITARCH ASSEMBLAGES IN CAMBRIAN MUDROCKS FROM THE HOLY CROSS MOUNTAINS (POLAND) AND THEIR STRATIGRAPHIC SIGNIFICANCE



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Key words:

acritarchs, Cambrian, Holy Cross Mts.

33

The Holy Cross Mts. (Central Poland) are one of the most important areas of Cambrian research in Europe. An almost complete sequence of Cambrian system (containing all four Series) is exposed on this small area. The Cambrian rocks are developed in almost exclusively siliciclastic facies, dominated by various mudrocks with not so common sandstones. The trilobites which are the basis of stratigraphy of the Cambrian in the Holy Cross Mts. are unevenly distributed and connected mainly with sandstone facies. Bulk of the Cambrian claystones and mudstones yields rich acritarch assemblages, which confirms, as well as in many periods complements trilobite biostratigraphic data.

The oldest acritarch assemblage, which has recently been recognized by the author in the south-west part of Holy Cross Mts., points to the presence of the Terreneuvian Serie. Those assemblages are dominated by *Leiosphaeridia* accompanied with *Comasphaeridium*, *Granomarginata* and *Pterospermella* with important taxon *Pulvinosphaeridium antiquum* which is characteristic for the oldest Cambrian series.

The younger acritarch assemblages (Cambrian Series II) are more diversified and numerous. They contain com-

mon specimens of *Skiagia*, *Heliosphaeridium* and many other acritarch taxa which are typical for this Series in many regions of the whole world and which allow to identify acritarch biozonation typical for this part of Cambrian profile. In the upper part of "lower" Cambrian profile in the Holy Cross Mts., stratigraphically very important taxa *Volkovia dentifera* and *Liepaina plana* appear.

The mudrocks of the III Series of Cambrian yield characteristic assemblages with *Eliasum Ilaniscum*, *Cristallinium cambriense* and in upper part of this sequence – *Tomofeevia* sp. Such acritarch assemblages are typical for all areas where "middle" Cambrian rocks are present. In Holy Cross Mts. it is especially important because trilobite data for them is extremely rare.

Furongian sequence, which is developed in the northern part of the region, is clearly divided. Lower part of "upper" Cambrian profile contains relatively few acritarch taxa with *Tomofeevia*, *Vulcanispharea* and simple "galeae" forms. "Middle" yields characteristic assemblage with first *Diacriodae* and *Ninadiacriodidium* whereas "upper" is extremely rich in "galeate", *Diacriodae* and other characteristic acritarchs.

ANALYSIS OF FACTORS IMPEDING IMPLEMENTATIONS OF THE MICROSEISMIC MONITORING

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Key words:

microseismic, monitoring, noise, signal

34

Microseismic monitoring is an emerging tool in the petroleum industry that enables better reservoir management, reducing risk of failure and allowing to increase maximum production from the well. In recent years a significant development of this technique could be observed. However, as demonstrated by the results of mea-

surements from many regions in the world, also from Poland, these surveys are often proved ineffective. That is why this paper focuses on factors impeding the implementation of the measurements, as well as state of art solutions.

UPPER FURONGIAN BLACK SHALE IN THE HOLY CROSS MOUNTAINS (POLAND): STRATIGRAPHY, INORGANIC GEOCHEMISTRY AND ACRITARCH ASSEMBLAGES

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Key words:

Furongian, Tremadocian, stratigraphy, shales, acritachs, geochemistry

35

The uppermost Cambrian in the northern Holy Cross Mountains (HCM) is represented by the Brzezinki Formation consisting largely of dark shales dated by trilobites of the *Acerocare sensu lato* and *Peltura scarabaeoides* zones (Żylińska, 2002) and graptolites of *Dictyonema* (*Rabdinopora* ?) species (Tomczyk, Turnau-Morawska, 1967). Moreover, this apparently monotonous succession yielded a great number of acritarchs displaying high taxonomic diversity. The microphytoplankton assemblage includes *Actinotodissus achrasii*, *Dasydiacrodium obsonum*, *Veryhachium mutabile*, *Acanthodiacrodium snookense*, *Arbusculidium* aff. *A. polypus*, *A. destombesii*, *Acanthodiacrodium* cf. *angustum* similar to communities recognized both on Gondwana and Baltica, and indicative for the Furongian and Tremadocian stratigraphic interval. Noteworthy, this palynomorphs reveal very close similarity to coeval assemblages from the East-European Craton and Newfoundland in Canada. The acritarch specimens display a high degree of thermal maturity indicative for condensate to gas window in the Amoco Standard Thermal Alteration Index.

The biostratigraphic data clearly show that the Brzezinki Formation represents a continuous deposition of fine-grained sediment across the Cambrian/Ordovician boundary, referred to the Scandinavian Alum Shales. This

lithologically monotonous mudshale formation contains subordinate carbonate and sandstone beds that seem to be an important marker horizons in sequence stratigraphical context. The redox-sensitive trace metal ratios (V/Cr, Ni/Co, U/Th) and TOC data from the Wilków IG 1 well indicate permanent oxic conditions during deposition of the Brzezinki Formation. However, the V/(Ni+V) indices display uniform and relatively high values suggesting dysoxic to anoxic environment. The geochemical data are consistent with sedimentary record of the Brzezinki Formation revealing decreasing upwards siltstone and sandstone beds, indicating intermitted deposition from bottom currents contributing to increase of oxygenation level at the sediment/water interface in largely hemipelagic setting.

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PROSPECTS OF PALEOZOIC ROCKS WITHIN THE EASTERN PART OF THE PODLASIE-BREST DEPRESSION ON SHALE GAS

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Key words:

Podlasie-Brest depression, Ordovician deposits, geochemical data

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The Podlasie-Brest sedimentary basin is a part of the Baltic-Dniester pericratonic area with developed subsidence on the western outskirts of the East European Platform. The Podlasie-Brest depression area is about 9900 km² within the eastern part of Belarus. According to drilling data, the sedimentary cover of the depression within Belarus varies from 557.2 m in borehole 6-K and to 1621.0 m in borehole 28-K, respectively, increasing in western direction towards the Teisseyre-Tornquist zone. The main part of the cross-section has been formed by Baikalian and Caledonian structural complexes deposits.

The objects of study were terrigenous rocks of Cambrian, carbonate and clay-carbonate rocks of the Ordovician and Silurian. According to the results of lithological and geochemical studies of core material more than 20 boreholes identified several intervals with the most promising parameters to search for shale gas.

The data of laboratory studies shown that the content of C_{org} is $\leq 1\%$, in the Cambrian deposits and T_{max} is 440°C; in Ordovician sediments – $C_{org} \leq 1-5.1\%$, T_{max} 435°C; in sediments of the Silurian – $C_{org} 1.52-5.1\%$, T_{max} 425°C.

Cambrian deposits are represented by interbedded siltstones, mudstones and sandstones. Several lithological

rock types have been identified by petrographic studies: massive crystalline limestone, partially dolomitic limestone, sometimes ferruginized, organogenic-clastic limestone, fine-grained carbonate-clayey rocks in the Ordovician and Silurian sediments. The trend of facies changes have been observed from more shallow to deeper facies.

Among negative factors could be noticed: low TOC, aquifers, low thickness of deposits, frequent breaks in sedimentation, the degree of transformation of OM.

In the result of analytical geological and geochemical studies have been determined that Ordovician deposits accumulated in marine basin of normal salinity in a reducing environment with significant input of organic matter in the sediment with favorable conditions for his burial. In our point of view, these deposits are the most promising at this stage of research the eastern part of Podlasie-Brest depression.

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ORGANIC MATTER MATURITY OF THE LOWER OLIGOCENE BLACK SHALES OF THE TARCĂU AND VRANCEA NAPPES FROM THE FLYSCH EASTERN CARPATHIANS, ROMANIA

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Key words:

Lower Oligocene, organic matter, maturity, Tarcău and Vrancea Nappes

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Throughout the Paratethys the Lower Oligocene sediments are organic matter-rich, of which Menilite Shales are important hydrocarbon source rocks widely studied in the Western Carpathians but significantly less in the Eastern Carpathians. The purpose of this study is to characterize maturity of the organic matter (OM) from the Eastern Carpathians basing on the bulk geochemical data and biomarker maturity parameters. The samples were collected in the selected sites of the E–W transect of the basin: the Vrancea Nappe (VN) and the Tarcău Nappe (TN) of the Carpathians.

Bulk geochemical data show that the TOC content is in the range of 0.5–7.9% (avg. 3.2%) in VN and 0.7–4.3% (avg. 1.8%) in TN. The TOC values vary significantly within each section, which is influenced by lithology and OM type. The investigated rocks could be considered as hydrocarbon source rocks with good hydrocarbon potential. According to T_{max} values, which decrease from ~429°C in inner TN to ~425°C in outer VN, and the C_{31}

homohopane (HH) $22S/(22S+22R)$ ratio oscillating around 0.33–0.54 (avg. 0.5) for both nappes, the rocks are approaching to the onset of oil generation window. For all the investigated settings the C_{30} moretane/(hopane+moretane) ratio is in the range of 0.1–0.26 indicating that for some samples (TN) the threshold of hydrocarbon generation (0.15) has been barely reached. In turn, trisnorhopane ratio, $Ts/(Ts+Tm)$ values in the range of 0.17–0.33, does not systematically vary with maturity but seems to be influenced by lithology and redox conditions.

The differences in the discussed parameters reflect distinct evolution of the individual Carpathian subbasins. Within the subbasins, maturity oscillates around the onset of hydrocarbon generation, and decreases from the internal nappe towards the nappe proximal to the foreland. This results from differences in burial/erosion and heat flows. The further research aiming to include more sections is in progress.

MICROBIOLOGY OF THE KUPFERSCHIEFER BLACK SHALE (FORE-SUDETIC MONOCLINE, SW POLAND)

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Key words:

Kupferschiefer, black shale, microorganisms, diversity, subsurface

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Lithosphere is the sphere of our planet which is inhabited by the largest number of diverse microorganisms. Particularly interesting environments for microbiological research are those under the earth surface. An example of such subsurface environment is polymetallic, *organic-rich* Kupferschiefer black shale. Although this environment seems to be unfriendly for microorganisms, the surface of the shale is colonized by dozens of bacterial strains.

Microorganisms isolated from the underground black shale belong to different taxonomic groups. The dominating representatives of culturable microorganisms are Gram-negative Proteobacteria, particularly γ -Proteobacteria – *Pseudomonas* spp., *Acinetobacter* spp., *Psychrobacter* spp., *Stenotrophomonas* spp. The representatives of other classes of this phylum – primarily *Brevundimonas* spp., *Paracoccus* spp., *Sinorhizobium* spp., *Ochrobactrum* spp., *Sphingobium* spp., *Sphingomonas* sp. (α -Proteobacteria) and *Alcaligenes* spp. (β -Proteobacteria) also occur. In addition, the black shale is inhabited by bacteria belonging to other phyla such as Bacteroidetes (*Sphingobacterium* spp.) as well as Gram-positive Actinobacteria (*Microbacterium* spp., *Arthrobacter* spp., *Amycolatopsis* sp., *Micrococcus* sp.) and Firmicutes (*Bacillus* spp.).

Bacteria colonizing the black shale represent different physiological groups. These are mainly aerobic microorganisms but facultative anaerobes also occur. Bacteria inhabiting the black shale are mesophililes and neutrophiles. However, most of them tolerate a wide range of physicochemical conditions. A very interesting particular feature of indigenous microorganisms is high resistance to heavy metals. The majority of the isolated bacteria is organoheterotrophs capable of degrading fossil organic matter present in the rock. Some of them, however, are facultative chemolithotrophs oxidizing reduced Fe and S compounds, as well as As, Mn, Se and V. Indigenous bacteria are also able to use oxidized compounds of S, Fe, Se, Mn as electron acceptors. From the surface of the shale we isolated strictly anaerobic sulfate and iron reducing consortia of microorganisms as well as bacteria capable of degrading aromatic hydrocarbons.

An interesting issue associated closely with the research in indigenous microorganisms colonizing the Kupferschiefer is their origin, adaptation to extreme conditions, and their role in shaping the underground deposit in the past, as well as in the ongoing processes of rock transformation.

BIOMARKER PALEOENVIRONMENTAL PROXIES OF THE UNSTUDIED LOWER OLIGOCENE BLACK SHALE SECTIONS FROM THE SILESIAN AND SUBSILESIAN UNITS (EASTERN PART OF POLISH OUTER CARPATHIANS)

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Key words:

biomarkers, Oligocene, black shales, Parathetys, Outer Carpathians

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In the Outer Carpathians, so-called "menilite" facies are common in all tectonic units and are considered as main source rocks for the Carpathian oils, therefore a number of studies were dedicated to their hydrocarbon potential, maturity and importance for the Carpathian petroleum systems.

During the Lower Oligocene a new regime in the Carpathian domain of the Parathetys was established leading to high bioproductivity associated with formation of oxygen depleted zone resulting in good preservation of organic matter (OM) in fine grained sediments. In this preliminary study an attempt was made to characterize major OM sources, paleoenvironment conditions and thermal maturity based on biomarkers distribution in the Lower Oligocene black shales of the Menilite Fm.

Representative samples for the studied area (E part of the Silesian and Subsilesian unit) were collected from selected outcrops and analysed to assess mineral composition (XRD), total organic carbon (TOC) and biomarkers using GC-MS after standard EOM fraction separation procedures.

The analysed shales consist mainly of quartz and micas with accessory pyrite and often calcite, and are all OM rich (3.0–4.6% TOC). Co-occurrence of different groups of biomarkers (e.g. acyclic isoprenoids, chromans, hopanes,

steranes as well as aromatic sesqui- and diterpenoids) indicates heterogeneity of the OM source. Variations in the observed hopane/sterane ratios show significant differences in the contribution from prokaryotes and eukaryotes to kerogen. Generally, prokaryotic OM prevails, however, in few samples eukaryotic OM is dominant. The presence of highly branched isoprenoid (C₂₅ HBI) shows that diatoms played significant role in the phytoplankton community contributing to the sedimentary OM. The occurrence of biomarkers characteristic for vascular land plants (cadalene and retene groups) indicates supply of terrigenous OM. The distribution of *n*-alkanes, pristane, phytane and homohopanes in some samples suggests development of anoxic conditions during sedimentation/diagenesis. Application of chromans to assess paleosalinity seems to be doubtful for the analysed samples. There are no premises for photic zone anoxia in water column (no isorenieratene derivatives), but sediment (or bottom water) anoxia/dysoxia is suggested by the presence of pyrite in all samples. Assessment of the thermal maturity based on hopane isomerisation (22S/(22S+22R) and $\beta\alpha/\alpha\beta$ ratios) reveals immature/very low mature OM.

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MOBILIZATION OF ORGANIC COMPOUNDS FROM SHALES DURING SHALE GAS EXTRACTION

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Key words:

shale, water extraction, mobility

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Using a combination of horizontal drilling techniques and hydraulic fracturing, natural gas is now economically recoverable from shales. Large volumes of water return to the surface after the fracturing procedure, which contain variable and generally high concentrations of organic and inorganic chemical species and constitute a potential source of pollution if not disposed of responsibly and according to strict regulations. Different hydrocarbons (e.g. PAHs, phenols, fatty acids) have been reported in the flowback water from both Marcellus and New Albany shales (Orem *et al.*, 2014) with compositions noted as being dependent upon the composition of the shale, its pressure and temperature environment, and the chemistry of the fluid used to do the fracking.

Here we report upon the chemical composition of dissolved species released from the water extraction of shales. Shale samples were selected from different geological settings, covering a broad range of shale compositions and a maturity range from immature to overmature. The samples were extracted with distilled water for 48 hours at a temperature of 100°C and ambient pressure. The water soluble fractions were analyzed by different chromatographic methods (LC-OCD, IC). Humic substances, low molecular weight organic acids (LMWOAs)

and low molecular weight neutral compounds comprise three important fractions of the dissolved organic carbon (DOC) that have been detected in the leachates using size-exclusion chromatography (SEC). The DOC released from the shale samples show quite different proportions within the SEC fractions. In the case of the Bakken and Posidonia shale, the oxygen index and maturity of kerogen were demonstrated to strongly influence the concentration of the LMWOAs, thereby reflecting a direct link between the different structures of kerogen within the sample set and the chemical species released in aqueous solution. For samples from the Posidonia Shale, the concentration of released even-numbered carboxylic acids decreased with progressive maturation up to peak oil window ($R_m = 0.88\%$) and reserved afterwards. This V-shaped trend positively correlates with the porosity which is available for storing gas. Thus, the types and concentration of extraction products reported here provide information not only on potential environmental loadings, but also on the chemical and physical properties of the shales.

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