

Post-Doc Research Proposal 2016

Advanced geochemical characterization of unconventional shale resource systems: Application to the new Rock-Eval Shale Play method coupled with mass spectrometer and TD-GC-MS/FID analysis to better evaluate the reservoir quality.

proposed by

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INTRODUCTION - CONTEXT

During the last decade, unconventional shale plays have become important since unexpelled gas and oil from source rock intervals can now be produced thanks to hydraulic fracturing and horizontal drilling, which enhances natural bulk rock permeability over substantial play areas. However, the potential resources of these unconventional petroleum systems are still poorly quantified because the estimates of recoverable oil and gas reserves remain difficult to calculate by the existing analytical techniques and numerical models. As shale plays are considered simultaneously both source and reservoir rocks, it is necessary to describe (1) the organic porous network (nature and distribution of pore structures in organic matter), (2) the gas and oil trapping mechanisms and (3) the hydrocarbon composition, quality and quantity in these shale samples (e.g. Romero-Sarmiento et al., 2013; 2014; 2015).

A new IFPEN Rock-Eval method for characterizing unconventional shale resource systems has recently been developed and patented by IFPEN: the Shale Play method (Romero-Sarmiento et al., 2015; Pillot et al., 2014). This new pyrolysis program provides a better assessment of free or sorbed hydrocarbons still present within these source and reservoir-rock samples, conventionally captured as the S1 peak hydrocarbon yield during the standard pyrolysis cycle. It also provides a more accurate determination of the Rock-Eval T_{max} thermal maturity parameter for samples still impregnated by hydrocarbons. A fundamental improvement of the new Rock-Eval method is that it separates the conventional S1 peak into two sub-peaks (Sh0, Sh1), each representing a distinct pool of organic compounds with unique thermal reactivity and chemical composition.

For a correct economic assessment of unconventional resources, the quantification of the oil and gas *in place*, within both source and reservoir rocks, is essential. A quick look determination of hydrocarbons in place in unconventional shale resource systems can be made using the proposed methodology by Downey et al. (2011). Following thus the conversion recently proposed by Romero-Sarmiento et al. (2015) (Figure 1), which considers the new Rock-Eval Shale Play parameters (Sh0 & Sh1), it is now possible to compute an early and more accurate quantity of the original oil- in- place (OOIP).

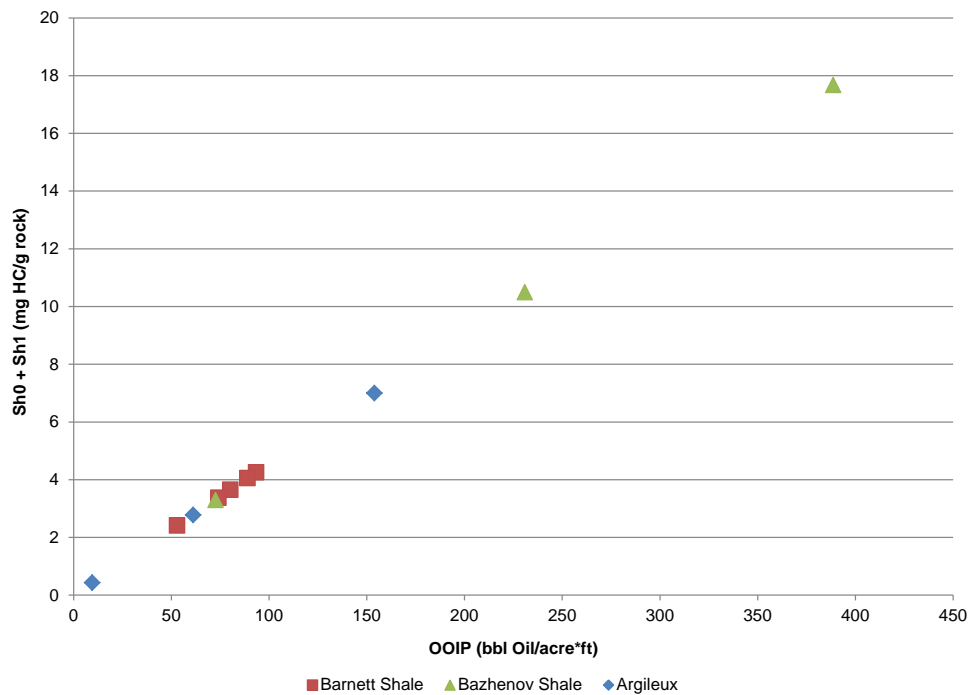


Figure 1: Conversion of Sh0 + Sh1 peaks to Oil (Romero-Sarmiento et al., 2015)

The new Shale Play method was mainly developed and tested on several samples from Bazhenov Shale (Russia), Barnett Shale (USA), Toarcian Shale (France), Cretaceous Shale (Jordan), Silurian Shale (Algeria) and Triassic Formation (Canada) with Rock-Eval T_{max} values varying between 410 and 470 °C. It means that the proposed pyrolysis program has only been validated for samples showing a thermal maturity of organic matter from immature to the end of the oil window (Romero-Sarmiento et al., 2015). In this post doc project, we therefore propose:

- (1) to investigate more organic-rich samples worldwide from oil-window thermal maturity in order to refine and expand the range of applications of the Rock-Eval Shale Play method.
- (2) to feed the IFPEN Geochim Database with new data specific to unconventional resources.
- (3) to evaluate the hydrocarbon composition detected by the two first acquired Rock-Eval Shale Play parameters (Sh0 & Sh1) on organic-rich source rocks.
- (4) to compare the obtained results with production data to better estimate the hydrocarbon reservoir quality at the basin scale.

One of the main objectives of the current post doc project is to provide useful information to understand the mechanisms of hydrocarbons generation / production from unconventional source rock intervals (IFPEN Project Unconventional Resources MGH) to improve analytical devices and numerical modeling tools (IFPEN Project Rock-Eval and TemisFlow).

EXPERIMENTAL PROGRAM

The challenge is now to determine more specifically the molecular composition of organic compounds related to the first two peaks (Sh0, the low-molecular weight thermovaporized hydrocarbons and Sh1, the high-molecular weight thermovaporized hydrocarbons) obtained during the improved temperature thermovaporization program, on one hand and the trapping mode of the hydrocarbon content relative to the rock (free, adsorbed or sorbed), on the other hand.

With this aim, we propose the following analytical program:

- (1) Thermovaporization experiments on rock samples will be performed to characterize the composition of hydrocarbons released by each Rock-Eval Shale Play peak (Figure 2). An analytical design to couple our Rock-Eval 6 device with a continuous flow mass spectrometer (MS) will be set up and validated, in order to give compositional data on the C₁ – C₅ fraction of the thermovaporized fluids.
- (2) Molecular characterization will be made on hydrocarbons thermally desorbed with respect to the two first Rock-Eval Shale Play parameters following the procedures described in Sanei et al. (2015) and Romero-Sarmiento et al. (*submitted*). Thermal desorption coupled with gas chromatography-mass spectrometry/flame ionization detection (TD-GC-MS/FID) analyses will be performed, in support with the **Geological Survey of Canada**, at 100°C, 200°C and 350°C to compare the composition of hydrocarbons thermally desorbed with the thermovaporized hydrocarbons (Figure 2).
- (3) Finally, we will define the initial gaseous hydrocarbon composition of the studied samples using a novel vacuum crushing technique developed in house. The isotopic compositions of the hydrocarbons will be analyzed as well, in order to examine the isotopic evolution of trapped fluids with thermal maturation, and trapping mechanisms. This approach will allow to define a good compositional reference for the Rock-Eval analysis to be compared with, and make robust mass balance calculations of the thermovaporization process.

The proposed analytical workflow will revolutionize the application of Rock-Eval pyrolysis® as it will enable to determine the chemical composition of vaporized/pyrolyzed carbon as well as establish the hydrocarbon trapping mode (free, adsorbed or sorbed ?) in the rock.

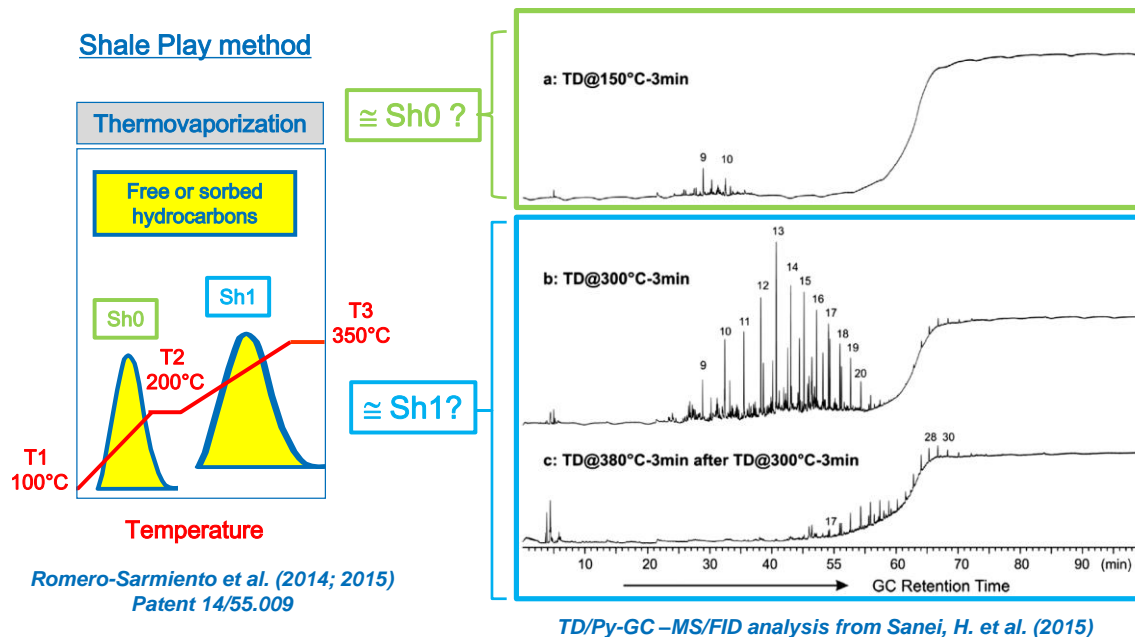


Figure 2: Rock-Eval Shale Play parameters by Romero-Sarmiento et al. (2014; 2015; Patent 14/55.009) versus the composition of hydrocarbon thermally desorbed from the rock sample by Sanei et al. (2015)

To complete the source rock investigations, a detailed organic geochemical and physical analytical workflow (including improved Rock-Eval, organic petrology and biomarker analysis, porosity, permeability, pore throat, carbon isotope measurements) can be also conducted in scientific collaboration between the IFPEN and the Geological Survey of Canada. Rock-Eval Shale Play analysis assessing the unconventional source potential of the investigated target, and improved organic petrology will complement the previously mentioned techniques in assessing the depositional environment, the thermal maturity, and the kerogen type of source rocks.

THE CASE STUDY

For the first time, the entire proposed analytical program here will be tested on source rock samples from the Goddard Formation in the South Central Oklahoma Oil Province (USA). These organic-rich samples will be provided by the oil company Vitruvian (contact person Frederic Gallice; frederic.gallice@vexpl.com).

RESULTS

- (1) Test and validation of the analytical design coupling the Rock-Eval 6 device with a continuous flow mass spectrometer.
- (2) Molecular composition of hydrocarbons thermally thermovaporized and desorbed during the Sh0 and Sh1 peak temperature ranges.

- (3) A quantitative description of the distribution of hydrocarbons among the various trapping modes available in shale formations (free, adsorbed or sorbed) at the sample scale.
- (4) A reference case study for geochemical characterization of unconventional shale resource systems.

DELIVERABLES

The Post Doc student will be able to provide a scientific manuscript as well as a technical report as follow:

Article 1: "Molecular composition of hydrocarbons still present in reservoir source-rock samples from South Central Oklahoma Oil Province (USA)"

IFPEN technical report: "Coupling of the Rock-Eval 6 with a continuous flow quadrupole mass spectrometer for the quantification of C₁-C₁₄ *n*-alkanes extracted by open-system pyrolysis. Design, validation and first case application on shale play samples"

DURATION (12 months)

Months 1-2: Bibliographic work – sample collection and preparation – experimental setup design

Month 3: Initiation of a set of experiments

Months 4-6: Development and validation of analytical procedures

Months 7-12: Experiments, tests, interpretation of results, writing scientific paper/report.

Laboratories: IFP Energies Nouvelles petrophysical and geochemistry department (Rueil-Malmaison) and the Geological Survey of Canada (Calgary).

This project will be also a great opportunity to expand the scientific collaborations between the Geosciences division (R16) and the Geological Survey of Canada (Calgary) in organic geochemistry domains.

CANDIDATE PROFILE

Previous experience in geosciences and/or geochemistry

Interest in analytical design

Critical thinking, scientific creativity, and synthesis skills

Good knowledge over the petroleum systems and organic geochemistry.

PhD's degree or equivalent in Geology, Petroleum Geochemistry.

ADVISOR: (IFPEN)

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This Post Doc Project will be carry out in collaboration with the Geological Survey of Canada

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This Post Doc Project will be also followed by Dr. Valérie Beaumont and Dr. Virgile Rouchon (IFP Energies Nouvelles).

This Post Doc Project will be integrated to the IFPEN Project MGH36 Shale Play (Manager: Dr. William Sassi)

SELECTED REFERENCES

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