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# An application of microscopic analyses in shale gas rock description

## Introduction

As a standard procedure in the analyses of shale rocks being reservoirs of shale gas in order to determine the type of minerals contained in examined rock and their percentage contents the XRD analysis is made (quantitative-qualitative analysis). It is a relatively inexpensive and quick method. As a result, a mineral composition of examined rock is obtained. It is indispensable to determine the brittleness of the shale. The XRD analysis also enables determination of type and percentage of argillaceous minerals content in examined rocks based on selected minerals, which allows to draw conclusions concerning the reaction of the rocks in contact with fracturing fluids. The influence of the fracturing fluids on analyzed rock can also be assessed on the basis of CST (capillary suction time) analysis; however it will not provide information concerning the mineral composition of the rock.

Although the XRD analysis provides information about mineralogical content of analyzed rocks, it does not say anything about texture, structure and distribution of particular minerals in the rock. It is not known whether a particular mineral appears in the form of detritic grains or if it is only a component of cement or if it appears in both forms. The petrographic analyses provide solution to the problem. It is one of testing methods which can be defined as microscopic examination (by microscope examination we understand all the analyses executed with the aid of petrograpfic microscope, supported by computer analysis of microscopic images and cathodoluminescence).

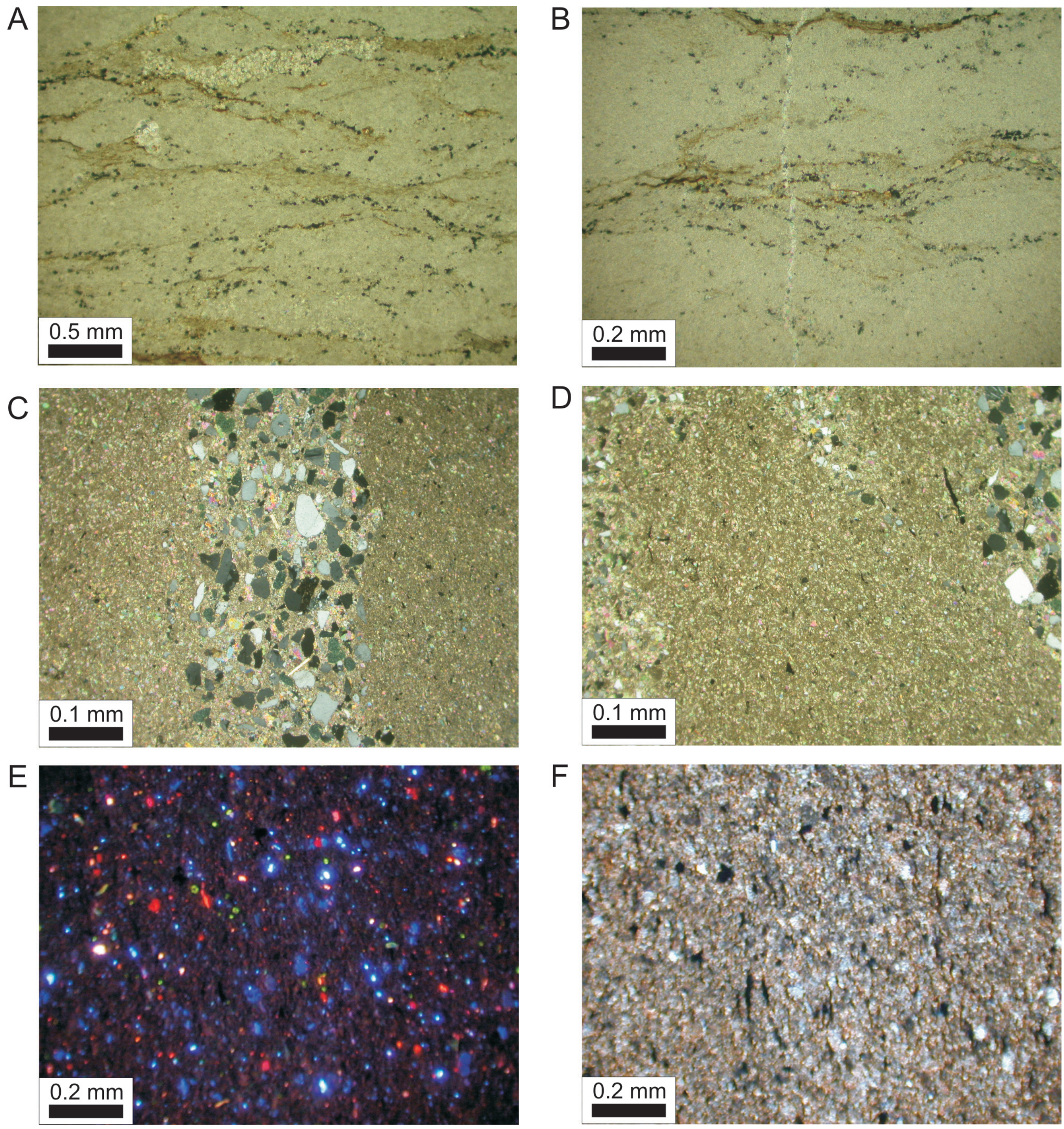
## Sample preparation

In contemporary microscopic analyses it is very important for the thin sections to be prepared properly. In oil companies this issue was solved already in the 90s of the 20th century. Special methodologies were elaborated and they are applied in microscopic examination to suit the requirements of the oil geology. The samples should be prepared in such a way that it would be possible to use them both in the classical petrographic analyses as well as in cathodoluminescence, computer analysis of microscopic images and in SEM. The rock sample should be vacuum saturated with thermally resistant resin (resistance to heating up in cathodoluminescence and in SEM) and tinted with blue dye. Next, a thin section is prepared from the sample. The blue dye added to the resin allows the observation of the pore space in the rock. The application of thermally resistant resin permits observation of cathodoluminescence and application of the thin sections in SEM examination. While preparing samples for microfracturing analysis, however, yellow dye should be used instead of the blue one (observation is executed both in the transmitted light as well as in fluorescence).

## Petrographic analyses

The basic task of petrographic analyses is to determine mineral composition and texture (the layout and distribution of mineral components as well as the extent of filling the rock space) and structure (size and shape of grains

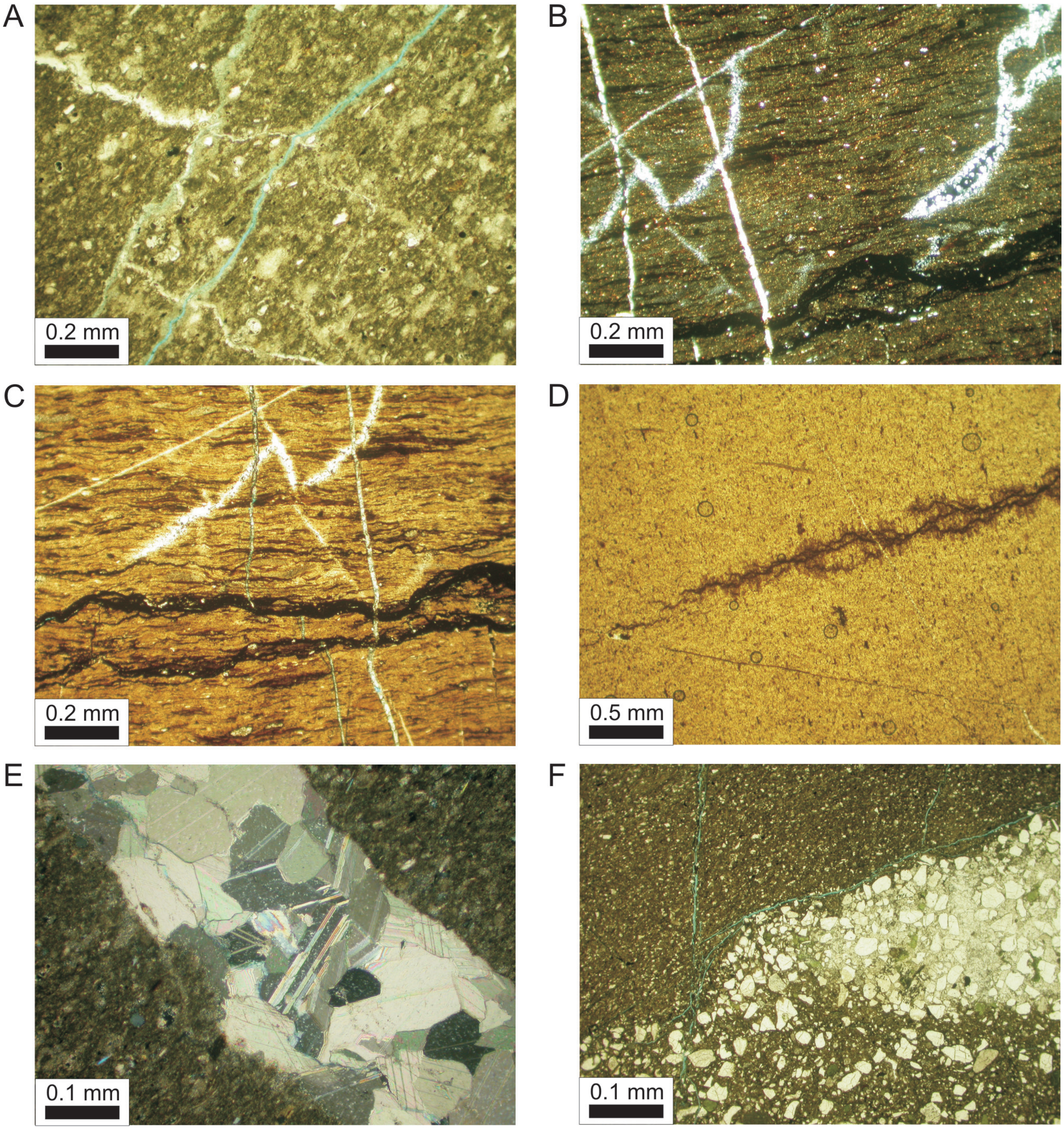
# PLATE I




**Photomicrographs of thin sections**

A. Argillaceous mudstone shale. Sedimentary microstructures highlighted with iron hydroxide and pyrite lamination.
B. Argillaceous mudstone shale. Sedimentary microstructures highlighted with iron hydroxides and pyrite lamination. In central part a micro fracture can be seen, filled with calcspar cutting through the lamination.
C. Argillaceous mudstone shale with silt shale lamines. On the right – higher content of clayey minerals. On the left – higher content of mudstone fraction.
D. Argillaceous mudstone shale with silt shale lamines.
E. Carbonate argillaceous mudstone. Photography in cathodoluminescence – visible grains of feldspar, plagioclases and quartz calcspar.
F. Carbonate argillaceous shale. In the rock matrix – single detritic grains visible, difficult to identify.

# PLATE II




**Photomicrographs of thin sections**

A. Carbonate-argillaceous mudstone. Rock cut through by microfracture net. Some microfractures are filled with calcite, some are open (blue color).
B. Silt-argillaceous shale, laminated with organic matter. Rock cut through by microfractures net filled with quartz. On the right there is a cavern created by circulation of solutions, currently filled up with quartz crystals.
C. Argillaceous shale, lamination highlighted with organic matter. Rock cut through by system of microfractures filled with calcite. Some microfractures are open (blue color).
D. Argillaceous mudstone shale cut through by two systems of microfractures. One is filled up with calcite and the other one is filled with hydrocarbons.
E. Argillaceous shale cut through by fracture filled up with thick calcite crystals. Inside the fracture, a secondary fracture can be seen cutting through the calcite crystals.
F. Mudstone-argillaceous shale with lamination of silt shale. A system of open microfractures (blue color) can be seen cutting through the rock.

and features of their surfaces) of analyzed rocks. Shale gas rocks are usually silt, mud and argillaceous shale. Very often there are alternate layers of all these types of shale or predominance of just one type (Plate I – C, D; Plate II – F). The objective of the petrographic analyses is to determine which type of shale there is and which predominates in a particular layer. It also provides crucial information for sedimentologists, who determine sedimentation microstructures, lamination types appearing in shale and relate them to particular mineral phases (Plate I – A-D; Plate II – B, D, E).

The principal objective of petrographic analyses is to determine mineral composition, distribution of detritic grains and types of cement appearing in analyzed rocks. Due to small dimensions, cathodoluminescence is helpful in the analysis of detritic grains (Plate I – E, F), the findings of cathodoluminescence in relation with computer analysis of microscopic images allow to determine very precisely the mineral composition of detritic grains. Another very important task is to determine diagenesis processes noticed in analyzed rocks and relating them to subsequent stages of geological development of the analyzed basin. The processes of diagenesis which brought improvement as well as those resulting in reduction of reservoir properties should also be examined.

In the analysis of cements the cathodoluminescence method will also be helpful as it allows to determine precisely the materials which constitute the cements even if their size is very small. In order to determine quantitative content, the cathodoluminescence may be combined with computer analysis of microscopic images. Microscopic observations may also be corelated with observations in SEM (chemical element distribution maps).

The application of thin sections with tinted resin permits the observation of the pore space in the analyzed rock. Such observations allow to corelate the pore space with the processes of diagenesis, appearance of detritic grains, organogenic clasts or cements. According to Loucks et al. [3], the pore space in shale is related to pores between grains, in the grains, in matrix rock and mixed pores. In optical microscopy the first two types of pore space can be observed. Due to computer analysis of microscopic images it is possible to determine mathematic parameters of pore space in the rock (taking measurements of interesting parameters of particular pores), to evaluate the connection of single pores and prepare numerical data for modeling the pore space.

## The micro-fracturing analysis

The microscopic analyses also allows to observe the microfractures which appear in examined rock. In planning the fracturing treatments it is very important to determine the natural microfracture net and its layout in rock. It is also very important to determine whether the microfractures are open or filled with e.g. calcite or quartz. Microscopic observations are also helpful in precise determination which microfractures are natural and which appeared as a result of the so-called 'human factor' (pulling out the core, rapid drying of a sample). In order to minimize the human factor on samples it is recommended to dry samples in a relatively low temperature up to 40–45ºC. Then the samples are saturated (5 × 5 cm) with resin and fluorescent dye in vacuum. Out of such sample a cube is cut out with side measuring 4 cm and thin sections, polished in directions $X$, $Y$, $Z$ are made. The thin sections allow observations both in the transmitted light as well as in fluorescence. By observations in the transmitted light is possible to determine if the microfractures are natural and what types of minerals are filling them, provided they are filled up (Plate I – B, Plate II – A-F). Observations in fluorescence allow to determine which microfractures are open and which are closed and help to take measurements [1, 2, 4, 5, 6] to determine porosity and microfracture permeability.

The major issue is to determine if the microfractures are natural or not. When the microfractures are filled, there is no such problem. However, it is important to know if they are open despite being filled and if their opening is natural or not. In this case, very helpful is observation of the direction of the open microfracture in fluorescent light and the control of its direction and opening in the transmitted light. It should be observed if the opening of the microfracture runs along the crystal intergrowth, which would suggest artificial opening or whether it cuts it through, which suggests its natural origin. In case of open and not filled microfractures the case is more difficult. Facing such a problem, it should be noted if the microfracture cuts through the detritic grains, laminations or if it runs along, how the cements are cut through by the fracture behave, whether a microfault can be observed along it and to follow closely its route looking for the filling and crystallization traces on its edges. Such observations permit quite precisely to determine which microfracture is natural and which is artificial. Knowing it, we can proceed to calculating porosity and micro-fractural permeability.

The information obtained from microscopic analyses broadens considerably our knowledge concerning analyzed rocks and it is used in sedimentology, facies analyses, sedimentation basin development analyses, assessment of reservoir properties, hydrocarbon migration paths as well as in planning the fracturing operations.

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