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About the Chemico-Mineralogical Characteristics of the Rocks from a Natural Gas Deposit in North-Western Romania

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Abstract*: Identifying rock types and establishing their chemico-mineralogical composition are essential components of geological characterization process of hydrocarbon reservoirs. Based on the global chemical-mineralogical composition and rock type, recipes and treatments are developed aimed to prevent the blocking of productive formations both during drilling and during the production of wells. Knowing the detailed lithology helps us in the optimum choice of drilling fluids compatible with the formations on the investigated structure and also to choosing recipes during acidizing/stimulation treatment of the wells. The complex geological investigations (petrographic study, EDX, XRD) performed on cores/sample rocks allow through the information provided a better characterization of the rocks in a reservoir and the efficient modelling of the hydrocarbon reservoir production. In the paper is presented a detailed description based on macroscopic and microscopic petrographic investigations, EDX spectrometry and X-rays diffraction of several drill cuttings collected in the wells spudded in a gas field in north-western Romania (Pannonian Basin).*

Keywords: *Pannonian Basin, Neogene formations, EDX, XRD, geological characterization*

1. Introduction

A key element in the geological modelling of hydrocarbon reservoirs is identifying of the rock types in the reservoir. Identifying rock types and establishing their chemico-mineralogical composition are important components of geological characterization process of hydrocarbon reservoirs. The paper presents a detailed description based on macroscopic and microscopic petrographic researches, EDX analysis and X-rays diffraction of several drill cuttings collected in the wells spudded in the Moftinu gas structure (North-Eastern Pannonian Basin). It was pursued a detailed characterization of the samples studied in terms of petrographic types, composition and microtexture, because as it is well known that such details cannot be provided by geophysical investigations.

The gas field studied in the paper is located in the North-Eastern Pannonian Basin and in Carei sub-basin (graben) respectively. The reservoir was discovered in 1970 by Moftinu-950 well, which tested gas in Pliocene with a maximum gas flow rate of 11000 m^3 / day under test, the gas flow rate decreasing later to 5000 m^3 / day (the gas flow being non-commercial at that time). Later, in Early 2012, was spudded Moftinu-1000 well that encountered two hydrocarbon-bearing zones for which production tests were carried, the two zones having a combined flow rate of 51000 m^3 / day. The drilling was resumed by the Serinus Energy/Winstar company at the End of 2014 by the Moftinu-1001 well (and later Moftinu-1002bis well) which were discovered commercial gas flow rate (approx. 200000 m^3 / day in production tests) and confirmed good exploration potential of Satu Mare oil concession. Currently, three production wells (Moftinu-1003, Moftinu-1004, and Moftinu-1007) are tied-in and brought in production and flow through the Moftinu Gas Plant with 450000 m^3 / day nominal capacity [1-3].

Pannonian Basin is a back-arc basin with a complex tectonic history, characterized by a system of Neogene extensional rift and strike-slip basins lying in the Carpathian central belt. The rifted sub-basins are separated by uplifted basement blocks but linked by a comprehensive post-rift sedimentary cover of Neogene and Quaternary formations.

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The Neogene back-arc extensional basin started an evolution in Middle Miocene during the final stages of Carpathians thrusting. Syn-rift sediments were deposited in a setting of grabens, half grabens, pull apart and shear basins. Deposition initially limited to the marginal areas (as is the area of the investigated field) became widespread in the geological time. Overall Pannonian Basin consists of a series of smaller extension basins bordered by uplifts. The uplifts began to form during the extension phase in the Middle Miocene and were filled up during sin-rift and post-rift stages. In Romania, along the eastern edge of the Pannonian Basin, in the West and the North of the Apuseni Mountains, there are four smaller sub-basins (Figure 1) named from North to South: Carei, Abramut, Socodor and Tomnatec. [2-7]

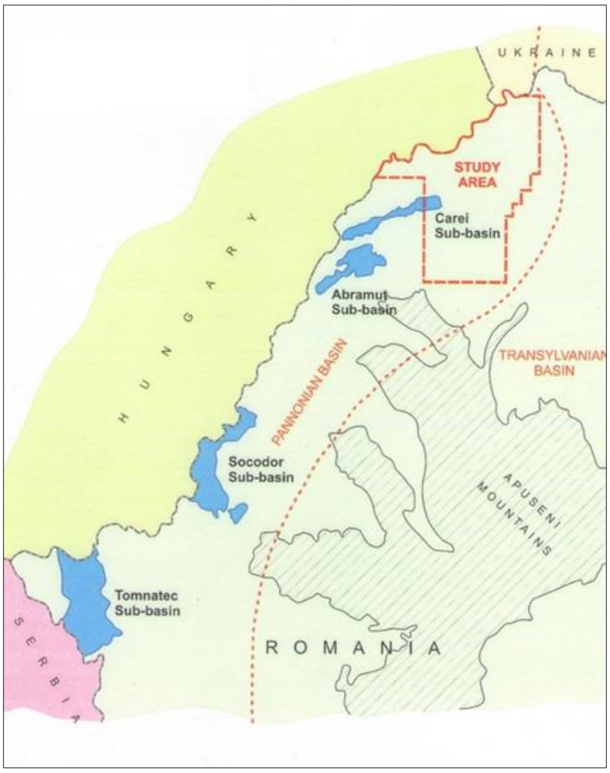

Figure 1. Pannonian sub-basins in Western Romania [3]

Carei graben, to which the gas field investigated belongs, is a Miocene rift bordered by Faget fault and has significant depth. The edge of the graben is characterized by coarse deposits (conglomerates and sandstones) while the fine-grained sediments characterized the inner part of the basin. Carei sub-basin is asymmetric, having slightly inclined south flank and steep north flank. It reaches a maximum thickness of about 3600 m and has a thick sequence of Miocene and Pliocene deposits, creating a proper context for the hydrocarbons generation. In Carei sub-basin, there are several normal faults and tilted half grabens (Figure 2). Between Moftinu uplift and Nisipeni uplift, there is a wide depression in which there are several smaller structural uplifts bordered by a normal fault. These were investigated by drillings but didn't confirm the existence of hydrocarbons. From south to north, there are three prominent heights, namely Nusfalau, Moftinu and Nisipeni. The structure plunges to the north in Babesti area. In this area, there are characteristics of smaller structures, like the one in the Babesti 2805 well, which confirmed the existence of gas in Miocene. Some key attributes of Carei sub-basins are as follows: (1) Pre-Tertiary sub-sections consist of Paleogene-Senonian rocks, belonging to Szolnok sequence (flysch suite composed of sandstones, clay shales, marlstones and bioherms/limestones) in north flank and Paleozoic metamorphic basement in the south side; (2) Miocene sediments are overlying the upper part of the basement. Pliocene sediments cover the entire sub-basin. The thickness of the Pliocene sedimentary sequence varies depending on the Pre-Tertiary morphology, but the sediments are generally conformable [2-8].

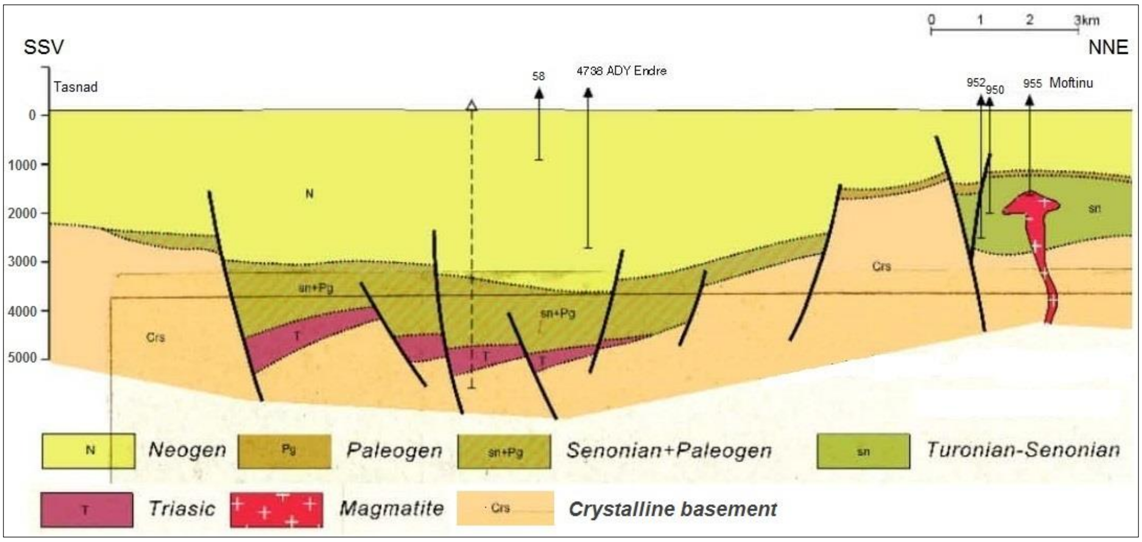

Figure 2. The geological section in the Carei sub-basin [3]

Moftinu gas field lies on the steep northern flank, faulted, of Carei Graben of northeast-southwest orientation. Moftinu field is dependent on closure up against the main Moftinu fault with south-dipping. Structurally, in Pliocene and Miocene formations, the Moftinu field is characterized by a three-way closure on the footwall of the Moftinu fault. The geological setting is changing rapidly near the Moftinu fault, which seems to be the result of a flower structure (Figure 3) which in returns resulting in rapid geological offset. To the northeast, the reservoir is affected by a transversal fault (Crasna fault), which strikes parallel to shoreline trend, and determines a partial four-way closure along the western edge. The wells spudded in the Moftinu structure crossed geological formations belonging to Pliocene, Miocene, Paleogene, and rarely to Pre-tertiary basement, fractured and weathered (suite of magmatic, meta-morphic and sedimentary rocks of Precambrian, Paleozoic and Mesozoic age). The recent drillings executed in the studied area shows that productive reservoirs consist of a suite of vertically stacked interbedded sandstones and shales sequences. These suggest a prograding fluvial-deltaic environment where fluvial sands usually cover and replace underlying marine shoreface sands. Correlation and interpretation of lithostratigraphic, micropaleontological and geophysical data obtained in the drillings executed led to the identification of several sand reservoirs labelled A1, A2, A2.2 (Mid to Late Miocene), and A3, B1, C1 (Pliocene), possible hydrocarbon-bearing (Figure 3) [1-6].

2. Materials and methods

The drill cuttings samples used in the study was collected during the drilling of the X1 and X2 wells in the Moftinu gas field (Satu Mare district, Romania). Drill cuttings were washed with water over a set of sieves in order to clean the material from mud contamination, then were dried and packed in self-sealing plastic bags. Every sample bag was labeled with well name or number and depth interval.

Chemical elemental analysis of the samples was performed by SEM/EDX using a Hitachi S3400-N microscope equipped with an Oxford Instruments X-act energy dispersive X-ray (EDX) spectrometer belonging to the Petroleum-Gas University of Ploiesti, operated using the Inca software package. The generated spectra were determined and analyzed by an EDX spectrometer, with 125 eV resolutions, and active detector area 10 mm^2. During tests, the working conditions were kept stable in order to minimize any effect on the statistical nature of the production of radiation.

For establishing rock types were manufactured thin sections of each sample and were performed mineralogo-petrographical investigations in the polarizing optical microscope Leica type in the Mineralogy-Petrology Laboratory from the Petroleum-Gas University of Ploiesti.

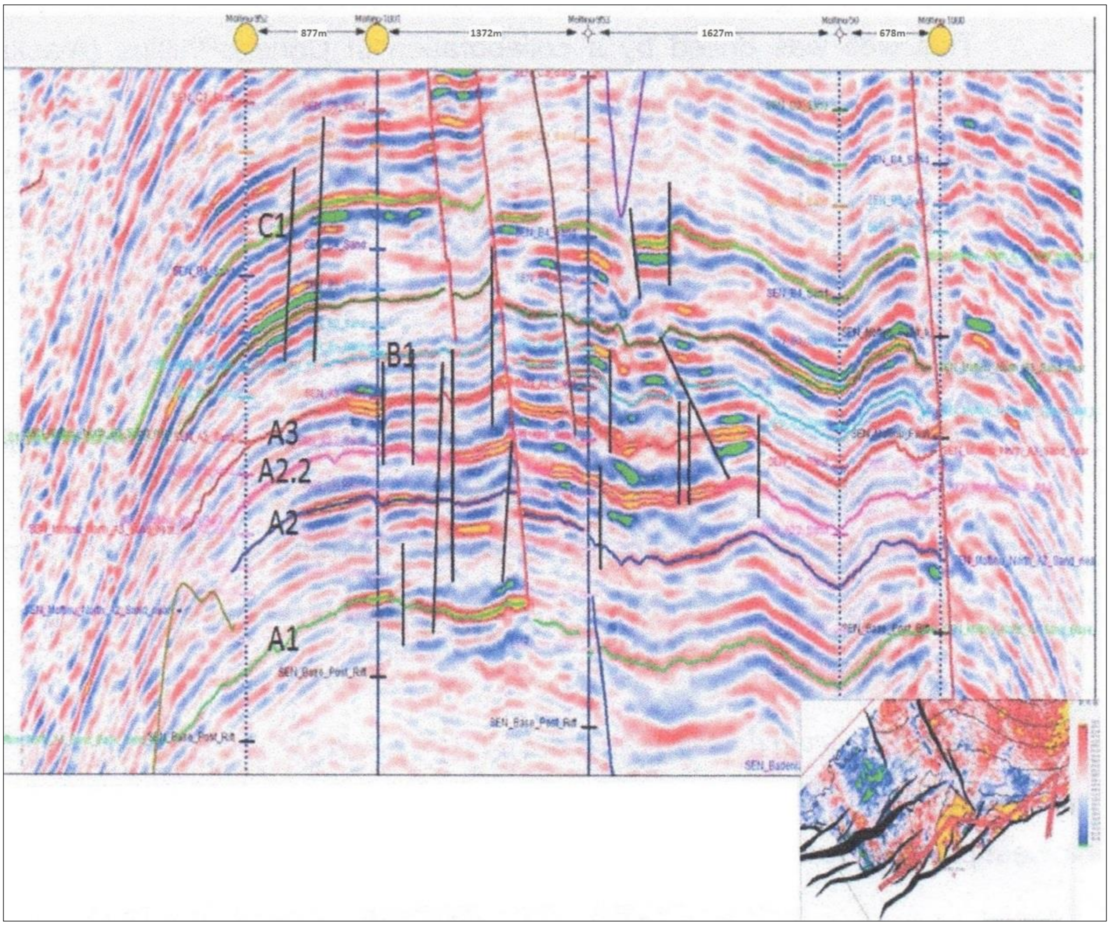

Figure 3. Seismic section in Moftinu structure (no scale) [2]

X-ray diffraction (XRD) was performed by powders method using a Bruker D8 Advance diffractometer belonging to the Petroleum-Gas University of Ploiesti, with following measurement parameters: Bragg-Brentano geometry, θ-θ type, CuKα radiation (λ = 1,54Å; 40 kV; 40 mA), step 0.1º, scanspeed 0.1º/5s, measurement range (2θ) 1-60º. Kβ radiation was eliminated by a Ni filter. Primary and secondary Soller slits were 2.5º. A fixed aperture and divergence slit of 0.6 mm, a 0.6 mm nondivergence slit and 0.1 mm width detector slit were used. The samples were grinded in an agate mortar by fine grinding, and it was placed in cavity mount in such a way as to minimize preferred orientation. XRD qualitative interpretations were carried out using Diffracplus EVA software and database PDF-ICDD 2-2008. XRD quantitative interpretations (Rietveld refinements) were carried out using the TOPAS 4.1 software. Pseudo-Voigt profile function was used for the fit of the peaks.

3. Results and discussions

In order to establish the chemico-mineralogical composition of the drill cuttings collected during the drilling of X1 and X2 Moftinu wells were carried out chemical analysis by EDX spectrometry, and complex mineralogical investigations by optical microscopy and X-rays diffraction.

3.1. EDX analysis

In table 1 is presented the chemical composition obtained by EDX/SEM analysis carried out on the samples analyzed collected during the drilling of the Moftinu wells. The standard deviation of EDX analysis shows normal values for this type of investigations in the range 0.1-0.6. Based on the previous studies/papers [2-9], in the studied area were identified silica sediments (siltstones, mudstones and sandstones), as well as carbonate sediments (limestones and marlstones). They differ mainly by contents of Si, O, Al, Ca, Fe. The chemical composition of the samples shows a high concentration of SiO_2, moderately high concentration of Al_2O_3 and CaO, and low concentration of Fe_2O_3, MgO, K_2O, Na_2O, and TiO_2. Usually, the sandstones contain more SiO_2 than those of the shales, but the concentrations of other major elements (like Al_2O_3, K_2O and Na_2O) are higher in shales. Contents of K_2O and Na_2O and their ratios suggest that K-feldspar dominantly source rocks and enrichment of K_2O is associated with the illite in shales and sandstones. The lower content of TiO_2 suggests more felsic materials in the source areas. According to the silica content, the samples analyzed corresponds to sedimentary clastic rocks. The dominant chemical components are those which can be found in the composition of typical silicate minerals: Si, O, Al, Ca, Mg, Fe. Also, the presence of Ca, Mg, Fe, can be assigned to carbonate minerals. The quartz and feldspar (albite) determine a higher silica content while the calcite determines a significant content of calcium as it can be observed in Table 1. This fact is in accordance with the X-ray diffraction study presented below.

Table 1.Chemical compositions of the samples analyzed by EDX

Sample (depth)	O	Si	Al	Ca	Fe	Mg	Na	K	Ti
X1 well (1255)	56.4	28.3	57	6.2	1.1	0.6	0.5	1.1	0.1
X1 well (1265-1285)	55.5	30.4	8.3	1.3	1.4	0.5	1.3	1.2	-
X1 well (1285-1320)	57.2	30.8	7.1	0.9	1.5	0.8	0.7	0.9	0.1
X1 well (1320-1340	55.8	29.8	8.2	1.1	1.7	0.7	0.8	1.7	0.1
X1 well (1340-1357)	51.4	29.2	10.1	3.3	1.8	0.5	1.1	2.5	0.1
X2 well (1245)	47.1	27.3	11.3	9.4	1.6	0.4	1.2	1.7	-
X2 well (1425)	46.2	27.5	10.3	9.6	1.4	0.7	1.9	2.4	-
X2 well (1445)	51.2	27.8	9.2	6.7	1.5	0.9	1.6	1.1	-
X2 well (1463)	51.5	28.1	9.7	5.1	1.9	0.8	0.5	2.2	0.1

3.2. Optical microscopy study

Samples analyzed (drill cuttings) looks like a medium-fine granular, grey-brown material, with arenite-silt granulometry (rarely rudite) and clastic structure. Most of the samples have a positive reaction at attack with 0.1N HCl.

Macroscopic and microscopic observations performed on the samples analyzed reveals the presence of the subangular to subrounded fragments (clasts) of metamorphic rocks (quartzites and micaschists), clay shales, microcrystalline limestones, microconglomerates and fine sandstones embedded in a subordinate clay-carbonate matrix.

The rock sequence of the studied area is mainly composed of fine to medium-grained sandstone, silty shale, shale, sandy siltstone, siltstone, and conglomerate. Sediments are mainly fine to medium-grained sand, with variable (but reduced) amounts of silt, mud and shell material suggesting a syn-rift depositional setting represented by a widespread transgression in which open-marine and nearshore clastic were deposited. The investigations also show that the samples studied originates by geological formations which were deposited in a shallow marine setting. Petrographic investigations suggest that the samples analyzed originate from clastic rocks rich in quartz, sedimentary and metamorphic lithic fragments which indicating a recycled orogen source and have deposited in an active or passive continental margin setting and originate from felsic source rocks.

Microscopic descriptions of thin sections are summarized in Table 2. Relevant microphotographs made on thin sections of the samples in the gas field investigated are presented in Figures 4-21.

Table 2. Mineralogo-petrographic characteristics of the samples investigated by optical microscopy

Well (depth)	**Geological age**	**Microscopic description**	**Rock diagnosis**
X1 (1255)	Neogene (Miocene)	The sample composed mainly of silt-sized clasts of quartz (Ømax 0.18 mm), subangular clasts of quartzite sandstones (Ømax 2.47 mm) and fragments of fine granular, subangular to subrounded, brown limestones (Ømax 3.80 mm) with cryptocrystalline structure (fig. 4-5). In some quartz clasts can be observed recrystallization. As accessory appear plagioclase clasts and muscovite and biotite lamellas. Chemical cement of quartz	polymictic sand
X1 (1265-1285)	Neogene (Miocene)	The sample composed of angular to rounded clasts of quartz (Ømax 2.47 mm), clasts of plagioclase (Ømax 0.19 mm), and clay shales fragments (fig. 6-7). Rarely, subrounded clasts of fine granular brown limestones (Ømax 2.28 mm), and lamellas of muscovite	clayey sand
X1 (1285-1320)	Neogene (Miocene)	The sample composed mainly of quartzite clasts (Ømax 1.18 mm), rare fragments of microconglomerates with calcite cement, clasts of plagioclase and clay shales (fig. 8-9). Rarely angular and subangular clasts of fine granular, brown limestones (Ømax 1.33 mm).	oligomictic sand
X1 (1320-1340)	Neogene (Miocene)	The sample composed mainly of rounded clasts of quartzite (Ømax 1.14 mm), rare angular clasts of quartz (Ømax 0.45 mm), clasts of plagioclase and clay shales (fig. 10-11). Subordinate, brown clasts of fine granular, rounded-subrounded (Ømax 1.58 mm). Rarely, lamellae of muscovite	oligomictic sand
X1 (1340-1357)	Neogene (Miocene)	The sample composed mainly of quartzites clasts (Ømax 1.40 mm) and angular clasts of siltic quartz (Ømax 0.19 mm), clasts of plagioclase and orthoclase feldspars, polysynthetic twinning (fig. 12-13), fragments of clay shales, and rounded to subrounded clasts of fine granular limestones (Ømax 1.90 mm), without fossils.	clayey sand
X2 (1245)	Neogene (Miocene)	The sample composed of rounded fragments of microquartzites (Ømax 0.15 mm) and angular clasts of silt-sized quartz (Ømax 0.19 mm), clasts of twinned plagioclase (fig. 14-15), fragments of clay shales, and angular, fissured fragments of brown-yellow limestones (Ømax 4.56 mm).	clay-calcareous siltstone
X2 (1425)	Neogene (Miocene)	The sample composed of clasts of detrital quartz (Ømax 1.14 mm), clasts of quartzites (Ømax 1.17 mm), clasts of plagioclase (Ømax 0.76 mm), frequent twinned, fragments of clay shales, angular (Ømax 3.40 mm) and rounded (Ømax 0.38 mm), and brown clasts of fine granular limestone, rounded and subrounded (fig. 16-17).	calcareous- clay siltstone
X2 (1435)	Neogene (Miocene)	The sample composed of clasts of microquartzites (fig. 18-19) and arenite-silt quartzite (Ømax 0.80 mm), twinned plagioclase, angular fragments of brown-yellow limestones. Rarely subangular and angular pieces of clay shales (Ømax 2.66 mm), and lamellae of muscovite	polymictic sand
X2 (1463)	Neogene (Miocene)	The sample composed of clasts of microquartzites (Ømax 1.19 mm), clasts of quartzites (Ømax 0.73 mm), fragments of clay shales (Ømax 2.37 mm), rounded and subrounded clasts of fine granular limestone, and rarely clasts of twinned plagioclase (fig. 20-21) and lamella of muscovite	clay-calcareous siltstone

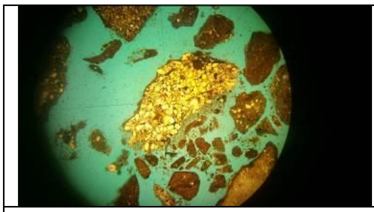

Figure 4. Subangular to subrounded clasts of quartzites and brown microcrystalline limestone (N II, 40x)

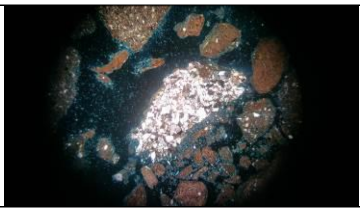

Figure 5. Subangular to subrounded clasts of quartzites and brown microcrystalline limestone (N +, 40x)

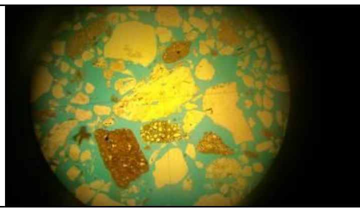

Figure 6. Subangular to subrounded clasts of quartzites, clay shales and brown limestone (N II, 40x)

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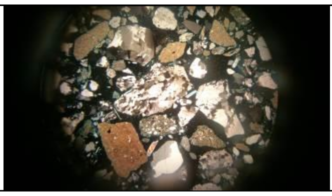		
Figure 7. Subangular to subrounded clasts of quartzites, clay shales and brown limestone (N +, 40x)	**Figure 8.** Arenite-silt, angular to subangular clasts of quartzites, limestones and clay shales (N II, 100x)	**Figure 9.** Arenite-silt, angular to subangular clasts of quartzites, limestones and clay shales (N +, 100x)
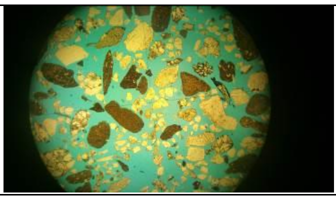	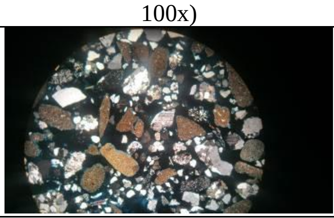	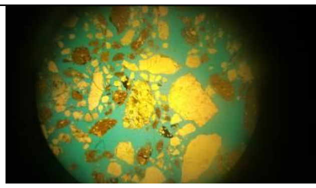
Figure 10. Subangular to subrounded clasts of quartzites, clay shales and limestones (N II, 100x)	**Figure 11.** Subangular to subrounded clasts of quartzites, clay shales and limestones (N +, 100x)	**Figure 12.** Subangular to subrounded clasts of quartzites, plagioclase, clay shales and limestones (N II, 100x)
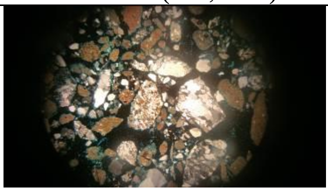	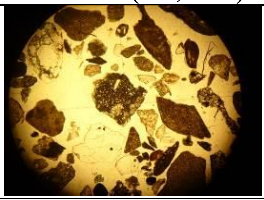	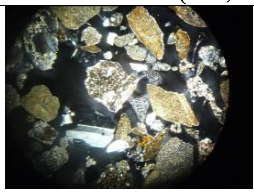
Figure 13. Subangular to subrounded clasts of quartzites, plagioclase, clay shales and limestones (N +, 100x)	**Figure 14.** Subangular to subrounded clasts of quartzites, plagioclase, clay shales and limestones N +, 100x)	**Figure 15.** Subangular to subrounded clasts of quartzites, plagioclase, clay shales and limestones (N +, 100x)
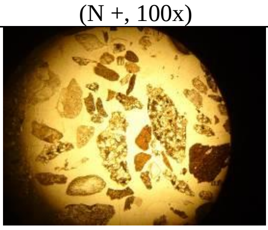	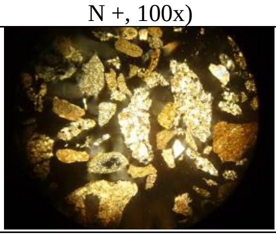	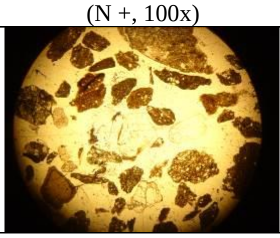
Figure 16. Subangular to subrounded clasts of quartzites, plagioclase, clay shales and limestones (N II, 100x)	**Figure 17.** Subangular to subrounded clasts of quartzites, plagioclase, clay shales and limestones (N +, 100x)	**Figure 18.** Angular to subrounded clasts of quartzites, plagioclase, clay shales and limestones (N II, 100x)
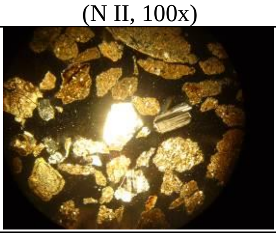	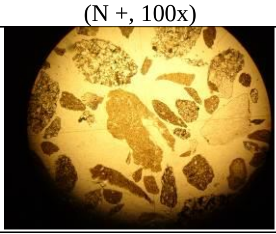	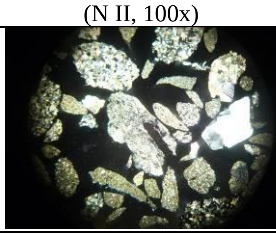
Figure 19. Angular to subrounded clasts of quartzites, plagioclase, clay shales and limestones (N +, 100x)	**Figure 20.** Angular to subangular clasts of quartzites, clay shales and limestones (N II, 100x)	**Figure 21.** Angular to subangular clasts of quartzites, clay shales and limestones (N +, 100x)

3.3. X-rays diffraction

X-rays diffraction was performed both qualitative and quantitative. Identification of crystalline phases (minerals) was made using "best quality marks": (*=high quality) and (I=indexed), after removing the background and Kα2 radiation. XRD quantitative interpretations were performed by Rietveld method. Rietveld refinement quality was expressed by R-values represented by Rwp (R-weighted pattern), GOF (goodness-of-fit), and DW (Durbin-Watson). GOF values were in the range 1.20-1.34 and indicated an excellent refinement quality. DW values were in the range 1.91-2.19 revealed a minor serial correlation, either negative or positive [10, 11].

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X-rays diffraction investigations show the presence in higher quantities, but the variable of quartz, feldspars, phyllosilicates (illite and chlorite) and calcite. Mineralogical compositions of samples analyzed resulted by quantitative analysis (Rietveld method) is presented in Table 3. It can be observed that the mineralogical compositions of the samples are similar to those of the typical clastic sedimentary rocks.

Table 3. Mineralogical composition of the samples analyzed by XRD (wt% Rietveld)

Minerals	**X1 well (1255)**	**X1 well (1265-1285)**	**X1 well (1285-1320)**	**X1 well (1320-1340)**	**X1 well (1340-1357)**	**X2 well (1245)**	**X2 well (1425)**	**X2 well (1435)**	**X2 well (1463)**
Quartz	76.82	77.58	87.15	82.94	65.44	47.27	45.61	62.03	66.35
Albite	1.86	11.03	5.62	7.45	9.51	11.60	21.25	18.62	2.84
Clinochlore	2.47	3.24	3.12	2.34	3.46	5.12	3.88	1.14	2.58
Illite	4.94	6.06	3.17	5.12	13.54	11.56	6.50	-	15.83
Calcite	12.06	1.35	0.45	1.12	4.90	17.39	17.51	12.72	11.30
Dolomite	0.68	-	-	-	-	-	1.90	-	1.13
Muscovite	1.17	0.47	0.49	1.02	0.52	-	-	0.38	0.97
Microcline	-	-	-	-	2.61	-	-	-	-
Kaolinite	-	-	-	-	-	7.06	-	-	-
Montmorillonite	-	-	-	-	-	-	3.30	5.11	-
R-values									
Rwp	6.45	7.25	8.11	7.35	6.76	9.68	10.11	13.79	10.48
GOF	1.28	1.22	1.30	1.31	1.34	1.30	1.20	1.26	1.28
DW	1.91	2.19	1.96	1.93	2.08	1.92	1.96	2.17	2.04

4. Conclusions

Identifying rock types and establishing their chemico-mineralogical composition are important components of geological characterization process of hydrocarbon reservoirs.

Knowing the detailed lithology helps in the optimum choice of drilling fluids compatible with the formations on the investigated structure. The problems of well hole instability during drilling of X1 and X2 wells from the investigated structure at depths of over 1700 m are well known.

The chemico-mineralogical composition of clastic sedimentary rocks is controlled by numerous factors including parent rock composition, weathering process, transportation mechanisms of the sediments, depositional environment and post-depositional processes.

According to the chemical composition of the samples analyzed, their silica content corresponds to sedimentary clastic rocks. The quartz and feldspars determine a higher silica content while the calcite determines a significant content of calcium, in accordance with the X-ray diffraction investigations which also shows a mineralogical composition similar with the typical clastic sedimentary rocks.

Macroscopic and microscopic observations performed on the samples analyzed reveals the presence of the subangular to subrounded fragments (clasts) of metamorphic rocks (quartzites and micaschists), clay shales, microcrystalline limestones, microconglomerates and fine sandstones embedded in a subordinate clay-carbonate matrix. The investigations also show that the samples studied originates by geological formations which were deposited in a shallow marine setting.

Petrographic investigations suggest that the samples analyzed originate from clastic rocks rich in quartz, sedimentary and metamorphic lithic fragments which indicating a recycled orogen source composed mainly of felsic rocks and have deposited in an active or passive continental margin setting.

The investigations performed in this paper complete other types of physico-chemical investigations that will be performed on cores/ sample rocks and allow through the information provided a better characterization of the rocks in the reservoir and the efficient modelling of the hydrocarbon reservoir production.

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