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# Changes in the material composition of fluid seals of bitumen deposits during thermal effects on the productive formation

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**Abstract.** Fluid seals are the most important element of oil fields, because they prevent the spread and dispersion of hydrocarbons and contribute to the preservation of deposits. The role of the fluid seals increases in the later stages of oil field development, especially when using thermal methods of intensification. As a result, changes in the mechanical, mineralogical, and lithological properties of the fluid seal rocks can occur. The deposits of natural bitumen of the Volga-Ural petroleum province are a complex geological and geochemical object. The degree of preservation of hydrocarbon deposits is determined by the filtration characteristics of the fluid seal. The composition of the fluid seals is represented by dense clay components of the rock. The use of thermal or steam-thermal methods of exposure to hydrocarbon reservoirs, primarily affects the minerals-indicators (clay minerals of cement collectors). The results of thermal effects on the reservoir can lead to changes in the material composition, parameters of porosity and permeability of the fluid seal rocks: the clay component is destroyed with the active participation of organic matter.

## 1. Introduction

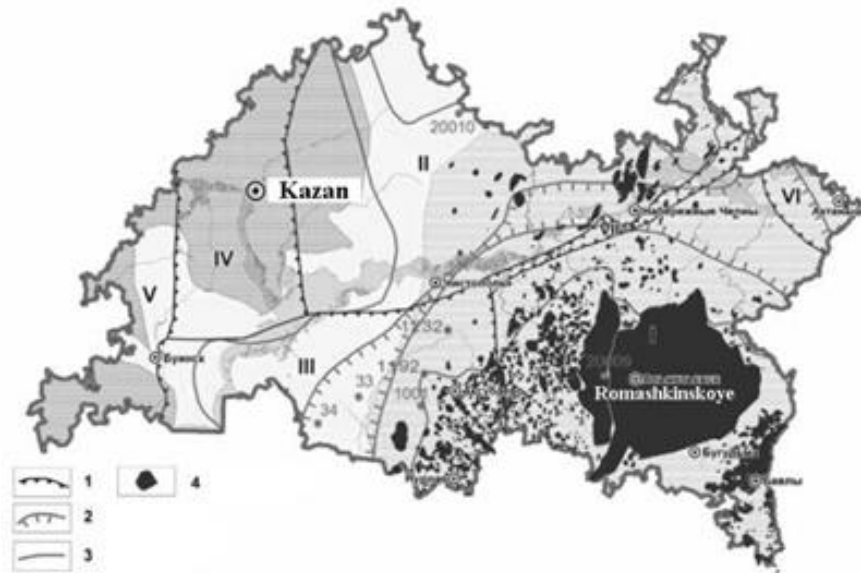
Within the Volga-Ural petroleum province in the Permian sediments numerous accumulations of natural bitumen and bitumen occurrences are known (Figure 1).

Features of distribution of bitumen accumulations in the Permian section are controlled by the spread of fluid seal horizons and reservoir rocks [1]. In the distribution and preservation of bitumen deposits, a large role is played by the peculiarities of fluid seal rocks. The quality of the fluid seal rocks can be assessed by the following criteria: the thickness of the fluid seals, their solidity, integrity, and material composition. An important criterion is areal sustainability of fluid seals [2].

The main criteria for the quality of fluid seals allowed the authors involved in the study of bituminous rock complexes to distinguish the following types of shielding fluid seals: tires of



regional, zonal and local types. Rocks of fluid seals have different thickness, in some places reach up to 100 meters or more. Local tires are developed only in certain areas of bitumen deposits, however, they are thin.



**Figure 1.** Schematic map of the Romashkinskoye oil field and its satellites (within the territorial boundaries of the Republic of Tatarstan) [2].

In the distribution of types of the fluid seals and reservoirs in the Permian sediments section are distinguished bitumen-bearing complexes: Lower Permian ( $P_1$ ) carbonate, Ufa ( $P_{1u}$ ) terrigenous, Kazan ( $P_{2kz}$ ) terrigenous-carbonate and carbonate-terrigenous [3]. In the Republic of Tatarstan, the development of natural bitumen deposits is associated with thermal methods of formation effects [3]. Thermal development methods are intra-layer combustion, steam-thermal treatment of the well bottom-hole zone, injection of heat-transfer fluids into the formation — steam or hot water. However, the use of thermal methods is significantly limited by the conditions of stability of the most important element of the bitumen deposit – fluid seals.

## 2. Methodology

We carried out a laboratory study of the stability of fluid seals of bitumen deposits in Cheremshano-Yamashinskaya structural zone (Figure 1). They are represented by a dense clay formation of “lingul clays”. The object of study is the core material of the fluid seals from wells of natural bitumens. Special attention was paid to the study of the rocks of the fluid seals of the Ashalchinskoye field. At Ashalchinskoye field, the development of natural bitumen is carried out using thermal methods of extraction.

The main research methods are macro-structural-texture analysis, optical methods on a Leica microscope. The method of X-ray diffractometry was used to study the phase composition of clay minerals of the initial rocks and subjected to thermal effects (diffractometer DRON-3M, Cu  $K\alpha$  radiation, shooting modes: 20kv x 20ma, slits 0.5= =0.5,

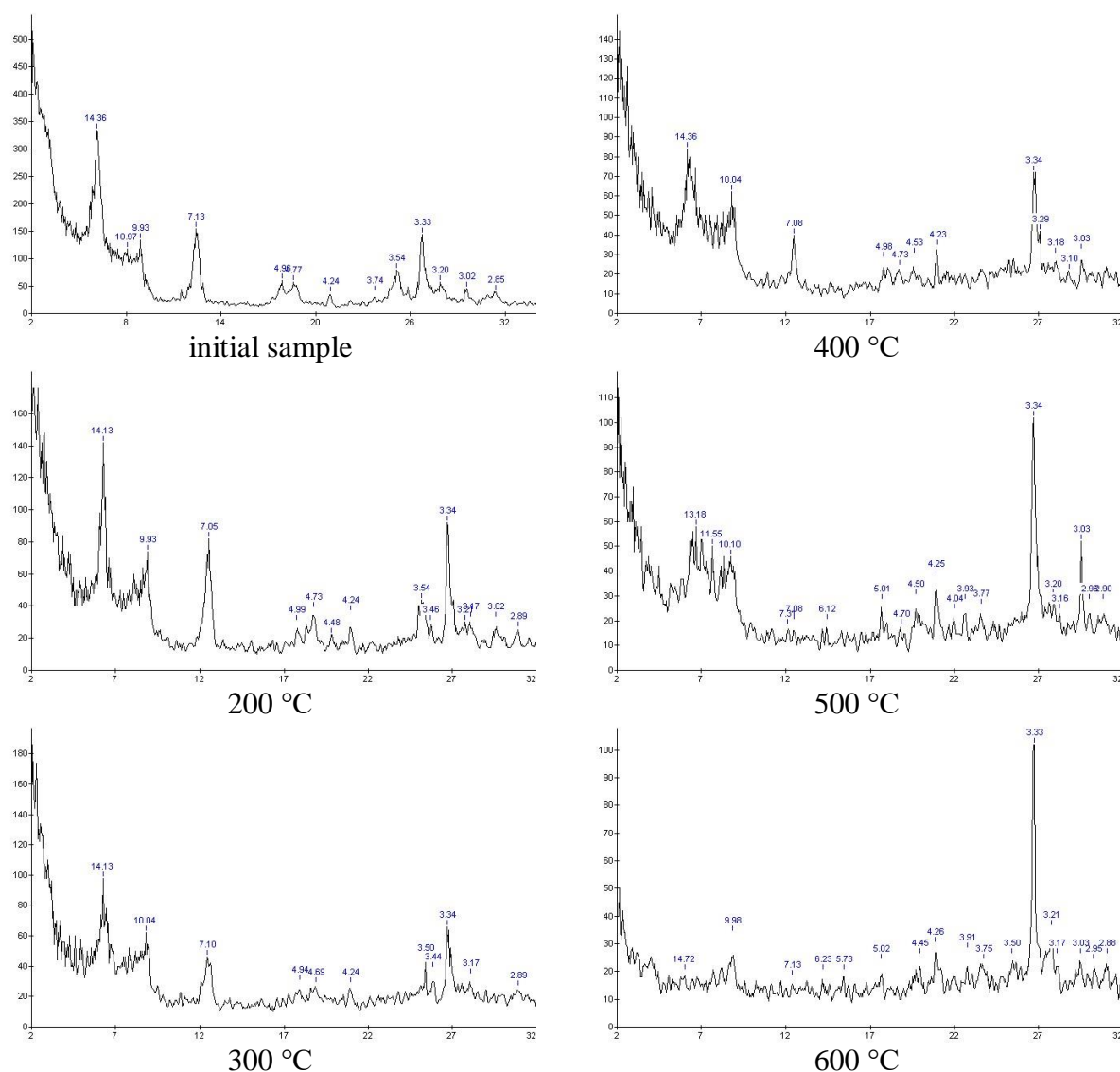
preparation shooting range  $2-36^{\circ}2\theta$ ). Oriented preparations of clay minerals were made by centrifugation method, studied in air-dry state and after thermal exposure. Heat treatment of initial samples of rocks of fluid seals was carried out in the muffle furnace SUOL in the temperature range from 200 °C to 600 °C.

The study of the features of the distribution of manganese ions was carried out by electron paramagnetic analysis (EPR) on a spectrometer CMS8400 3 cm range. The samples were heated for 30 minutes in a hydrogen atmosphere at temperatures of 350 °C and 600 °C in a horizontal electric furnace of the SUOL type. Morphological transformations take place at temperature effects of fluid seals samples, because for their study was used the method of scanning electron microscopy. Samples of fresh rock chips, decorated with a thin layer of gold in the Agar sputtering system, were studied. The investigations were carried out with a Philips XL-30 scanning electron microscope in the Hi-vac mode, detector of backward scattered electrons SE, voltage of 20 kv, spot 4, magnification from 500x to 2500x.

### 3. Results and Discussions

The rocks of the fluid seals are composed of a clay-aleuritic series with complex mineral composition. The bulk of the rock is a complex of clay minerals: illite, mixed-layer minerals such as mica-smectite with a ratio of 80:20 layers, chlorite [4, 5]. The fine substance consists of quartz, feldspar, and pyrite framboids (Figure 2). Carbonaceous excrements and fragments of organic residues are also present in the samples.

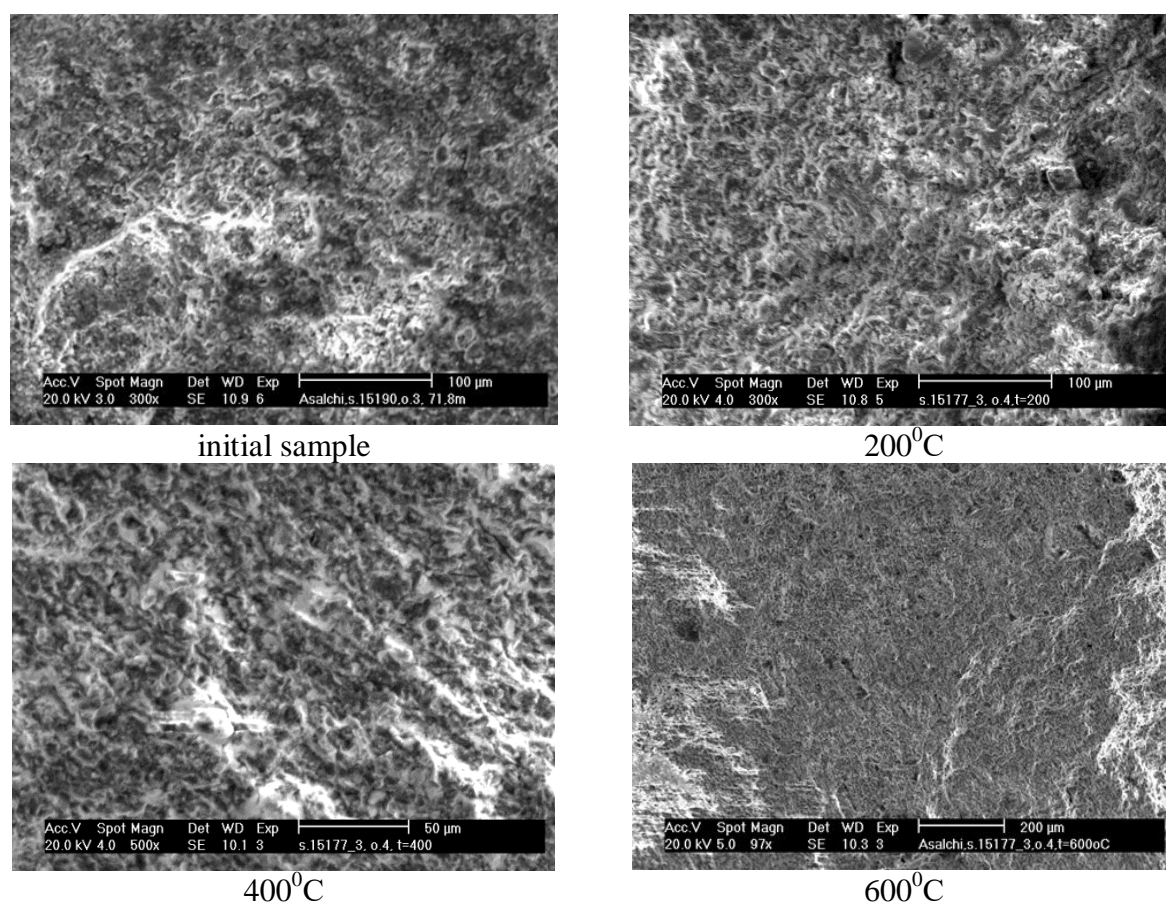
In order to determine the stability of the rocks of the fluid seals deposits during thermal effects on the reservoir, differential thermal analysis (DTA) was performed in the temperature range from 20 ° to 950 °C. According to the data of DTA, a number of thermal effects are observed in the studied samples of fluid seals. In the range of 110-250 °C, the loss of interlayer water molecules in the mixed-layer phase mica-smectite occurs. Then there is a loss of hydroxyl groups of the tetrahedral and octahedral layers of the crystal lattice. The most active losses occur in the temperature range 650-750 °C. According to X-ray phase analysis, the final degradation of the illite phase occurs with its transition to a stable mica phase (Figure 2). Thus there is a restructuring of the clay component of the fluid seals samples. Rocks almost completely lose their plastic properties, which is accompanied by deformation processes: the emergence of micro-fracturing, secondary porosity, etc. (Figure 3).



**Figure 2.** Degradation of the phase composition of clay components (illite, mixed-layer minerals such as mica-smectite, chlorite) and fine minerals (quartz, calcite, orthoclase) at thermal processing of samples of fluid seals rocks.

The heterogeneity of the mineral composition of fluid seals samples is also confirmed by the results of the EPR analysis [6]. The survey spectrum of the EPR analysis (360 mT scan) shows structural iron lines ( $g = 4.3$ ), six characteristic lines of the impurity radical  $\text{Mn}^{2+}$  in the carbonate structure, and lines corresponding to the organic radical  $\text{C}_{\text{org}}$  (Figure 4).

The change in the intensity of the EPR signal of manganese ion in the carbonate part of the rock before and after thermal (350 °C) exposure is shown in the diagram (Figure 5).

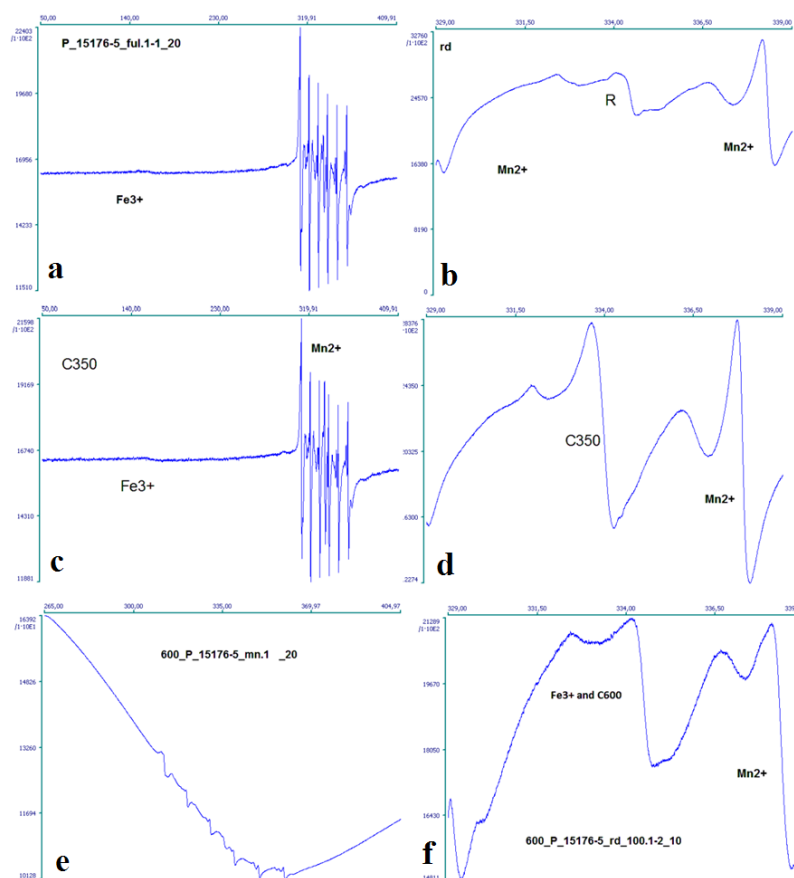


**Figure 3.** Changes in the morphological properties of samples during thermal effects on the rocks of fluid seals.

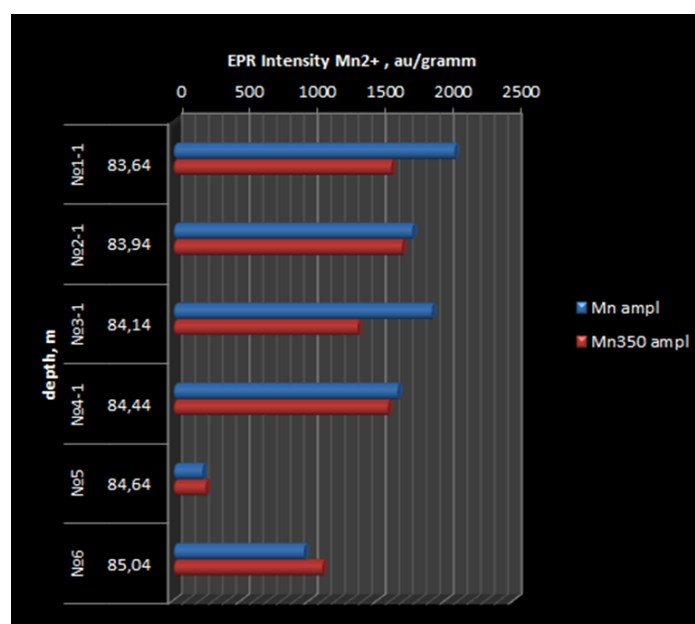
The variations in the intensity values of the EPR signal under thermal effects even within a few meters of thickness are established. At a depth of 83.64 m and 84.14 m (samples 1 and 3), the manganese signal decreases as a result of its possible leaching from the rock. As a result, the pore space of the sample increases. Minor changes in the intensity of the manganese impurity ion at depths of 83.94 m and 84.44 m (samples 2 and 4) are only indicative of a change in post-sedimentary conditions and the preservation of pore space.

Also, according to the results of the EPR analysis, it was noted that at the depths of 84.64 m and 85.4 m (samples 5 and 6) there was a sharp change in the material composition of the rocks from substantially carbonate to substantially clay.

Thermal effects up to temperatures of 600 °C, unlike exposure at 350 °C, are accompanied by intensive leaching and oxidation of iron ions with the formation of supramagnetic fractions, which contributes to the formation of spin density and favorable conditions for the generation of hydrocarbon gas fractions [7-9].



**Figure 4.** Overview EPR spectra of samples: a, b - initial samples, c, d - after thermal exposure at temperatures of 350 °C, e, f - after thermal exposure at temperatures of 600 °C.



**Figure 5.** The change in the intensity of the EPR signal of manganese ion in the carbonate part of the rock before and after thermal (350 °C) effects.

#### 4. Conclusions

The deposits of natural bitumen of the Republic of Tatarstan are distinguished by a high lithological heterogeneity, uneven distribution of bitumen. Deposits of natural bitumen in Ufa sediments (P<sub>1u</sub>) are being actively developed. For the preservation of deposits of bitumen, the quality of fluid seals horizons becomes relevant. It is especially important to take into account the state of the fluid seals rocks in the thermal methods of oil field development.

Fluid seals horizons of bitumen and bitumen-bearing rocks are clay rocks of Kazan age (P<sub>1kz</sub>), the so-called pack of “lingul clays”. Illite, mixed-layer complexes of illite-smectite, chlorite and finely dispersed minerals predominate in the mineral composition of fluid seals rocks.

According to the results of laboratory tests of fluid seals samples, a number of thermal effects can be stated. At temperatures up to 200–250 °C, loss of interlayer water molecules of the swelling phase of clay minerals occurs. At higher temperatures, the restructuring of their crystal lattice begins with the loss of hydroxyl groups of tetrahedral and octahedral layers. The most active losses of hydroxyl groups at temperatures of 650–750 °C are associated with the processes of degradation of the illite phase with its transition to stable mica.

Temperature transformations of rocks cause morphological transformations that change the overall appearance of the rock. Initial homogeneous microporous samples at temperatures above 400 °C experience the appearance of microcracks and interconnected pores. There is a microstructural restructuring of the matrix of fluid seals rocks with the loss of their plastic properties.

Increasing porosity values in reservoir conditions can lead to breakthroughs of the coolant, liquid and gaseous components of bitumen in the overlying rocks, leading to negative environmental consequences. It is necessary to take into account the thickness of fluid seals horizons, to conduct a detailed study and control of the mineral composition of the fluid seals rocks and the physical condition of the rocks during active thermal methods of development.

#### Acknowledgements

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