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## Comparison of the effect of inhibited drilling muds parameters on the physical and mechanical properties of shale rocks

This paper presents the results of laboratory tests on the effect of inhibited drilling muds of different rheological and inhibition properties on the physical and mechanical parameters of shales. Polymer-potassium and glycol-potassium muds were selected for these studies. Measurements of the physico-mechanical properties of rocks were done using the Szeiner method, first in the air-dry state and after 48 hours, the impact of muds at ambient temperature and atmospheric pressure and under HPHT. Also determined were the inhibition properties of prepared drilling muds through the study of Miocene shale dispersion. The results were the basis for the analysis and determination of the impact of muds on selected rocks.

Key words: drilling mud, polymer-potassium mud, glycol-potassium mud, shales, physico-mechanical properties of the rocks.

### Porównanie wpływu parametrów inhibitowanych płuczek wiertniczych na fizyczno-mechaniczne właściwości skał ilasto-łupkowych

W pracy przedstawiono wyniki badań laboratoryjnych określających wpływ inhibitowanych płuczek wiertniczych o różnych właściwościach reologicznych i inhibitacyjnych na parametry fizyczno-mechaniczne skał ilasto-łupkowych. Do badań wytypowano płuczkę polimerowo-potasową oraz glikolowo-potasową. Pomiarów parametrów fizyczno-mechanicznych skał dokonano z zastosowaniem metody L. A. Szeinera, najpierw w stanie powietrzno-suchym a następnie po 48-godzinnym oddziaływaniu płuczek w warunkach temperatury otoczenia i ciśnienia atmosferycznego oraz w warunkach HTHP. Określono również zdolności inhibitacyjne przygotowanych płuczek wiertniczych poprzez badanie dyspersji łupku miocenijskiego. Otrzymane w ten sposób wyniki były podstawą do wykonania analizy i określenia wpływu płuczek na wybrane skały.

Słowa kluczowe: płuczka wiertnicza, płuczka polimerowo-potasowa, płuczka glikolowo-potasowa, skały łupkowe, parametry fizyczno-mechaniczne skał.

### Introduction

The process of obtaining hydrocarbons from shale formations requires advanced drilling technology. In the context of shale gas exploitation, particular attention is paid to the process of hydraulic fracturing, while an equally important step is the proper design and correct drilling process. The later efficiency of gas production is a consequence of both the quality of the drilled borehole and the size and spatial orientation of the fractures.

The process of drilling and hydraulic fracturing, which is performed later, is highly influenced by the type of drilling mud used. It is very important that drilling muds used during the drilling process caused minimal physical and mechanical changes to the parameters of drilled rocks. That is why drilling muds should have suitable rheological and inhibition properties. The water phase of drilling muds have the most adverse influence, which as a result of processes such as

filtration, osmosis and capillary suction, enters into rock causing it to swell and deteriorates the physical and mechanical properties. In order to minimize this negative effect, inhibited drilling muds are used, which contain special chemicals, such as polyglycols or partially hydrolyzed polyacrylamide also known as PHPA. These chemicals are designed to create on

the surface of the drilled rocks a seal film that prevents the penetration of the water phase [2, 4, 11].

This paper presents a comparison of the influence of two types of inhibited drilling muds with various rheological and inhibition parameters on physical and mechanical properties of shale rocks.

### Characteristics of inhibited drilling muds

The type and parameters of drilling muds should be adapted to geological conditions and drilling technology. An appropriately selected drilling mud should be designed to provide a proper and trouble-free drilling process. Drilling through layers of shale rocks is accompanied by the risk of complications caused by the loss of stability of the drill hole. This is due to the particular vulnerability of such rocks of the hydration phenomenon, by which the crystal structure of minerals is disturbed. Therefore, it is essential what type of drilling mud is used, particularly its inhibition properties. Such muds should primarily be capable of counteracting the dispersion and swelling of shale material and do not cause deterioration of hydraulic conductivity in the production area [3, 9]. Depending on the type of used inhibitor, or a set of several inhibitors, muds can be divided into:

- polymer-potassium drilling mud,
- glycol-potassium drilling mud,
- silicate drilling mud,
- triple inhibited drilling mud.

In water-based drilling muds currently used, mainly two mechanisms to inhibit the hydration of shale rocks are used: ionic inhibition and polymer inhibition. Ion inhibition requires in the drilling mud composition, the use of chemical compounds, which are a source of anions and cations which may react with clay minerals. The most effective influence is achieved by using potassium compound, especially potassium chloride. This is mainly due to the small size of  $K^+$  ions, so during ion exchange they can penetrate deep into the interlayer spaces of clay minerals. By gluing clay layers they produce a durable structure with high resistance to hydration and swelling [5].

Other types of substances used in drilling mud technology for protection of shale rocks against the effects of the water phase are polymer agents. The most common in use, is partially hydrolyzed polyacrylamide also known as PHPA. PHPA's task is to produce a very thin and resilient coating (film) on the surface of drilled rocks and cuttings due to ionic attraction and hydrogen bonds. This film prevents the penetration of mud filtrate into the rock. Furthermore, PHPA dispersed in the mud causes increased viscosity, which can

further reduce the hydration of the shale rock [7]. Studies and long-term practice have shown that PHPA is much more effective in combination with an ionic inhibitor in the form of potassium chloride. This configuration allows to achieve a high degree of reducing the hydration and swelling of drilled rocks.

The next agents from the group of polymers are glycols or otherwise polyglycols. These are polymers having a low degree of polymerisation, present in the solid or liquid state, water-soluble at ambient temperature [10]. Heating a mixture of water and glycol causes a decrease of solubility of the glycol, which finally results in its complete separation from the water. The temperature in which there is complete separation of glycol and water is called the cloud point. Clouding of the mixture occurs by the secretion of polyglycol in the form of microdroplets. Lowering the temperature results in redissolving glycol in water. The cloud point is variable, and depends on the type and concentration of polyglycol (with increasing molecular weight cloud point decreases) in the mixture [8]. Polyglycols acting as inhibitors of shale rocks hydration is quite complex and has never been fully explained, despite lots of comprehensive research. It is assumed that the polyglycols penetrate into the interlayer space of clay minerals, where the hydroxyl groups interact with the surface. Penetrating into the rock causes displacement of water, which is due to the greater affinity of the clay into glycol than into water, and then forming an impermeable protective layer against hydration. Polyglycols, like in the case of PHPA are much more efficient in the presence of ion inhibitors in the form of potassium chloride. This synergetic effect of polyglycol and KCl is difficult to explain. Increased efficiency may be due to properties of  $K^+$  ion, which is characterized by lower hydration energy compared to  $Na^+$  or  $Ca^{2+}$  ions. It is believed that  $K^+$  ions interact with the inner part of the polyglycol molecules chains, which repel water and form a stable water resistant complex [1, 10]. By combining the beneficial effects of different inhibition mechanisms, in practice drilling muds have combined inhibition systems, for example polymer-potassium or glycol-potassium [7].

### Measurement of the physical and mechanical parameters of rocks using the Szreiner method

To determine the physical and mechanical properties of rocks, devices called “rockmeter” are used, which operate according to the Szreiner method. Knowledge of rocks properties originally helped in the selection of drilling tools and optimal drilling parameters. Currently, these tests are also applicable in determining the impact of drilling fluids on shale rocks. Observing the changes of physical and mechanical parameters of shale rocks under the influence of drilling muds, gives the opportunity to assess the composition of the muds from the perspective of ensuring the stability of the borehole wall. The results of these studies also allow to determine the optimal values of the technological parameters of drilling muds, such as the rheological properties or inhibition parameters [2].



Fig. 1. Cylindrical indenter acting on the sample [6]



Fig. 2. Rock sample after the test [6]

The measurement is based on pressing the cylindrical indenter onto a specially prepared sample of rock. The sample's estimated hardness value, determines the choice of the indenter base area: 1, 2 or 5 square millimeters. For testing shale rocks, usually indenters with a base area of 2 or 5 square millimeter are used. When the adequate indenter is attached, the force is continuously increased until the structure of tested rock is destroyed. A set of “rockmeter” sensors measure the force which act on the rock and deforms it. These quantities allow to determine the hardness of the rock. Due to the heterogeneous nature of the rocks, on indenter-rock contact, a resultant hardness is obtained. The value of the resultant hardness, area and volume of crushed rock allow to determine the other physical and mechanical properties. The impact of the indenter on the sample produces a triaxial stress state, under the condition that the tip of the indenter has a dimension that does not cause splitting of the rock sample. Therefore, physical and mechanical properties of the rock are determined by “rockmeter”, this refers to the triaxial stress state [2, 12].

Measuring with the “rockmeter” allows to determine the physical and mechanical parameters of the tested samples, which allows to estimate the impact of the drilling mud on the properties of the rocks. In order to facilitate the interpretation of the results, six ratios of physical and mechanical parameters are used. These are dimensionless quantities, obtained as a result of dividing the value of each parameter, measured after the impact of mud by values of these parameters, in the air-dry state. This implies that the value of the ratio is closer to 1, the smaller the change occurred in the parameter. To assess the impact of drilling muds on the shale rocks, the resultant hardness ratio and plasticity factor ratio were used.

In order to estimate the impact of muds on the physical and mechanical parameters of rocks, the following steps were taken:

- 1) measurement of parameters of rock samples in air-dry state,
- 2) measurement of parameters of rock samples after drilling mud impact for a period of 48 hours,
- 3) comparison of the obtained results to determine the changes in physical and mechanical parameters of rock samples.

In order to explore the influence of muds on rock samples deeply, besides measurements after impact of muds in ambient temperature and atmospheric pressure, it was also decided to execute the test after holding the samples in the muds in a high temperature and high pressure. To simulate the down-hole conditions autoclaves were used in which the sample was placed flooded with mud and then adjusted to a pressure of 1 MPa and the mixture was placed in an incubator for the duration of 48 hours at 80°C. With this methodology, it was

possible to compare the impact of drilling muds with different characteristics even under downhole conditions, which gives

a lot wider possibilities when assessing the effectiveness of their impact.

### Discussion of the results of laboratory tests

Polymer-potassium and glycol-potassium drilling muds were selected for laboratory testing. This choice was made mainly due to the frequency of use of the above drilling muds in shale rocks in Poland. Polymer inhibitors used in the composition of these muds have different physico-chemical properties and mechanisms of rock protection against the negative effects of the water phase. These differences also result in various degrees of impact on the physical and mechanical parameters of rock, which was confirmed by the results of the tests carried out using “rockmeter”.

Both types of muds used in the laboratory tests are based on a similar set of structure-forming polymers. These were: Polofix LV, XCD, PAC R and Rotomag. Similarly, in both muds one of the shale hydration inhibitors was potassium chloride. The muds had the same density of 1.25 g/cm<sup>3</sup>, obtained through the use of barite. To determine the effect of the rheological and inhibition properties of muds on the physical and mechanical parameters of rock, five types of each of the mud were prepared:

- starting mud,
- mud with reduced values of rheological parameters,
- mud with increased values of rheological parameters,

- mud with reduced values of inhibition parameters,
- mud with increased values of inhibition parameters.

Examination of the impact of inhibited drilling muds on physical and mechanical parameters of shales required the choice of appropriate rock samples. The selected samples came from cores taken from two boreholes located within the Leba elevation. Samples named “1” were cut from the core collected in the hole L3 at a depth of 2858 m. Samples named “2” were cut from the core coming from the hole Z6K at a depth 2699 to 2775 m.

Figure 3 shows the resultant hardness ratios of rock 1 depending on the type of mud. It is clearly evident that the value of the ratio was closer to 1 for glycol-potassium mud (muds 6–10). Only the polymer-potassium mud with increased concentration of potassium chloride and PHPA allowed to achieve similar effectiveness. Other variants of tested polymer-potassium mud was far worse than glycol-potassium mud. The least negative impact on the rock 1 parameters was achieved by mud 10 (glycol-potassium mud with increased concentration of glycol and KCl) – in its case the resultant hardness ratio was 0.90 at ambient temperature and 0.75 at HPHT conditions. The worst results were obtained with mud 4

Table 1. Parameters of tested drilling muds

Number	Drilling mud type	Density [g/cm <sup>3</sup> ]	Viscosity [mPa · s]		Yield point [Pa]	Gel strength [Pa]	Filtration API / PHT [cm <sup>3</sup> ]		pH
		$\rho$	$\eta_{pl}$	$\eta_s$	$\tau_y$	II	20°C	80°C	
1.	Polymer-potassium	1.25	34	57.0	22.0	4.3	4.1	6.0	9.3
2.	Polymer-potassium with reduced values of rheological parameters	1.25	27	40.5	12.9	2.4	4.6	8.2	9.3
3.	Polymer-potassium with increased values of rheological parameters	1.25	43	75.0	30.6	6.2	4.4	6.4	9.3
4.	Polymer-potassium with reduced values of inhibition parameters	1.25	31	52.0	20.1	2.4	4.4	6.8	9.2
5.	Polymer-potassium with increased values of inhibition parameters	1.25	35	58.5	22.5	2.9	4.0	7.2	9.3
6.	Glycol-potassium	1.25	30	49.0	18.2	3.8	7.5	7.6	9.2
7.	Glycol-potassium with reduced values of rheological parameters	1.25	23	33.5	10.0	1.9	4.6	11.0	9.2
8.	Glycol-potassium with increased values of rheological parameters	1.25	43	75.0	30.6	6.7	3.0	6.0	9.2
9.	Glycol-potassium with reduced values of inhibition parameters	1.25	29	47.0	17.2	3.3	3.8	7.8	9.1
10.	Glycol-potassium with increased values of inhibition parameters	1.25	30	48.5	17.7	3.3	3.9	8.4	9.1

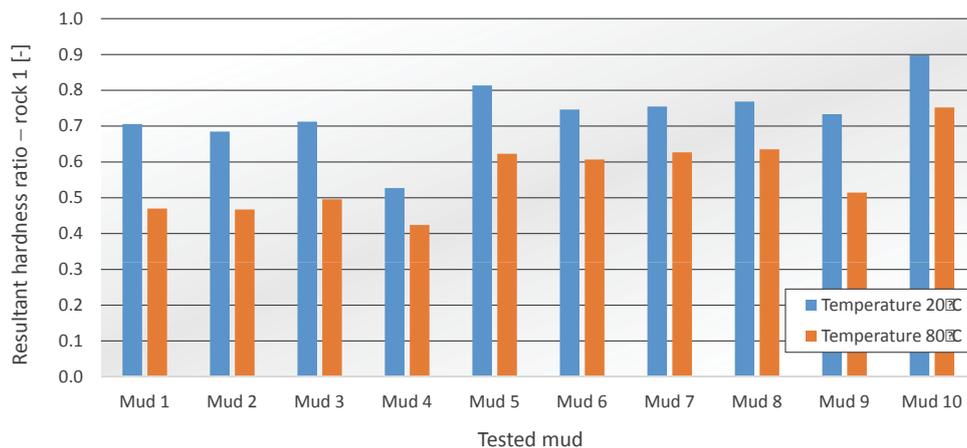


Fig. 3. Comparison of the resultant hardness ratios after impact of muds on rock 1

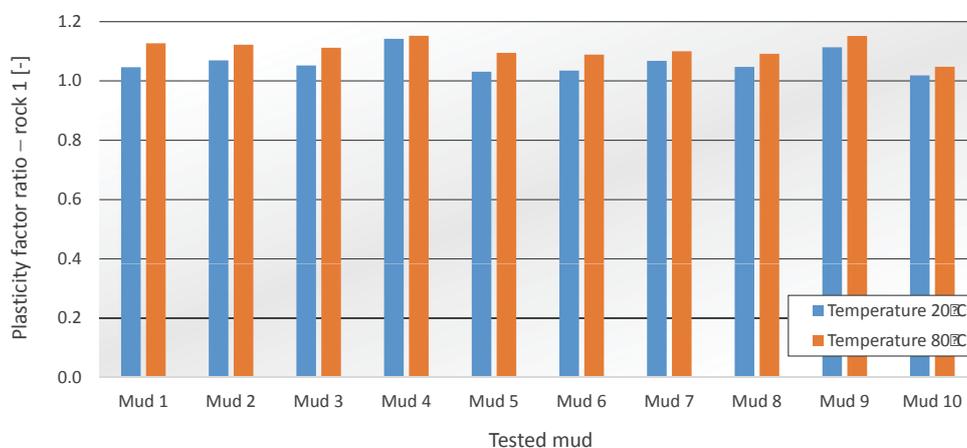


Fig. 4. Comparison of the plasticity factor ratios after impact of muds on rock 1

(polymer-potassium mud with reduced content of PHPA and KCl) – the resultant hardness ratio was 0.53 at ambient temperature and only 0.42 at high temperature and high pressure.

A similar conclusion can be reached by analyzing Figure 4, which shows the plasticity factor ratios of rock 1 under the influence of tested muds. Particular variants of polymer-potassium mud resulted in an increase of the plasticity factor in the range of 5÷14% at ambient temperature and 11÷15% at HPHT conditions. Only mud 5 efficiency was equal to glycol-potassium muds, causing an increase in the plasticity factor value by 3% at ambient temperature and by 9% under downhole conditions. In comparison, the glycol-potassium muds caused a change of plasticity factor value in the range of 4÷11% at ambient temperature and 9÷15% under HPHT conditions, and the

of glycol-potassium muds, numbered 6–8. The ratios for the muds 6–8 at ambient temperature had a value 0.77÷0.80, while after the impact of mud 5 the value of the ratio was 0.85. Under HPHT conditions, the influence of muds 6–8 resulted in a reduction of the resultant hardness by 34÷38%, and the impact of mud 5 resulted in a change of this parameter by 39%. Definitely the most efficient was mud 10 (glycol-potassium mud with

mud with a higher concentration of glycol and potassium chloride resulted in an increase of the factor by 2% at ambient temperature, and only 5% under conditions of high temperature and high pressure.

Figure 5 shows the resultant hardness ratios for rock 2. Analogously as occurred in the case with rock 1, glycol-potassium mud was more efficient. Resultant hardness ratios after the impact of polymer-potassium muds marked 1–3 at ambient temperature, were in the range of 0.66÷0.72, and under HPHT conditions the range was 0.47÷0.49. The worst result was observed for the mud 4, polymer-potassium mud with a reduced concentration of hydration inhibitors. The resultant hardness ratio has a value of 0.51 at ambient temperature and only 0.42 under HPHT conditions. The best from amongst the group of polymer-potassium muds, mud 5, was marked by a degree of influence on the level

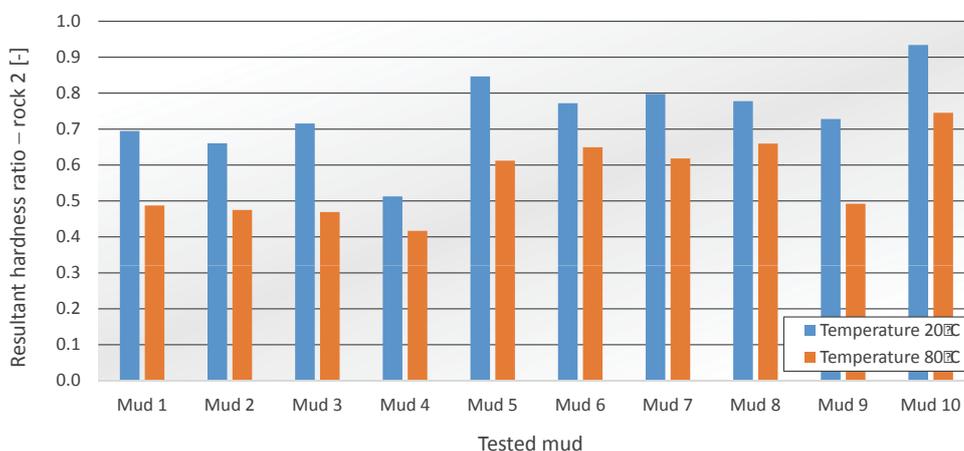


Fig. 5. Comparison of the resultant hardness ratios after impact of muds on rock 2

increased concentration of glycol and potassium chloride). At ambient temperature the resultant hardness ratio had value 0.93, and 0.75 under HPHT conditions.

The analysis of the plasticity factor ratio of rock 2 also shows the higher efficiency of glycol-potassium muds. The influence of muds marked 6–8 increased the plasticity factor by 3÷5% at ambient temperature, and by 7÷11% under HPHT conditions. Again, the least negative impact on the parameters of rock showed mud 10, for which the change in the plasticity factor was 4%. Measurement

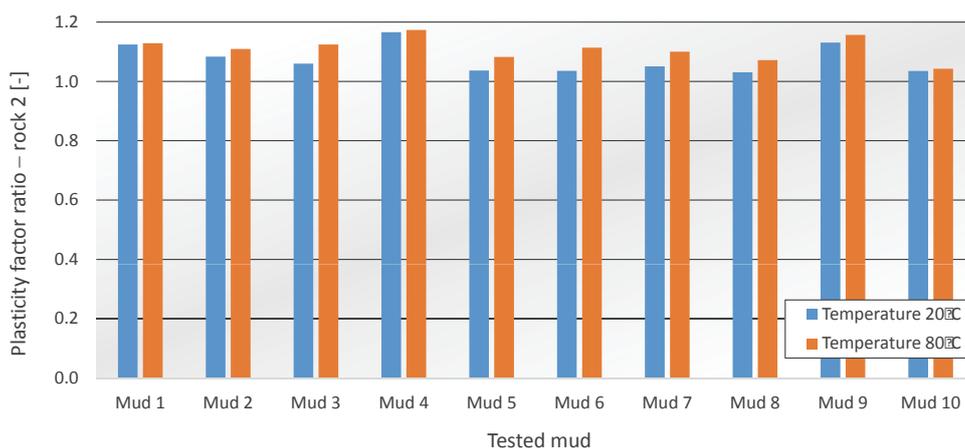


Fig. 6. Comparison of the plasticity factor ratios after impact of muds on rock 2

uncertainty is estimated on the basis of accuracy class of measuring instruments.

### Evaluation of shale inhibition properties of muds using the Miocene shale dispersion method

The results of measurements of the physical and mechanical properties of rocks allow to conclude that the effect of polymer-potassium mud is not as effective as glycol-potassium mud. This is evidenced by the changes of the resultant hardness and plasticity factor ratios of both rocks 1 and 2, obtained at ambient temperature and atmospheric pressure and at a temperature of 80°C and pressure of 1 MPa. This is confirmed by the results of the Miocene shale dispersion shown in Figure 7.

A high correlation between the impact of muds on the physical and mechanical parameters of rock, and the degree of protection against the hydration of the Miocene shale can be observed. After a 6-hour immersion in the polymer-potassium muds, the recovery of cuttings was measured in the range of 70÷92%, and after a 2-hour

exposure to tap water the remaining amount of Miocene shale was in the range of 2÷24%. After a 6-hour impact of glycol-potassium muds, Miocene shale recovery was as much as 88÷98%. These muds provided very effective protection against the hydration phenomenon, but cuttings recovery after a 2-hour exposure to tap water was still high – between 46% and 60%.

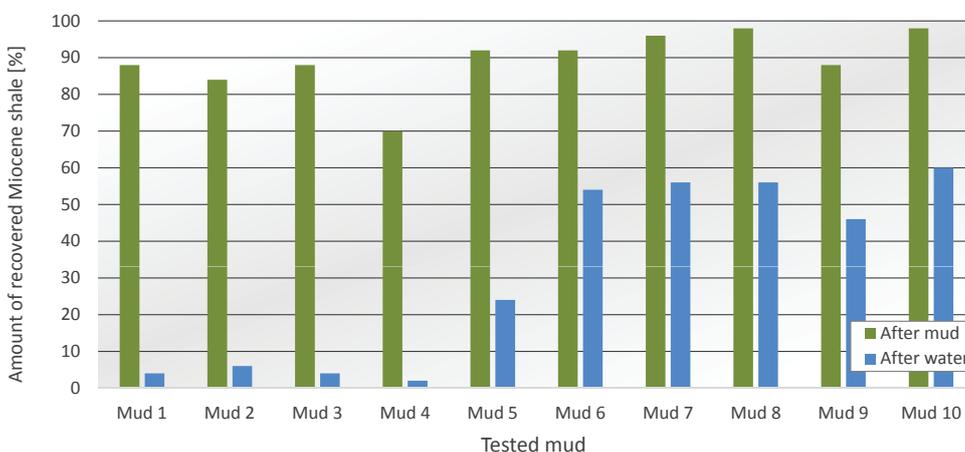


Fig. 7. Results of Miocene shale dispersion test

### Summary and conclusions

In the course of studies the impact of two types of inhibited drilling muds on the physical and mechanical properties of shale rocks were examined. These drilling muds are designed to provide efficient and trouble-free drilling through shale rock layers, which ensures the mechanism of hydration prevention. In the polymer-potassium mud the set of PHPA – potassium chloride is responsible for the

prevention of shale hydration, while in the glycol-potassium mud it is a polyglycol-potassium chloride combination. To measure the impact of these drilling muds on the physical and mechanical properties of rocks, both types of muds were prepared in five versions, differing in rheological and inhibition properties. Studies with use of “rockmeter” were carried out on two types of shale rock after immersion in

prepared drilling muds, at ambient temperature and atmospheric pressure, and under HPHT conditions.

Analysis of the results of laboratory tests can provide the following conclusions:

1. Adjusting the rheological parameters such as viscosity, yield point and gel strength has little effect on the resultant hardness and plasticity factor of studied rocks. The differences between the effects of the starting mud and those with reduced or increased values of rheological parameters are only a few percent.
2. A significant impact on the rocks parameters was noted in the muds with different concentrations of hydration inhibitors. In the case of polymer-potassium muds, reduction in the potassium chloride content from 5% to 3%, and the PHPA from 0.3% to 0.2% causes a substantial reduction of the resultant hardness and an increase of the plasticity factor of the studied rocks, both at ambient temperature and under the HPHT conditions. The opposite trend takes place by increasing the content of KCl to 7%, and PHPA to 0.4%.
3. Glycol-potassium muds are characterized by more effective inhibition of shale rocks than polymer-potassium muds, which is confirmed by both the results of research on the impact on the physical and mechanical parameters of rock, as well as testing the Miocene shale dispersion. The apparent correlation is very high according to the results of both studies.
4. Increasing the temperature to 80°C and the pressure to 1 MPa, results in a significant deterioration in the efficiency of the muds. Regardless of the type of tested mud, the rheological and inhibited parameters, in HPHT conditions the resultant hardness and plasticity factor of rocks are subject to major changes than it is at ambient temperature and atmospheric pressure [6].

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