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Biomonitoring studies and supervisory research for the purposes of underground gas storage and currently exploited natural gas deposits

Badania biomonitoringowe oraz kontrolne prace badawcze dla potrzeb podziemnego magazynowania gazu i obecnie eksploatowanych złóż gazu ziemnego

Anna Turkiewicz, Marek Janiga

Oil and Gas Institute – National Research Institute

ABSTRACT: This paper refers to several aspects of research studies supporting the oil and gas industry – in particular underground gas storage (UGS) in depleted deposits and salt caverns – and focuses on questions related to the formation of hydrogen sulphide contamination in reservoir conditions as well as on methods for limiting unfavourable biogenic phenomena. The main problem found at gas storage facilities is the activity of sulphate reducing bacteria (SRB). The elimination or limitation of H_2S generation in microbiologically contaminated environments have been the subject of many extensive studies. Biocides, biocorrosion inhibitors and H_2S scavengers are widely applied to protect reservoir structure, gas storage infrastructure as well as water-based drilling fluids from the negative effects of bacterial activity. One of the most popular biocidal products, recommended for oil and gas industry are triazine derivatives, laboratory tested in the presented biomonitoring studies. Triazine products prove very effective in biomass reduction and elimination of anaerobic bacteria, especially SRB. Before any industrial operation (based on technology of using biocides), it was necessary to analyse the sulphur compounds in the stored natural gas in different exploitation gas wells of UGS. It was also necessary to investigate the selection of a specific biocidal product and its proper concentration. A concentration that is too low may even stimulate the microbial growth; since the substance is not toxic for microorganisms, they may start to metabolise it. Moreover, the wrong choice of biocides may even generate an economic loss or environmental hazard. Generally, the application of biocides, H_2S scavengers and nitrate-based treatment are one of the most effective world strategies to decrease microbiological contamination, which affects various areas of the oil and gas industry. These products have also been successfully applied to control bacterial growth in Polish natural gas wells. The issue of the influence of microorganisms and biomass on the permeability of reservoir rocks was also presented. In addition, the paper refers to biodegradation processes, that take place in the environment of drilling fluids. Also, the issue of choice of biocide/H_2S scavenger preparations for industrial applications is presented. The choice of chemicals includes efficiency tests of nanoparticles in contaminated media.

Key words: hydrogen sulphide, microorganisms, deposit waters, biocides, H_2S scavenges, permeability, nanomaterials.

STRESZCZENIE: Artykuł dotyczy kilku aspektów prac badawczych stanowiących wsparcie dla przemysłu nafty i gazu, głównie podziemnego magazynowania gazu w złożach sczerpanych i kawernach solnych. Skoncentrowano się na zagadnieniach związanych z powstawaniem skażeń siarkowodorowych w warunkach złożowych oraz na metodach ograniczania niekorzystnych zjawisk o charakterze biogennym. W obiektach magazynowania gazu duży problem stanowią aktywne bakterie redukujące siarczany z grupy SRB. Eliminacja lub ograniczenie zawartości siarkowodoru w skażonych mikrobiologicznie środowiskach jest przedmiotem wielu prac badawczych. Preparaty takie jak biocydy, inhibitory biokorozji lub neutralizatory H_2S są często stosowane do ochrony struktury złożowej, obiektów magazynowych, jak również wodnodyspersyjnych płuczek wiertniczych przed niekorzystnym oddziaływaniem bakterii. Jednym z najbardziej popularnych środków biobójczych stosowanych w przemyśle nafty i gazu są pochodne triazyny, testowane również w ramach omawianych badań biomonitoringowych. Produkty triazynowe są bardzo efektywne w procesach redukcji biomasy oraz eliminacji bakterii beztlenowych, głównie z grupy SRB. Przed każdorazowym zabiegiem przemysłowym (opartym na technologii stosowania biocydów) niezbędne jest przebadanie zawartości związków siarki w magazynowanym gazie, pobranym z różnych odwiertów eksploatacyjnych podziemnego magazynu. Konieczny jest również dobór odpowiednich preparatów oraz wytypowanie optymalnych stężeń preparatów biobójczych. Zbyt niskie stężenie może bowiem stymulować wzrost mikroorganizmów, które są zdolne do wykorzystania danej substancji w procesach metabolicznych. Ponadto niewłaściwy dobór biocydu może powodować straty i wpływać negatywnie na środowisko przyrodnicze. Generalnie stosowanie biocydów, neutralizatorów siarkowodoru i związków azotanowych stanowi jedną z najbardziej skutecznych światowych strategii ograniczenia skażeń mikrobiologicznych w wielu obiektach złożowych. Wymienione

Corresponding author: A. Turkiewicz, e-mail: *anna.turkiewicz@inig.pl*

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wyżej środki chemiczne były również z sukcesem stosowane w polskich odwiertach gazowych. W artykule przedstawiono także problem wpływu mikroorganizmów i biomasy na przepuszczalność skał zbiornikowych. Ponadto nawiązano do zjawisk o charakterze biodegradacyjnym, które mają miejsce w środowisku płuczek wiertniczych. Zaprezentowano problematykę dotyczącą wspomnianego doboru preparatów typu *biocyd/H_2S scavenger* do zastosowań przemysłowych z uwzględnieniem badań efektywności działania nanocząsteczek w skażonych mediach.

Słowa kluczowe: siarkowodór, mikroorganizmy, wody złożowe, biocydy, pochłaniacze H_2S, przepuszczalność, nanomateriały.

The presence of hydrogen sulphide in deposits and the methods for its elimination under industrial conditions

Hydrogen sulphide is an important chemical compounds with lethal effect on biological life. It is microbiologically obtained as a result of sulphate bioreduction. It is also a byproduct of the activity of many industrial plants. At deposit conditions of normal temperature and pressure hydrogen sulphate is produced as a result of sulphate reducing bacteria.

In general, microorganisms play a very important role in sulphur transformations, mainly in oxidation and reduction processes. In specific conditions, one group of microorganisms can be more active than the other and as a result of these processes, sulphites or other sulphur compounds can be accumulated or destroyed. Partial or total reduction of sulphur compounds, accompanied by H_2S formation, plays an important role in nature. This reaction takes place in anaerobic conditions in the water in rivers and lakes, sea sediments, sulphur and gypsum deposits, crude oil, bitumen and gas deposits as well as hot springs.

Sulfate reducing bacteria (Atlas, 1997; Aullo et al., 2013; Bergey's Manual, 2015; Cypionka, 2000), including *Desulfovibrio desulphuricans* as an example, have the ability to reduce a number of sulphur compounds. They reduce sulphates, sulphites, different polysulphides, colloidal sulphur, although they are not capable of reducing crystalline sulphur. In these processes, revealing an oxidation-reduction nature, sulphate sulphur is reduced to sulphides, while organic carbon or gas hydrogen is oxidised to carbon dioxide and water. The aforementioned bacteria oxidise those chemical connections whose activity on sulphates results in the release of energy.

A number of experiments with isotopic sulphur showed that H_2S may be produced as a result of bacteria reduction of sulphates (Ullmann's Encyclopedia, 2001). Isotope studies of sulphur and sulphates from hydrocarbon deposits also showed that the processes of hydrogen sulphide formation and its oxidation to sulphur occurring in Central Asia, Forecarpathia, Sicily and America follow the biological way. Therefore, microorganisms on the one hand contribute to the formation of hydrogen sulphide in the lithosphere and, on the other, different groups of microorganisms are able to eliminate it.

Bacteria oxidising H_2S, sulphur and its compounds are present in natural biocenoses. Bacteria oxidising sulphur compounds do not form a morphologically or physiologically homogenous group. Upon chemosynthesis the above-mentioned microorganisms oxidise hydrogen sulphide, elemental sulphur, thiosulphates, tetrathionates and other inorganic sulphur compounds. Subsequent strains differ from each other as regards their abilities to use these substrates. When bacteria do not have enough access to hydrogen sulphide or sulphides, the second phase of sulphur oxidation to sulphuric acid (H_2SO_4) takes place. Bacteria capable of oxidising H_2S to sulphur lead to the formation of deposits.

Some accumulated H_2S may be neutralised, as there are naturally occurring microorganisms with metabolic capabilities enabling oxidation of hydrogen sulphide present in water reservoirs of different types as well as in the deposits of hydrocarbons.

Based on the data available in the literature it can be concluded that sulphur compounds present in formation waters usually take the form of sulphates, sulphides and hydrogen sulphide. Maximum concentration of hydrogen sulphide depends on the geological composition of the deposit, its hydrogeological properties and the activity of bacteria present in this environment.

Biomonitoring studies performed at the Oil and Gas Institute – National Research Institute have focused on microbiological and chemical tests of deposit media (Figure 2), sampled from deposit facilities, underground gas storage facilities as well as on the analyses of biocidal effect of biocides/H_2S scavenger products (Raczkowski et al., 2004; Abishek, 2006; Such et al., 2007; Harrington, 2014; Weers et al., 2018; Clark et al., 2019). The tested preparations were intended for the use in industrial practice in connection with their high efficiency in eliminating hydrogen sulphide.

The effectiveness of the aforementioned solutions was verified via the industrial application method patented by the Oil and Gas Institute – National Research Institute (P.186202P, P.199583, P.392737) (Figure 1), relying mainly on triazine and chemically complex amine preparations, based on quaternary amines and aminoalcohols, in a more than a dozen UGS boreholes during recent years of exploitation.

A different way for hydrogen sulphide elimination or reduction of its content in deposit media consists in applying the method based on the biding and precipitation of hydrogen sulphide produced in the waters and regulating their pH. Another

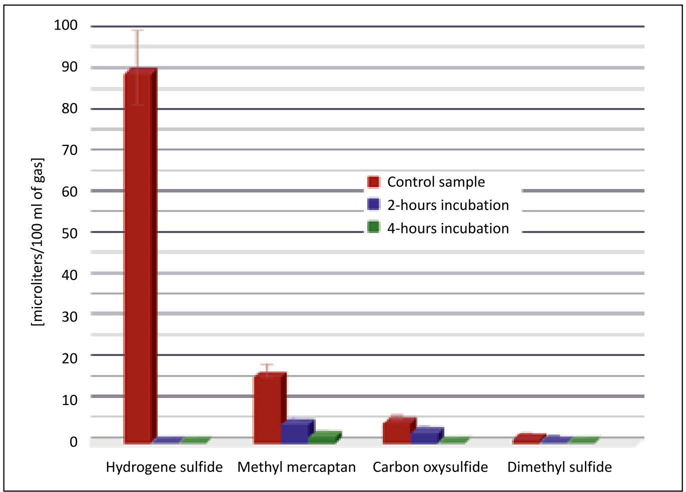

Figure 1. Example diagram showing the results of incubating the tested samples; the content of sulphur compounds in the gas tend to decrease after adding an H_2S scavenger, based on a triazine derivative

Rysunek 1. Wykres obrazuje wyniki przykładowej inkubacji próbek testowych, w których obniża się zawartość związków siarki w gazie, pod wpływem działania neutralizatora triazynowego

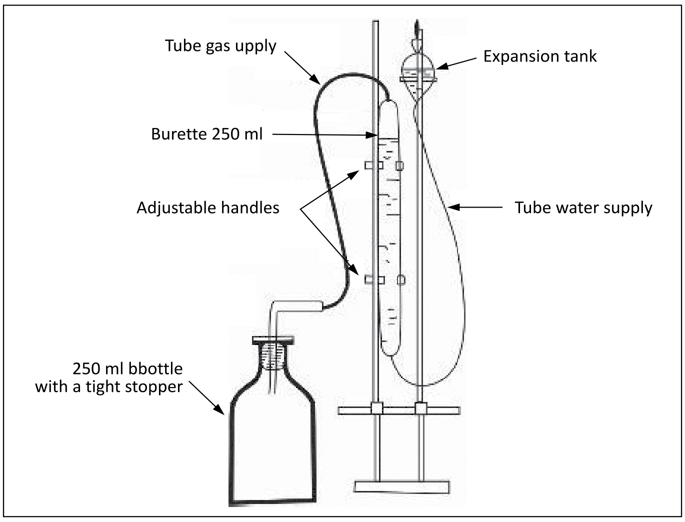

Figure 2. Laboratory equipment for biocide testing using the degasation method

Rysunek 2. Zestaw do badań testowych roztworów biocydów metodą degazacji

Figure 3. The incubation of sulphate reducing bacteria (anaerobes) isolated from formation waters of underground gas storage facilities. The photos show a 30-day incubation in a liquid medium

Rysunek 3. Inkubacja bakterii redukujących siarczany (beztlenowców) wyizolowanych z wód złożowych obiektów podziemnego magazynowania gazu. Na zdjęciach pokazano 30-dobową hodowlę w podłożu płynnym

applicable method ensuring the efficient purification of mine water containing hydrogen sulphide and iron compounds consists in subjecting them to alkalisation to pH = 8.0 by adding NH_4OH and preserving equivalent proportion of iron oxide and hydrogen sulphide connection or excess iron oxide. In the event of the excess of hydrogen sulphide connections, apart from alkalising compounds, also $FeCl_3$ solution is added to water in order to obtain further binding or re-binding of H_2S into iron sulphide (Hemme and van Berk, 2017). Subsequently, the waters containing iron sulphide need to be subjected, before their re-injection into the bore-hole, to the stabilisation process as iron sulphide, precipitating from formation waters, may cause the clogging and permeability reduction in reservoir rock.

In general, as mentioned above, sulphur compounds may be subjected to numerous transformations as a result of metabolic activity of microorganisms as well as, to a lower extent, due to abiogenic reactions. It ought to be emphasised here that the products of metabolic transformations obtained through the activity of one group of microorganisms often become substrates for the next group. The living conditions of microorganisms in environments rich in oxygen differ from environments where this element is scarce or totally lacking. Nevertheless, anaerobic environments are not lifeless and biochemical processes that take place there are triggered by numerous types of specialised microorganisms (Figure 3).

Research on preventing biodegradation in drilling fluids

In order to extend the life of fluids applied in the mining industry, methods for reducing the pace of degradation processes are applied. Reactions of destruction of drilling fluids, including in particular polymer compounds, contribute to the changes in their physicochemical properties. In order to prevent unfavourable processes and make it possible to maintain optimal rheological and technological parameters of liquids over a longer period of time, it is necessary to secure them appropriately already at the stage of preparing a given drilling fluid and then during its use.

Within the monitoring system, supervisory tests were performed at the Oil and Gas Institute – National Research Institute on water-dispersive polymer fluids, targeted at detecting the presence of aerobic and anaerobic microorganisms and biocide testing (Figures 4 and 5) (Kapusta et al., 2021). Fluids applied in numerous prospective regions of the country were subjected to microbiological and chemical tests, as were their dispersion center – i.e., base waters located in the vicinity of bore-holes. It was proved that they may become sources of undesired contamination of drilling fluid, and may lead to their downgrading and reducing of working life. Microbiological analyses of drilling fluids are continued by sampling the material while drilling new gas bore-holes within underground gas storage facilities. Biogenic contamination in drilling fluid may offer a warning sign indicating the risk of unfavourable phenomena occurring that, after some time, will constitute a decisive factor in terms of the quality of the gas stored in underground geological structures. Excessive development of biomass may cause not only gas sulphation, but also lead to biological plugging (colmatation) of porous rock spaces and biocorrosion (Enning and Garrelfs, 2014).

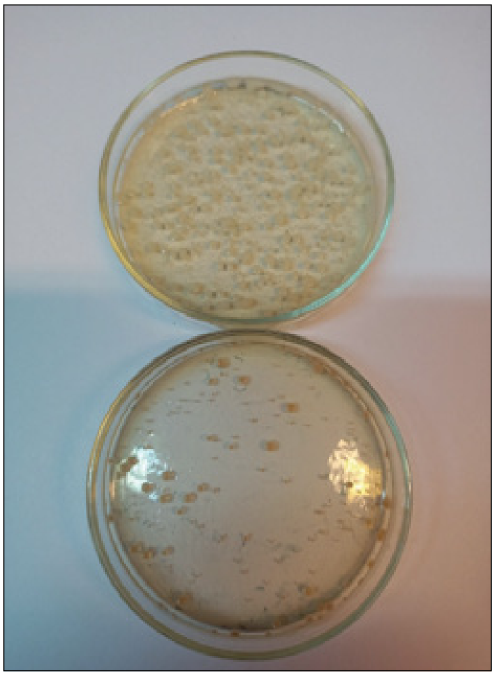

Figure 4. Colonies of aerobic bacteria isolated from polymer drilling mud

Rysunek 4. Kolonie bakterii tlenowych wyizolowanych z płuczek wiertniczych

Figure 5. Colonies of *Penicillium* sp. isolated from water-based polymer drilling mud

Rysunek 5. Kolonie grzybów pleśniowych *Penicillium* sp. wyizolowanych z polimerowych płuczek wiertniczych

Increased abundance of microorganisms in the environment of drilling fluids is possible due to the availability of water and necessary nutrients. The source of energy for microorganisms is provided by organic compounds, with polymers – in particular natural or semi-synthetic ones – as well as the materials intended for the liquidation of "drilling fluid leaks" (natural products such as nut shells, cellulose blocking agents and other organic materials used at industrial conditions).

A number of studies were carried out at the Oil and Gas Institute – National Research Institute on the degradation processes that polymers undergo when applied in drilling fluid technology. The studies were based on the strains isolated from water-dispersed drilling fluids as well as base waters sampled from wells in the vicinity of drilled holes. The results of these studies contributed to detailed recognition of biogenic phenomena taking place in the lithosphere in the course of the drilling process.

Testing the influence of solutions of chemical substances and microbiological suspensions on permeability changes in reservoir rocks

The previous chapter mentioned the phenomenon of colmatation – the clogging of rock pores under the influence of various factors. While strictly discussing the permeability of reservoir rock, which is an extremely relevant topic, one should state that the economically efficient exploitation of existing deposits of natural gas and crude oil as well as geological structures of underground gas storage requires knowledge of the mechanisms governing the flow of hydrocarbons through porous rocks (Civan, 2015). Reduced permeability is usually observed in these rocks as a result of the penetration of drilling fluid filtrate, cement slurry or the solid phase (clay, bentonite, weighting materials, drill cuttings and other) to the porous space.

Clay particles smaller than rock pores tend to settle in the porous rock surface. Colmatation may also result from the congestion of certain chemical substances or excessive and uncontrolled growth of bacteria in the porous surface (Plugge et al., 2011; Nazina et al., 2013; Liebensteiner et al, 2014; Ridley and Voordouw, 2018).

Tests have been performed on sandstone (Turkiewicz et al., 2017), characterised by similar parameters to the rock in a given deposit, e.g. an underground natural gas storage facility. When the quantity of core material from a given facility is insufficient, measurements were carried out on model sample characterised by the required permeability. The samples were prepared to be tested in cooperation with the experts from Petroleum Engineering Department of the Oil and Gas Institute – National Research Institute, where the measurements of efficient permeability for gas are carried out. Latest measurement series were performed on sandstone cores with the permeability within the range from 700 to 1200 mD.

The test solutions of biocides were performed, characterised by a wide range of volume concentrations, together with specific bacterial suspensions (Figures 6 and 7). The content of active substances in solutions (in the case of biocides) is diversified and usually amounts to from 0.5 to 10 percent concentration. In the tests directed at analysing the influence of bacterial suspension on the permeability of reservoir rock, in turn, various suspensions were tested, in which bacterial count is usually from 10^2 to 10^4 CFU/ml. A few samples of each bacterial suspension are tested in the studies.

Laboratory tests in the area of changes in the permeability of reservoir rock samples under the influence of test solutions or bacterial suspensions were performed with the use of HTHP filter press (OFI USA) modified by the Oil and Gas Institute – National Research Institute. The methodology includes filtration measurements of drilling fluids through cores in static and dynamic conditions as well as with the use of permeability meter by TEMCO. These activities make it possible to verify the scope within which a given substance at a specified concentration may cause unfavourable changes in porous rock surface as well as enable the selection of the most favourable substance from the point of view of the tested reservoir rock.

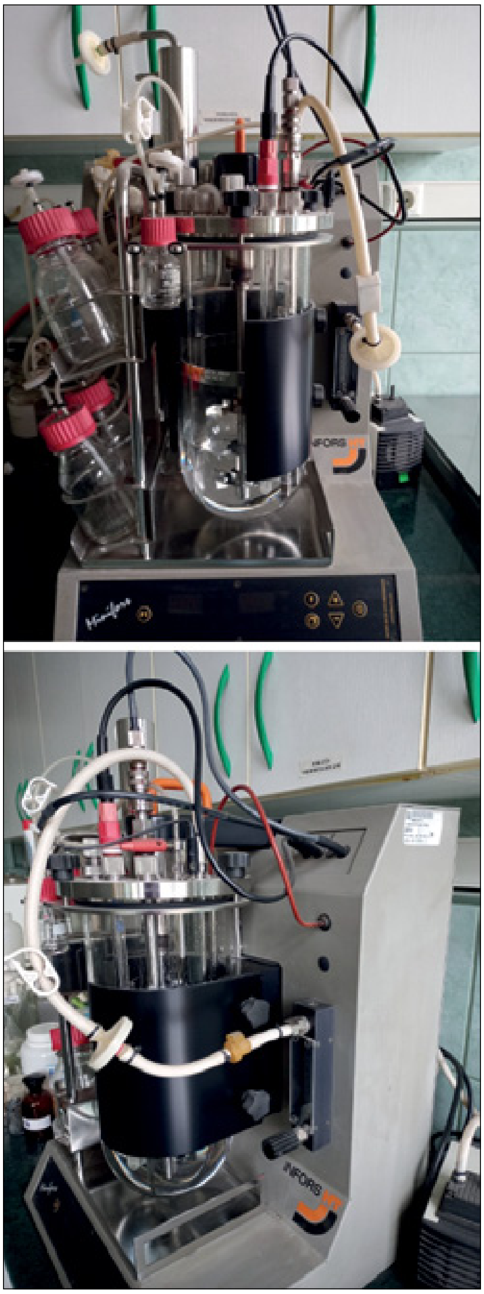

Figure 6. Bioreactor used for bacterial suspension preparation for the purpose of biocide testing

Rysunek 6. Fermentor używany do wytwarzania zawiesiny bakteryjnej do testów biocydów

The results of laboratory tests presented above are necessary while planning industrial procedures with the use of antibacterial agents or agents neutralising H_2S directly in the reservoir rock or underground gas storage facilities.

Tests of biocides in view of their application in industrial conditions

New research work includes detailed tests of biocidal products based on nano-particles (Peszke, 2014; Kachel-Jakubowska

Figure 7. Laminar workstation with air downflow used to study biological material under sterile condtions

Rysunek 7. Komora z laminarnym przepływem powietrza do pracy z materiałem biologicznym w warunkach sterylnych

et al., 2015), limiting unfavourable processes connected with microbiological contamination. Apart from biocides in different formulas, nano-materials were included in laboratory tests with the intention to assess their potential in eliminating contamination. They were tested within a wide range of concentrations as a factor counteracting biodegradation processes with reference to aerobic and anaerobic microorganisms.

Taking into account the adaptability of microorganisms to various environmental factors including specific chemical compounds, there still exists the need to search for new and efficient preparations to counteract the contamination of biogenic origin appearing in the conditions of petroleum and gas industry. These contaminations often reappear after a few months or even years from their detection in a given environment and their intensity is diversified throughout the period of exploitation. In the event of repetitive contamination of deposit waters it is necessary to schedule the works aimed at eliminating microbiological contamination and select appropriate preparation for this purpose. The aim of laboratory tests also consists in recognising the synergic effect of biocidal nano-preparations with other active substances by selecting the most optimal concentrations with reference to test samples. These tests have so far been performed on drilling fluids and deposit waters.

Summary

1. One of the main problems during the exploitation of underground gas storage facilities is the occurrence of H_2S. There are several methods of hydrogen sulphide formation prevention. The use of some biocides based on triazine derivatives or amines/amino alcohols seems to be effective and leads to a decrease in harmful bacterial activity in exploitation wells. The process of applying antibacterial substances was complemented by the use of H_2S scavengers and nitrate-based treatment in industrial conditions. What was new in this study was the testing of nanoparticles against biodegradation. Products based on nano silver were tested in a wide range of concentrations.
2. A number of studies were carried out at the Oil and Gas Institute – National Research Institute on the degradation processes affecting polymers applied in drilling fluid technology. The studies were based on the strains of aerobes and anaerobes isolated from drilling muds as well as technological waters sampled from wells. The results of these studies contributed to detailed recognition of biogenic processes taking place in the lithosphere.
3. Laboratory tests in the area of changes in the permeability of rock samples under the influence of biocide solutions or bacterial suspensions were performed with the use of HTHP filter press in static and dynamic conditions as well as with the use of permeability meter by TEMCO. In the tests directed at analysing the influence of bacterial suspension on the permeability of rock, various aerobic and anaerobic suspensions were tested, in which bacterial count is usually from 10^2 to 10^4 Colony Forming Units per millilitre.

This paper was written on the basis of original work: *Opracowanie zgłoszenia patentowego nt. sposobu oceny skuteczności działania pochłaniaczy siarkowodoru*, the work of the INiG – PIB; order number: 1845/SM/SG/2020, archive number: DK-4100-128/2020 and research work: *Przeciwdziałanie procesom tworzenia się biogennego H_2S oraz pobór i analiza gazu z KPMG Mogilno*, the work of the INiG – PIB, order number: 432/SM/SG/18/6, archive number: DK-4100-157/2020 (step 06).

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Anna TURKIEWICZ, Ph.D.
Assistant Professor of the Department of Microbiology
Oil and Gas Institute – National Research Institute
25 A Lubicz St. 31-503 Krakow
E-mail: *anna.turkiewicz@inig.pl*

Marek JANIGA, Ph.D., Eng.
Assistant Professor of the Department of Geology and Geochemistry
Oil and Gas Institute – National Research Institute
25 A Lubicz St. 31-503 Krakow
E-mail: *marek.janiga@inig.pl*