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ASSESSMENT OF ENVIRONMENTAL SAFETY OF SOLID PHASE OF DRILLING SLUDGE AFTER CENTRIFUSION SEPARATION

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Abstract

The purpose of the article is to determine the degree of environmental safety of the solid phase obtained after separating drilling sludge in a centrifuge for use as a commercial product, in particular, a filler for building materials. Samples of drilling sludge taken from wells No. 77 of the Semirenkovske field (No. 1, 2, 5, 6), Machukhi No. 54 (No. 7–10). The degree of ecological safety of the solid phase of the drilling sludge was assessed by the indicators of radioactivity (dosimeter device), mineralogical and chemical analysis. The phase composition of the samples under study was determined by the method of X-ray diffraction, the chemical composition, in particular the content of heavy metals, by the methods of X-ray fluorescence analysis and atomic absorption spectrometry. The level of radioactivity of the investigated samples of drill cuttings and its individual phases at different stages of separation in the VSD-950 centrifuge does not exceed the normative permissible threshold value ($< 0.3 \mu\text{Zv/h}$). The hypothesis of an increase in the concentration of elements characteristic of rocks, and, conversely, a decrease in the concentration of chemical elements that make up the drilling fluid and go to the liquid phase after separation in a centrifuge, has been experimentally confirmed. Heavy metals (iron, nickel, copper, lead, chromium, zinc) form a separate group of chemical elements identified in all samples. However, their content is very insignificant – no more than 0.3 %, and for some metals – at the level of traces.

Key words: environmental safety, drilling waste, drilling mud, secondary raw materials, centrifuge, dryer, heavy metals, hazard degree.

Problem statement.

The ecological situation in Ukraine can be described as critical due to non-compliance with the requirements of balanced nature management and subsoil development, in particular during oil production, moreover, scientifically based approaches to the use of low-waste technologies have not become widespread in practice [1]. The high regional concentration of oil production, refining, storage and transportation facilities on the territory of Ukraine leads to a significant man-made load on the environment [2]. One of the reasons is the irrational technological process and the accumulation of large amounts of drilling waste that comes into contact with atmospheric air, soil, surface and groundwater, polluting them, which adversely affects the health of the population [3].

Oil companies are introducing into production a variety of new technological solutions aimed at the disposal of drilling waste, but a unified method of processing drilling sludge for disposal and utilization has not been developed. One of the well-known ways of utilization of drilling sludge is its division into phases: liquid and solid with the subsequent transformation (utilization) of each of them. At the same time, drilling sludge can be used as a target product, however, to ensure environmental safety, it is necessary to analyze the cuttings, primarily for the content of heavy metals and the level of radioactivity.

Analysis of the recent researches and publications.

The practical implementation of technological solutions based on known methods of processing drilling sludge (thermal, physical, chemical, physico-chemical and biological [4]) differs significantly due to the end result and consistency of environmental safety requirements and economic efficiency indicators. Disposal of drilling sludge can be submitted according to the scheme described below. Collection, transportation and utilization of drilling waste is carried out at a special landfill, where the treatment and recycling of used drilling fluids is carried out. For further utilization of drilling sludge, various methods are used, in particular stabilization (curing) by mixing with sorbent and cement, as a result of which the sludge components are converted into sparingly soluble hydroxides. Such mixtures are effectively used as a material for the construction and repair of internal roads, dumping the foundations of drilling rigs, etc. [5].

Among the most advanced approaches to the disposal of sludge barns and disposal of drilling sludge used in Ukraine and abroad is a mobile system for treatment and processing of petroleum waste MTU 530, developed by ACS 530 (USA). The installation is mounted on the base car platform, capable of dividing oil sludge into different phases - oil, water, solids - by centrifugation of heated oil sludge. Water is suitable for further biological treatment; separated oil can be used for technical purposes; dehydrated sludge – for the production of building materials [6].

KHD Humboldt Wedag AG (Germany) has proposed a technology for separating oil sludge into phases with subsequent sludge incineration. The installation is equipped with the following components: a device for collecting oil sludge; vibrating screen - to separate the bulk from solid particles; three-phase centrifuge; separator for additional purification of the supernatant from the centrifuge; oven. The productivity of the installation is up to 15 m³/h for the initial oil sludge [7].

The most promising environmentally safe and cost-effective technology for processing drilling waste is their separation in a centrifugal field with prior physical and chemical treatment. To increase the efficiency of sludge separation into phases with their subsequent use and obtaining the target products, it is advisable to use a special installation with a tricanter. Tricanter unloads heavy liquid with an adjustable impeller under pressure, and the release of the light phase by gravity [8].

During the separation of sludges into phases, an oil-containing mixture is obtained, which can then be used as a raw material and an almost dry solid phase. After heat and chemical treatment, the solid phase is used as a mixture in the production of building materials and pavement or for the preparation of a solution during the cementing of a depleted oil well [9]. However, the use of components of drilling mud for such a purpose is possible only if its quality meets the requirements of environmental safety.

Drilling sludge is represented by particles of crushed rock, soil and drilling mud, and also includes various emulsifiers, salts and mineral additives [10]. In addition to solid forms, drilling sludge contains a small amount of liquid and solid components of drilling mud [11], and process fluids used during drilling, crushed elements of technologically destructive drilling equipment and particles of other drilling and related equipment. The mineralogical composition of drilling sludge is determined by the lithological composition of drilling rocks, so it varies significantly depending on the depth of the well. The chemical composition of drilling sludge, in turn, depends on its mineralogical composition and the properties of the reagents that are part of the drilling fluids [12]. Drilling sludges contain macronutrients (Ca, Mg, K, Na) and trace elements (Cu, Co, Fe, Mn, Zn, As, Al, Ba, Cr, Cd, Pb, Ni, Hg) [13]. Heavy metals such as Ba, Ni, Co, Cu and Zn can pose a threat to long-term mobility [14]. According to studies [15], barium is more common in drilling mud than in drilling wastes, because the main component of clay-based drilling muds is barite, while lead and zinc are more in the sludge than in drilling muds, which indicates high concentrations in drilled formations or sludge contamination by casing.

According to studies [16], one of the ways to immobilize heavy metals during the utilization of the solid phase of drilling sludge is the use of solidification. During curing, the solid preservative matrix prevents the dissolution of toxic substances as a result of contact with the components of the environment, further binding them physically and chemically. The formation of harmless sediment and the binding of inorganic

substances is carried out by introducing sorbents, in particular biosorbents from natural and agricultural wastes [17], and binders (cement, gypsum, etc.). Heavy metal ions from the sludge are converted into insoluble compounds by maintaining a high pH (up to 12). Further solidification of harmless waste leads to a stronger bond of neutralized toxic compounds, which prevents their dissolution under the influence of the environment.

However, the issue of the quality of the obtained products of drilling sludge separation remains unresolved, which requires research for each sample separately and the definition of specific methods of its utilization in order to increase the level of environmental safety.

Setting objectives. For purification of weighted and unweighted drilling fluids from excessive solids content and regeneration of the weighting agent from weighted drilling fluids in the process of drilling oil and gas wells, continuous centrifuges with screw discharge of various types of sludge are the most effective.

The purpose of the study is to determine the degree of environmental safety of the obtained solid phase for use as a marketable product.

To achieve this goal, the following research objectives are set:

- 1) to characterize the chemical and mineralogical composition of the studied samples of drilling sludge;
- 2) to analyze the level of hazardous pollutants in the samples of drilling sludge before its processing on the centrifuge and in the obtained solid phase;
- 3) to assess the degree of environmental hazard of the solid phase and provide recommendations for environmentally friendly methods and technologies for its further disposal.

The level of environmental safety of the obtained solid phase to justify the possibility of its intended use as a marketable product was assessed on the basis of a certain hazard factor in terms of quantitative content in the sludge of toxic pollutants and the value of radioactivity.

Research object and methods.

Characteristics and description of the field.

Samples of drilling sludge obtained during the drilling of wells at the Semirenkivsky gas condensate field, which is located in the Shishaki district of Poltava region, 15 km north of the district center of Shishaki and 50 km northwest of Poltava (Fig. 1, a).

At the Semirenkivsky field, 13 industrial gas condensate deposits were discovered, which are confined to the Upper Visou deposits – horizons B-16, B-17, B-18, B-19. Reservoir waters of lower coal deposits belong to the zone of very slow water exchange, have significant mineralization from 123.9 g/l to 212.2 g/l, high degree of metamorphism – 0.6–0.73 belong to the chlorocalcium type.

Table 1 – Test samples of drilling sludge

No. of the sample	Decryption of samples
1	Sludge sample No. 1 at the entrance to the centrifuge of sludge on a clay-polymer basis
2	Sludge sample No. 2 at the outlet of the centrifuge of the dry sludge fraction on a clay-polymer basis
3	Sludge sample No. 3 at the outlet of the centrifuge of the dry fraction of sludge on a hydrocarbon basis
4	Sludge sample No. 4 at the outlet of the centrifuge of the supernatant on a hydrocarbon basis
5	Sludge sample No. 5 at the entrance to the centrifuge on a clay-polymer basis
6	Sludge sample No. 6 at the outlet of the centrifuge of the dry sludge fraction on a clay-polymer basis
7	Sludge sample No. 7 at the entrance to the centrifuge sludge on the base IEP Witer II
8	Sludge sample No. 8 (without rinsing)
9	Sludge sample No. 9 (with rinsing)
10	Sludge sample No. 10 at the outlet of the centrifuge of the dry fraction of sludge on the basis IEP Witer II

Table 2 – The composition of the drilling mud on the basis IEP Witer II

Component	Characteristic	Content of the total volume of the mud, in %
diesel fuel	dispersion environment	80–90 %
water	water dispersed phase	20–10 %
E-4	emulsifier 1	6–7 %
E-5	emulsifier 2	2–3 %
H-1	solid phase water repellent	1–3 %
F-1	filtration regulator	2–4 %
organophilic bentonite	solid stabilizer, filler	5–7 %
CaCl ₂	mineralizer of the aqueous phase	25–30 % from the volume of water
M-25	carbonate colmatant	10–15 %
K-200	organic colmatant	1–2 %
CaO	neutralizer of free organic acids, pH regulator	1–2 %
hydrophobic barite	weight	to the required density

Research methodology.

Determination of radioactivity. The radioactivity of the drilling mud was evaluated by an equivalent dose, which was measured using a dosimeter-radiometer MKC-05 "Terra-P". The analysis was performed according to the procedure given in the manual for the operation of VIST (412129.012 KE) [20]. The equivalent dose (in zieverts) characterizes the effect of ionizing gamma radiation on a biological object (human). The background power level of gamma radiation is taken as 0.1 μSv/h, and the threshold level is 0.3 μSv/h.

Determination of mineralogical and chemical composition of samples. X-ray diffraction. The phase composition of drilling sludge samples was determined by X-ray diffraction (automated diffractometer DRON-4-07, SPE "Burevisnyk").

The DRON-4-07 automation system is based on a microprocessor controller that provides control of the GUR-9 goniometer and digital data transmission to a personal computer.

The following parameters were maintained during the survey CuKα radiation with a wavelength of

0.154 nm, Bragg – Brentano focusing θ-2θ (2θ – Bragg angle), current and voltage values on the X-ray tube – 20 mA and 40 kV. Samples were taken in continuous recording mode at a rate of 1°/min. and a range of angles 2θ from 15° to 105°. For pre-processing, the experimental results were transmitted directly to the software package to support the experiment DifWin-1 (LLC «Etalon PTTs»). The final identification of the crystalline phases was performed using the software package Crystallographica Search – Match (Oxford Cryosystems) with restrictions on the elemental composition of the sample by automatically comparing the results with the PDF-2 database cards, followed by manual sampling.

To determine the features and nature of the processes of leaching of chemical elements from the drilling mud, the elemental composition of the samples was identified. The study was performed by **X-ray fluorescence analysis (XRF)** on an ElvaX Light SDD. According to the results of the analysis, the values of the intensity of the peaks of chemical elements are obtained, which allows to quantitatively compare the content of a particular element in different samples.

Qualitative elemental analysis of drilling sludge samples was performed on an energy-dispersive X-ray fluorescence spectrometer ElvaX Light SDD (LLC "ELVATEKH", Kyiv), which is a modification of spectrometer ElvaX with an extended range towards light elements.

The device is designed for express qualitative and quantitative analysis of the composition of metal alloys, powders, liquids, bioassays for the content of chemical elements from Na (atomic number $Z = 11$) to U ($Z = 92$) in a wide range of concentrations. The accuracy of determining the mass fractions of metals is not lower than 0.1 %. Limits of detection of heavy metal impurities in the light matrix ≤ 1 ppm. The spectrometer is entered in the State register of measuring equipment approved for use in Ukraine under number U1411-01.

An X-ray tube with a rhodium anode was used to analyze the samples. In the study of the first group of elements (from Na to Sc) the voltage on the tube was 10 kV. There is no primary X-ray filter. To analyze the second group of elements (from Ti), a voltage of 40 kV was installed on the tube and an aluminum filter of primary X-ray radiation with a thickness of 800 μm . The current was selected automatically to achieve a load level of 50000 pulses/s.

Quantitative content of mobile forms of heavy metals (iron, copper, zinc, nickel, chromium) in drilling sludge was determined by **atomic absorption spectrophotometry**. Extracts are prepared for analysis with ammonium acetate buffer solution with a pH of 4.8 in accordance with standard methods (DSTU 4770.2: 2007, DSTU 4770.4: 2007, DSTU 4770.6: 2007, DSTU 4770.7: 2007, DSTU 4770.8: 2007). The prepared solutions were analyzed on a spectrophotometer C115-M1 (OJSC "SELMI", Ukraine) with an electrothermal atomizer. To determine each element, calibration graphs were constructed using standard element solutions. Measurements and calculations were performed on the basis of the AAS-SPECTR program. According to the results of measurements on the spectrophotometer, the content of elements in 1 kg of drilling sludge was calculated.

Statistical analysis. The statistical significance of data on the chemical composition of drilling sludge samples was assessed by analysis of variations (ANOVA). Data were considered significantly different if $P \leq 0.05$. Systematic errors are the same for all values of indicators are tracked during the experiment. This error was determined by the measurement accuracy and measurement class. The determination of random errors involved comparing the sample statistics, ie the average value of the sample with some standard value, and

finding the ratio of this difference to the standard deviation or standard error. Stata data analysis software, version 14.2 (StataCorp LLC, 2017) was used for all statistical analyzes and analysis evaluations. Each coded sample was considered independent and replicates were performed.

Research results.

Radioactivity research. The priority is to determine the radioactivity of the effective specific activity of natural radionuclides, the value of which should not exceed 370 Bq/kg (Class 1) for the use of waste for all types of construction without restrictions in accordance with NRBU-97, DBN B.1.4-1.01-97.

Based on the measurements, the values of the power of the equivalent radiation dose were obtained (table 3).

According to the results of radiological assessment, the level of radioactivity of the samples is within the normatively established (0.3 $\mu\text{Sv/h}$), so the sludge is environmentally safe.

Investigation of the phase composition of drilling sludge. Elemental composition and phase analysis of the solid phase of drilling sludge allows to determine the mineral composition and the presence of heavy metals. Based on this information, it is possible to recommend methods of waste disposal, such as use in the production of building materials, for backfilling roads, etc., i.e. its useful use, which has environmental advantages over landfilling.

For the phase composition, the following phase ratio was obtained for samples No. 2 and No. 3 (fig. 2 and fig. 3, respectively).

The content of the identified quartz phase (87 %) in the investigated sample of the solid phase obtained at the outlet of the centrifuge is due to the composition of the rock within the action of the drilling rig.

The content of aluminium oxide (10 %) is explained by the chemical composition of the drilling fluid, as the solution was used on a clay-polymer basis.

The results of X-ray phase analysis for sample №3, formed by the solid phase at the outlet of the centrifuge, show an excellent composition compared to sample №3, which is fully explained using drilling mud of other chemical composition, in particular on a hydrocarbon basis.

For this sample, the predominant phase was silicon oxide (62 %), while the quartz content decreased compared to the previous sample (26 %).

Despite the fact that the chemical formula of SiO_2 and the hexagonal crystal system are the same for both phases, the difference is in the parameters of the cells of the crystal lattices.

Table 3 – The results of measuring the radioactivity of the samples

No. of the sample	1	2	3	4	5	6	7	8	9	10
Value of radioactivity, $\mu\text{Sv/h}$	0.15	0.12	0.14	0.12	0.12	0.09	0.13	0.13	0.11	0.09

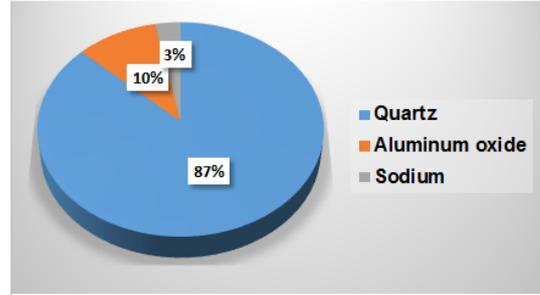
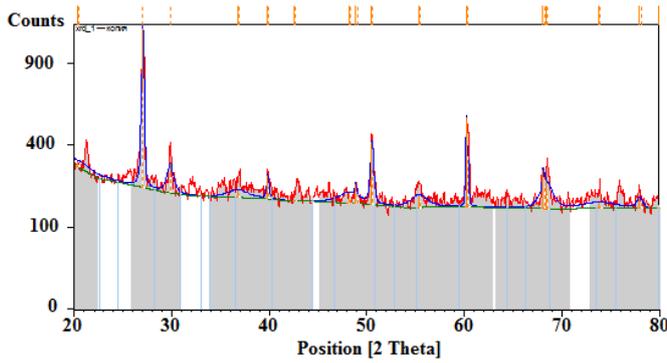


Figure 2 – Type of diffraction pattern (a) and the relationship between phases (b) for sample No. 2

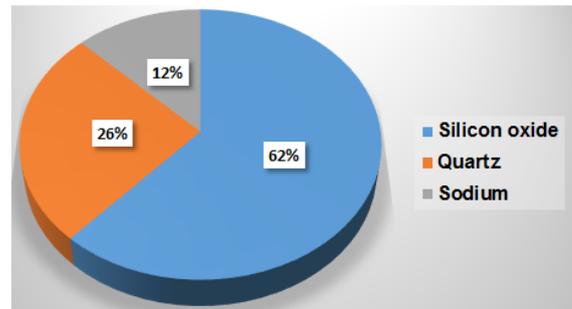
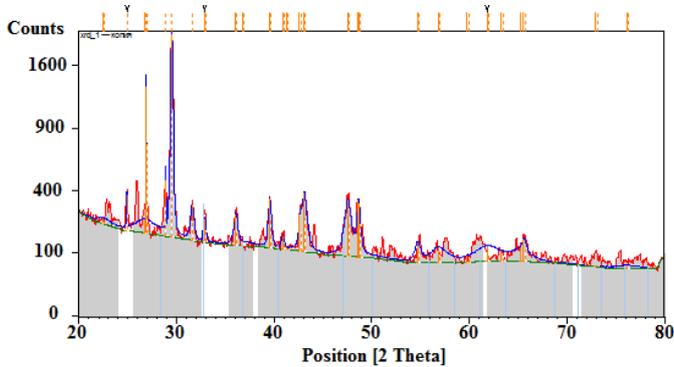


Figure 3 – Type of diffraction pattern (a) and the relationship between phases (b) for sample No. 3

Investigation of the elemental composition of the test samples. Based on the performed X-ray fluorescence analysis and the obtained results, it was established that among the chemical elements detected in the sludge a group of heavy metals formed for samples No. 1 and 2 with such elements as iron, nickel, chromium, zinc, lead has a certain toxicity.

If we compare the quantitative content of each metal, there is a tendency to increase their concentration in the sample at the outlet of the centrifuge compared to the entrance due to concentration in the solid phase, so we can assume that their content in the joint will decrease (Fig. 4 and Fig. 5). All values, except Manganese, do not exceed the established maximum concentration limits for soil.

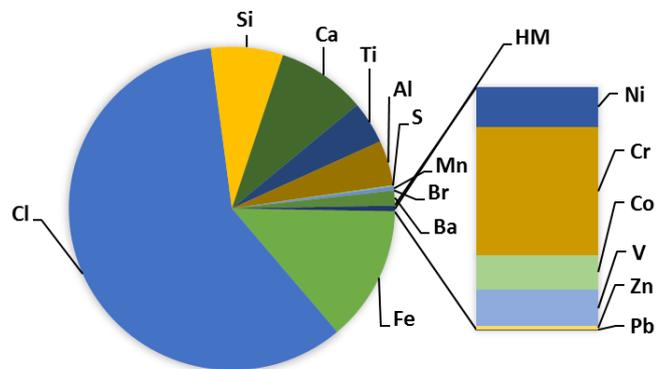
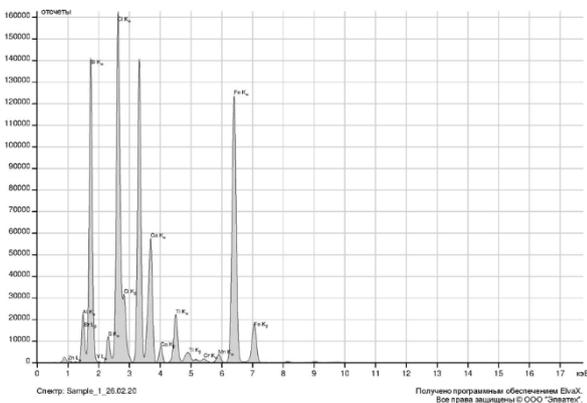


Figure 4 – Spectrogram (a) and elemental composition (b) of drilling sludge sample No. 1

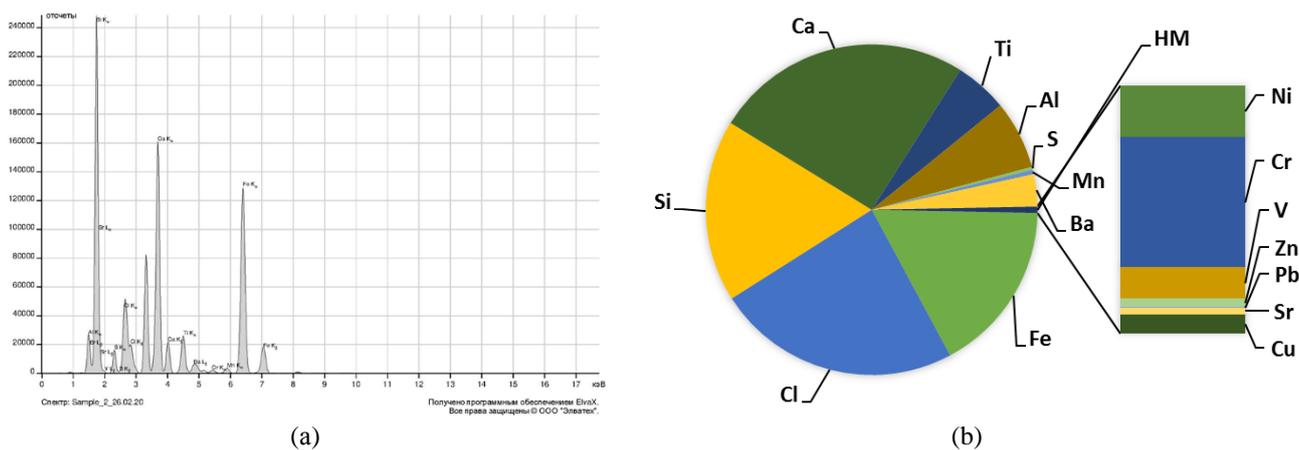


Figure 5 – Spectrogram (a) and elemental composition (b) of drilling sludge sample No. 2

Discussion.

Based on the comparison of the elemental composition of drilling sludge samples at the inlet to the centrifuge (№1) and the solid phase at the outlet of the centrifuge (№2), the hypothesis of increasing the concentration of elements characteristic of rocks and, conversely, decreasing the concentration of chemical elements are part of the drilling mud and go into the liquid phase after separation in a centrifuge.

The environmental safety assessment of drilling sludge is carried out on the basis of comparing the content of pollutants, mainly heavy metals, with the maximum permissible concentrations. This approach will prevent the danger to human health and the environment in the case of using drilling sludge as a target product.

The data of the chemical composition in Figure 4 and Figure 5 show a decrease in the concentration of chlorine (a chemical element of a clay-mineral-based drilling mud and saline solutions). At the same time, the share of silicon, iron and calcium increases, which is confirmed by the results of X-ray phase analysis with the identification of the predominant phases of the studied samples.

Individual group of chemical elements identified in all samples is formed by heavy metals. However, their content is quite insignificant – no more than 0.3 %, and for some metals – at the level of traces. It should be noted that for samples №№ 3 and 4 among heavy metals there are iron, nickel, copper, lead. The trend is similar to previous samples. Based on the performed X-ray fluorescence analysis and the obtained results, it was found that among the chemical elements detected in the sludge a group of heavy metals formed for samples №№ 7, 8 and 9 such elements as iron, nickel, copper, lead has a certain toxicity. All values do not exceed the established maximum concentration limits for soil. For sample № 10 among heavy metals there are iron, copper, lead, chromium. The trend is similar to previous samples.

Based on these results to justify the use of the obtained products after separation and drying of the sludge for a specific functional purpose, it is advisable to conduct additional research on chemical analysis of

aqueous sludge extract by indicators: cationic and anionic composition (K^+ , Na^+ , Ca^{2+} , Ba^{2+} , Cl^- , SO_4^{2-} , CO_3^{2-} , OH^-); hydrogen pH.

Such analysis is regulated by the Basel Convention, regulatory requirements for industrial water, and in the case of discharge as wastewater – the Rules of acceptance of wastewater into the city sewer and the Rules of protection of surface water from pollution.

According to the Basel Convention, sodium, potassium and calcium chlorides belong to List B, group B2040. However, since drilling sludge is not a dry waste, the vast majority of cations and anions, the content of which is due to the formulation of drilling mud, will be washed into the liquid phase. For evaluation the quality of water purification according to the methods approved in Ukraine, the analysis of the cationic and anionic composition and the determination of the hydrogen index – water with alkaline and acidic reaction is not allowed for use as technical water.

In order to implement the principles of rational nature management and environmental safety in oil areas, an effective and appropriate method is the utilization of the solid phase of drilling mud, based on the introduction of resource-saving technology of joint utilization of drilling mud and phosphogypsum to obtain the product of construction material [21].

In previous works [22] the efficiency and environmental friendliness of the use of drilling sludge and phosphogypsum for the production of gypsum concrete were scientifically substantiated, which simultaneously solves the problem of reducing anthropogenic pressure on the environment from industrial waste and limited reserves of natural raw materials.

The obtained gypsum concrete meets satisfactory ecological and technical characteristics (compressive strength and diffusion of heavy metals from gypsum concrete into the extract, respectively) due to chemical immobilization of heavy metals of drilling sludge, which contributes to the physical and chemical processes and formation of crystal structure of gypsum concrete.

Conclusions.

1. The results of chemical analysis of the studied samples of drilling sludge and its individual phases at different stages of separation in the centrifuge VSD-950 indicate the presence of chemical elements characteristic of the drilled rock and reagents of drilling mud. The results of X-ray phase analysis confirm the close correlation of the mineralogical composition of sludge samples with the chemical composition of drilling mud and the lithological structure of rock layers, which are destroyed as a result of drilling and are the predominant component of drilling sludge.

2. The level of dangerous pollutants (iron, nickel, copper, lead, chromium) in the samples of drilling sludge before its processing on the centrifuge and in the obtained solid phase differs, in particular there is a tendency to concentrate heavy metals in the solid phase, but their concentration is within valid values.

3. The coefficient of ecological danger of the solid phase is within the allowable value ($K < 1$), which allows us to conclude about the degree of ecological danger of the solid phase at the level of acceptable. It is established that the obtained solid phase does not pose a significant danger to the environment in terms of

radioactivity and heavy metal content, which are within the established standards. Despite the presence of heavy metal ions in the studied samples, recommendations on environmentally friendly methods and technologies for its further utilization were provided. The expediency of using chemical methods of utilization, which provide stabilization and solidification of the solid phase and immobilization of heavy metals and their transformation into a bioavailable form, has been confirmed.

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Аблєсва І. Ю., Пляцук Л. Д., Янченко І. О., Зінченко В. Ю., Бережна І. О., Луценко С. В., Праст А. Е. ОЦІНКА ЕКОЛОГІЧНОЇ БЕЗПЕКИ ТВЕРДОЇ ФАЗИ БУРОВОГО ШЛАМУ ПІСЛЯ РОЗДІЛЕННЯ У ЦЕНТРИФУЗІ

Мета статті полягає у визначенні ступеня екологічної безпеки твердої фази, одержаної після розділення бурового шламу у центрифугу, для використання її як товарного продукту, зокрема заповнювача для будівельних конструкцій. Дослідженням підлягали зразки бурового шламу, відібрані на свердловинах № 77 Семиренківського родовища (№№ 1, 2, 5, 6), Мачухи № 54 (№№ 3 та 4) та на свердловинах з використанням бурового розчину на основі ІЕР Witer II (№№ 7–10). Ступінь екологічної безпеки твердої фази бурового шламу оцінювали за показниками радіоактивності (прилад дозиметр), мінералогічного та хімічного аналізу. Фазовий склад досліджуваних зразків визначали методом рентгенівської дифракції, хімічний склад, зокрема вміст важких металів – методами рентгено-флуоресцентного аналізу та атомно-абсорбційної спектрометрії. Рівень радіоактивності досліджуваних зразків бурового шламу та окремих його фаз на різних етапах розділення у центрифугу ОВШ-950 не перевищує нормативно допустимого порогового значення (< 0,3 мкЗв/год). Експериментально підтверджена гіпотеза про збільшення концентрації елементів, характерних для гірських порід, та, навпаки, зниження концентрації хімічних елементів, які входять до складу бурового розчину та перейдуть до рідкої фази після розділення у центрифугу. Окрему групу хімічних елементів, визначених у складі всіх зразків, утворюють важкі метали (ферум, нікол, купрум, плюмбум, хром, цинк). Однак їх вміст досить незначний – не більше 0,3 %, а для окремих металів – на рівні слідів.

Ключові слова: екологічна безпека, відходи буріння, буровий розчин, вторинна сировина, центрифуга, осушувач, важкі метали, ступінь небезпеки.

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