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Effect of drilling sludge cleaning methods on proppant properties

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Abstract

The disposal of drilling sludge is a pressing issue in the oil and gas industry. The present study investigates the effect of three sludge cleaning methods for the Urengoy oil and gas condensate field (washing with water, washing with gasoline, and calcination at 400 °C) on the physicochemical characteristics and mechanical properties of model proppant samples. The scientific novelty of the present study lies in the description of a direct quantitative correlation between the phase composition of the model samples and their ultimate compressive strength. This correlation had not previously been demonstrated for drilling sludge from the Achimov Formation. The initial sludge was characterized using a combination of methods, including X-ray phase analysis, Fourier transform infrared spectroscopy, gas chromatography-mass spectrometry, and X-ray fluorescence analysis. It was shown that it contains 2.2 wt.% residual hydrocarbons and is a polymineral raw material. It has been established that washing with gasoline most effectively removes hydrocarbons (cleaning degree up to 97 %), while washing with water effectively removes chlorides (reduction to 0.05 %). Calcination at 400 °C does not ensure complete removal of hydrocarbons due to the coking of heavy fractions and leads to the amorphization of clay minerals. Using quantitative X-ray phase analysis of model proppant samples, a relationship between the mullite content and the ultimate compressive strength was established. The maximum strength (125.38 MPa) was achieved for the sample obtained from water-washed sludge, which is due to its high mullite content (23.5 wt.%) and minimal amount of amorphous phase. The lowest strength (74.79 MPa) was obtained for the model proppant sample made from calcined sludge, due to the destruction of clay minerals. The results of this work substantiate the choice of water washing as the optimal method for preparing drilling sludge for the synthesis of strong propping agents.

Keywords

drilling sludge; ceramic proppants; phase composition; structure formation; recycling; waste; Urengoy field; sintering; raw material cleaning

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Introduction

Currently, there is progressive development of environmental safety measures in Russia and worldwide across all industrial sectors, including the energy sector in oil and gas production [1, 2]. Contemporary environmental trends [3] in industrial development are aimed at reducing the negative impact on the environment while maintaining high production levels [4, 5]. Despite the recent trend toward a slight decline in oil production in the Russian Federation, the country continues to rank among the world's leading oil producers. The high rate of hydrocarbon exploration and production leads to an increase in well construction, which entails the generation of elevated volumes of waste [6].

Waste from hydrocarbon exploration and production is divided into the following categories: drilling waste (drilling fluids and sludge), produced water (salty water, brine brought to the surface together with oil and gas), and associated waste [7, 8]. There are no precise data on the amount of

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solid waste generated during oil production, but it is known that its volume depends on the rock geology, the depth and diameter of the well, the type of drilling fluid used, and other factors. However, there are studies that indirectly estimate this parameter. Thus, according to the estimate of the authors [7], approximately 10 % of drilling waste is solid and 90 % is liquid, with their total volume ranging from 1000 to 5000 m^3 per well. In offshore production of 229 million tons of oil, 530,000 t of waste were generated, of which 260,000 t were drilling sludge [9]. According to the calculations given in studies [10] and RD 39-133-94 "Instructions on Environmental Protection During the Construction of Onshore Oil and Gas Wells", a single well with a depth of 2 to 3.3 km and drill bit diameters of 250 and 311 mm generates from 260 to 1000 t of drilling sludge, the range of which depends on the cavernosity and rock decompaction coefficients. The accumulation of such volumes of drilling waste at specialized landfills leads to a significant reduction of large areas suitable for economic activity and to their contamination.

It is known that drilling sludge consists of fine rock particles (debris) formed when the formation is destroyed by the drill bit during drilling. According to the Federal Waste Classification Catalog, drilling sludge belongs to moderately hazardous and low-hazard waste (hazard classes III and IV) containing toxic chemical compounds. Improper handling of them creates environmental risks caused by salinization of soil horizons [8, 11, 12] and, less commonly, potential contamination of groundwater [11]. Drilling sludge also often contains heavy metals such as strontium, zirconium, zinc, chromium, and arsenic due to the use of drilling fluids and chemical reagents in field development [13-15]. Because these metals can accumulate in the soil layer and pose a significant threat to the biosphere, the use of effective methods for cleaning and disposing of drilling sludge is a prerequisite for preventing their emission and the salinization of soil horizons.

Currently, there are many scientific studies devoted to the cleaning and treatment of drilling fluids for their reuse [16-18], while significantly less attention is paid to the problem of drilling sludge disposal. Nevertheless, the following methods of their treatment can be distinguished: bioremediation – the use of microorganisms to detoxify contaminants [19] followed by the use of sludge for soil reclamation (no more than 5 wt.%) [12]; thermal methods – combustion of the hydrocarbons contained in the sludge to obtain a solid phase (ash) [20] or pyrolysis [21] to produce oil similar in composition to commercial diesel fuel; physicochemical methods – solidification of sludge by mixing with Portland cement followed by disposal [22] or centrifugation [23]. Although existing methods for treating drilling waste can significantly reduce their toxicity and, in some cases, even provide certain economic benefits, the problem of their final disposal remains pressing, since the solid phase still requires placement in sludge pits.

Thus, it is necessary to find a comprehensive approach to solving the problem of drilling sludge disposal that will make it possible not only to improve the environmental situation at oil-producing fields but also to obtain economic benefits from waste processing. According to the study [24], in addition to improving the environmental situation, the greatest economic efficiency and feasibility is achieved by processing drilling waste into construction materials. There are studies on the processing of drilling sludge into expanded clay [25], bricks [26], a cement additive [27], as well as ceramic propping materials called proppants [28, 29]. However, bauxite [28-30] and, less commonly, bentonite [31] often serve as the primary aluminosilicate raw material, which also does not solve the problem of drilling sludge disposal.

The studies on the synthesis of proppants from drilling sludge considered above practically do not consider the presence of residual components of drilling fluids or crude oil in them, which can have a significant effect on the quality of the proppants. Currently, pyrolysis of drilling waste is used as a cleaning method to separate it into fractions [29, 32], and the solid phase (drilling sludge) is then used as an additive for the synthesis of proppants, but not as the main precursor. Due to the high temperature of the pyrolysis process (above 500 °C), which exceeds the boiling points of most light and heavy hydrocarbon fractions, this method ensures almost complete removal of hydrocarbons from the drilling sludge, which is likely to have a positive effect on the mechanical properties of the proppants.

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There is a certain scientific and practical gap in the study of the effect of drilling sludge cleaning methods when it is used as the main raw material for the synthesis of proppants, as well as in the study of the impact of this process on the physicochemical characteristics of the resulting materials. In this regard, the aim of this work is to study the effect of various drilling sludge cleaning methods on the physical and mechanical properties of proppants.

Methods

Drilling sludge from the Urengoy oil and gas condensate field (Urengoy OGCF, Yamalo-Nenets Autonomous Okrug) and glass cullet were used as the main aluminosilicate raw materials for the synthesis of model proppant samples. The physical parameters of the waste, such as true density, average particle diameter, and specific surface area, are presented in Table 1. The particle size distribution of the raw material was obtained using a LASKA-TD laser analyzer (BioMedSystem, Saint Petersburg). X-ray phase analysis, X-ray spectral analysis, and infrared spectroscopy of the initial drilling sludge were carried out. Aluminum oxide (Al_2O_3) of ChDA grade (LenReaktiv, Saint Petersburg) and sodium fluoride (NaF) of ChDA grade (Krasny Khimik OJSC, Saint Petersburg) were used as modifying additives.

Table 1

Physical characteristics of waste

Material	True density, kg/m^3	Particle diameter, μm		Specific surface area, m^2/kg
		*D*50	*D*99	
Drilling sludge	2480	10.23	23.56	413
Glass cullet	2200	13.92	39.63	342

Three methods were used to clean the drilling sludge: a single washing with water or gasoline without prolonged mixing, and calcination. The sludge was washed using distilled water heated to 75 °C in a 1:3 ratio (drilling sludge and water) and AI-92 gasoline in a 1:1 ratio (drilling sludge and gasoline). Calcination was carried out in a TK.3.1100.ZF rotary tube furnace (Thermoceramics LLC, Moscow) at a temperature of 400 °C, an inclination angle of 4°, and a tube rotation speed of 2 rpm. Under these parameters, the drilling sludge remains in the maximum temperature zone for 5 min. The choice of this calcination temperature is due to the boiling point (370-390 °C) of most of the hydrocarbons present in crude oil.

To synthesize model proppant samples, the previously developed raw material mixture composition [33], was used, including, wt.%: drilling sludge – 80; glass cullet – 20; aluminum oxide – 5 (over 100); sodium fluoride – 4 (over 100); water – 5 (over 100). The introduction of Al_2O_3 is aimed at increasing the mechanical strength of model proppant samples through the formation of a mullite framework in the proppants during sintering. Glass cullet acts as a flux, slightly lowering the sintering temperature and partially filling the pores of the final product, which has a positive effect on the ultimate compressive strength. Sodium fluoride additionally reduces the sintering temperature of the proppant. The synthesis of model proppant samples was carried out according to the technology shown in Fig.1.

The Urengoy OGCF drilling sludge, pre-dried to constant mass and treated by various methods, was mixed with glass cullet, aluminum oxide, and sodium fluoride for 120 s in a TL-020 mixer (DzerzhinskTekhnoMash LLC, Dzerzhinsk). To ensure the required plasticity, the necessary amount of water was added to the mixture, followed by additional mixing for 60 s. Cubic samples with an edge length of 20 mm were formed from the resulting raw mixture without the use of excess pressure. They were then subjected to heat treatment in a TK.8.1300.N.1F chamber electric furnace (Thermoceramics LLC) at a temperature of 1100 °C with a heating rate of 13 °C/min and a holding time of 20 min. After that, the samples were cooled in the furnace to room temperature. Each series of synthesized model proppants contains four samples.

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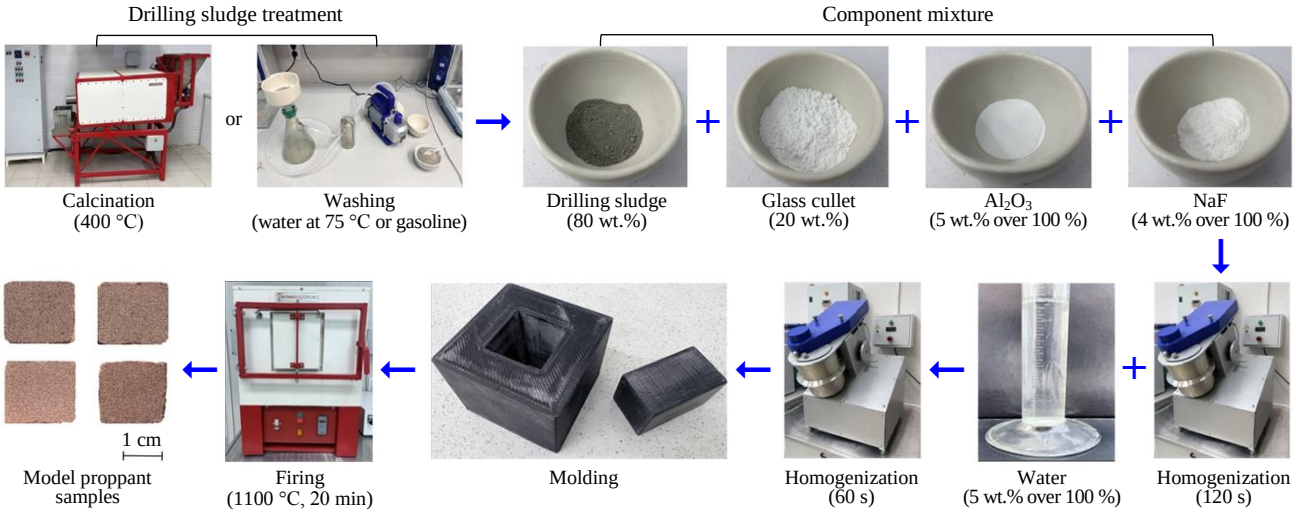


Fig.1. Technological scheme for the synthesis of model proppant samples

The choice of the firing temperature of 1100 °C is due to the combined effect of the flux additives – sodium fluoride and glass cullet. The introduction of 2-4 wt.% NaF into aluminosilicate batches makes it possible to reduce the crystallization temperature of mullite from 1200-1400 to 1050-1200 °C [34]. Sodium fluoride acts as a mineralizer: fluoride ions break the Si–O–Si and Al–O–Si bonds in the structure of the silicate melt, reduce its viscosity, and accelerate the diffusion of Al^{3+} and Si^{4+}, which promotes the nucleation and growth of mullite crystals at lower temperatures. When heated to 850-900 °C, glass cullet transforms into a silicate melt that coats the solid-phase particles. The addition of NaF additionally reduces the viscosity of this melt, creating optimal conditions for liquid-phase sintering. The results of derivatographic analysis [34] show that at a NaF content of 4 wt.%, mullitization in the melt begins at 795 °C and proceeds actively up to 918 °C. The crystallization of mullite is complete by 1100 °C, as evidenced by the maximum content of this phase (23.5 wt.%) with a minimal amount of the amorphous component (3.49 wt.%). An increase in the firing temperature to 1150 °C and above, as shown in [34] for a similar raw material system, leads to excessive melting, dissolution of mullite crystals in the melt, and foaming of the material, which is accompanied by a decrease in strength (from 73.6 to 41.2 MPa). Thus, the temperature of 1100 °C is optimal, ensuring maximum mullite formation and, as a consequence, the highest mechanical strength of the synthesized proppants.

The identification of mineral phases (XRD) of drilling sludge and model proppant samples was carried out using an ARL X'TRA diffractometer (Thermo Fisher Scientific, USA) with Cu radiation. X-ray diffraction patterns were recorded in the angular range (2θ) from 10 to 80° with a step of 0.04°. The data were interpreted using the Crystallographica Search-Match 3.1.0.2 database software package. Quantitative X-ray phase analysis was performed using MAUD (Materials Analysis Using Diffraction) software with the Rietveld method. X-ray phase analysis was carried out at the "Nanotechnologies" Shared Research Center of the M.I.Platov South-Russian State Polytechnic University (NPI).

X-ray fluorescence (XRF) analysis of drilling sludge and model proppant samples was performed on an Axios mAX sequential wavelength-dispersive vacuum spectrometer (PANalytical, Netherlands) according to the NSAM VIMS 439-PC (2010) procedure.

The functional groups of drilling sludge and model proppant samples were characterized by Fourier transform infrared spectroscopy (FTIR), which was performed on a WQF-530A FTIR spectrometer (Rayleigh, China). The spectra were interpreted in automatic mode using the built-in HAVEC database included in the MainFTOS Suite 1.18.2023.1227 software package. Measurements

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were carried out in absorption mode with 24 scans in the mid-infrared range of wavenumbers from 500 to 4000 cm^{-1}.

The methods and equipment required to determine the linear dimensions, as well as some physico-chemical characteristics (volume, density, sintering coefficient, and ultimate compressive strength) of model proppant samples, are presented in study [35]. Each recorded test value of the physico-chemical characteristics is the arithmetic mean calculated from four repeated measurements.

Hydrocarbons in the drilling sludge were identified by gas chromatography – mass spectrometry (GC-MS) using an Agilent 7890A system (Agilent Technologies, USA) equipped with an Agilent 5975C mass-selective detector (electron impact ionization, 70 eV) and an HP-5MS capillary column. Helium was used as the carrier gas at a constant flow of 1 ml/min. The compounds were identified using the NIST mass spectral library in accordance with the literature procedure [36].

In the studies conducted, the sludge treatment methods were labeled as follows: 1 – initial sludge without treatment; 2 – sludge washed with water; 3 – sludge washed with gasoline; 4 – sludge calcined at 400 °C. Model proppant samples were labeled with the corresponding number followed by the letter "p", e.g., 3p – a proppant sample made using drilling sludge washed with gasoline.

Results discussion

The identification and relative distribution of residual hydrocarbons in the drilling sludge were carried out on the initial (untreated) sludge 1 by gas chromatography – mass spectrometry using a stock solution. The solution was prepared by mixing a 1 g sample of sludge with 2 ml of dichloromethane for 16 h, after which the mixture was centrifuged. The molecular weight distribution of hydrocarbons in the sludge is presented in Fig.2 and Table 2.

According to the data obtained, the relative distribution of residual hydrocarbons in the initial drilling sludge is as follows: unsaturated hydrocarbons 66.8 % (carbon atom range C_{12}-C_{18} up to 46 %, C_{19}-C_{31} up to 20.8 %), n-alkanes C_{12}-C_{31} up to 11.3 %, isoalkanes C_{12}-C_{31} up to 16 %, and oxygen-containing compounds up to 5.3 %. The ratio of olefins to paraffins confirms the predominant prevalence of unsaturated compounds. According to the experimental data obtained, the following results are expected from the use of various treatment methods: washing with water will remove the chlorides and oxygen-containing compounds present in the sludge, while the bulk of the hydrocarbons will remain; washing with gasoline will remove nonpolar alkanes and, probably, some of the oxygen-containing compounds; calcination will remove almost all residual hydrocarbons.

To determine the amount of residual hydrocarbons in the drilling sludge, the extractability method was used, based on extraction of the initial drilling sludge before and after cleaning. A 5 g sample was mixed with 50 ml of dichloromethane at room temperature (22±1 °C) for 5 h. The resulting extract was filtered, after which the residue was washed with 50 ml of dichloromethane. The combined extract was then evaporated on a rotary evaporator at 25 °C and a pressure of 50 mmHg. The mass of the dry residue, showing the amount of residual hydrocarbons as a percentage of the mass of the initial sample, was as follows: composition 1 – 2.2; composition 2 – 1.2; composition 3 – up to 0.05; composition 4 – 1.0.

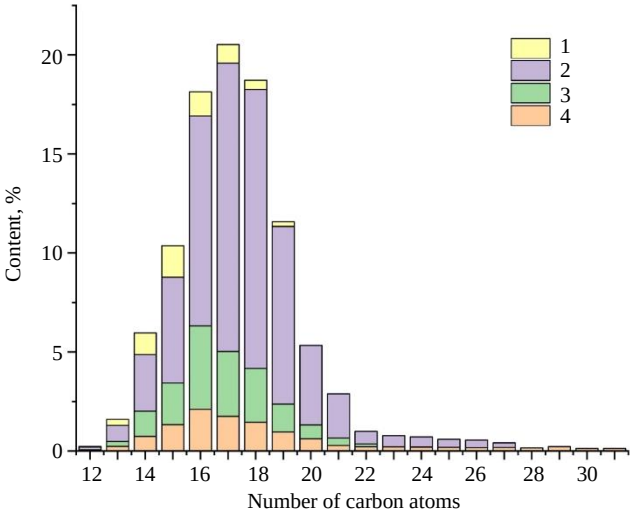


Fig.2. Molecular weight distribution of hydrocarbons in the initial drilling sludge

1 – oxygen-containing compounds; 2 – unsaturated hydrocarbons; 3 – isoalkanes; 4 – alkanes

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Table 2

Group composition of hydrocarbons in the initial drilling sludge, wt.%

Products	Group composition		Total	Iso/n-*	O/P**
	C_{12}-C_{18}	C_{19+}			
n-alkanes	7.1	4.8	11.9	6.9	2.4
Isoalkanes	13.0	3.0	16.0		
Unsaturated hydrocarbons	46.0	20.8	66.8		
Oxygen-containing compounds	5.0	0.3	5.3		
Total	71.1	28.9	100		

* Iso/n- – ratio of branched hydrocarbons to normal hydrocarbons.
** O/P – ratio of olefins to paraffins.

According to the data obtained, washing with water (composition 2) reduced the residual hydrocarbon content by 45 % (from 2.2 to 1.2 wt.%). Calcination of the sludge (composition 4) shows an almost similar result – the degree of hydrocarbon removal is about 55 %. This effect is probably due to the coking of the heavy hydrocarbon fraction as a result of the low calcination temperature (400 °C) required for the pyrolysis reaction to proceed completely. This conclusion is confirmed by the study of the authors [37], which shows that during the pyrolysis of hydrocarbons in the temperature range of 600-800 °C, liquid aromatic compounds are formed, followed by coke formation. However, at a temperature of 600 °C, coking processes already proceed quite actively, and the coke ratio is maximal at 650 °C. This conclusion correlates with the results of the study, since coking processes begin at temperatures below 600 °C. Washing with gasoline (composition 3), which is a nonpolar solvent, shows the best result in removing residual hydrocarbons – the mass of the dry residue was less than 0.05 %, which corresponds to a cleaning degree of more than 97 %.

To confirm the effectiveness of the applied treatment methods and to assess the reduction in the content of residual hydrocarbons present in the loss on ignition (LOI), the chemical composition of drilling sludge treated by various methods was analyzed. The results of the study are presented in Table 3.

Table 3

Chemical composition of drilling sludge, wt.%

Composition	SiO_2	Al_2O_3	CaO	MgO	BaO	Na_2O	K_2O	TiO_2	Fe_2O_3	MnO	P_2O_5	Cl	SO_3	LOI	∑
1	59.47	14.92	4.01	1.31	0.36	2.34	2.34	0.60	4.10	0.08	0.14	1.43	0.82	8.09	100
2	60.45	16.44	4.17	1.56	0.31	1.75	2.81	0.67	4.81	0.06	0.17	0.05	0.23	6.52	100
3	60.05	16.62	4.00	1.58	0.31	1.93	2.68	0.65	4.83	0.08	0.15	0.48	0.19	6.44	100
4	60.27	15.77	4.23	1.62	0.33	2.18	3.35	0.57	5.32	0.11	0.17	1.12	0.27	4.68	100

According to the data obtained, the drilling sludge from the Urengoy OGCF belongs to low-calcium aluminosilicate waste with a predominant content of silicon and aluminum oxides totaling 74.39 %, which is characteristic of terrigenous deposits of the Achimov Formation. The elevated chloride (Cl^-) content is explained by the use of sodium chloride (NaCl) or potassium chloride (KCl) reagents in the drilling fluid to inhibit clay swelling. The presence of barium oxide in the drilling sludge is due to the use of barite ($BaSO_4$) as a weighting agent for the drilling fluid. It should also be noted that the loss on ignition is high, amounting to more than 8 %. It includes residual hydrocarbons, the decomposition of carbonates, and the removal of chemically bound water.

Washing with water at 75 °C (composition 2) effectively removed some of the chlorides and sulfates. At the same time, the total LOI content decreased by 19.5 %, which correlates with the extractability data, since water removes hydrocarbons with 45 % efficiency. Washing with gasoline (composition 3) shows similar results, except for a higher chlorine concentration, while the other parameters are similar to those of composition 2. Calcination of the sludge (composition 4) shows the best results

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in reducing the LOI, probably due to the removal of chemically bound water and the partial removal of the volatile hydrocarbon fraction. A comparative analysis of drilling sludge treatment methods revealed their selectivity of cleaning: washing with water effectively removes chlorides but is worse at removing the organic fraction; gasoline effectively removes residual hydrocarbons but is worse at removing chlorides; calcination at 400 °C probably effectively removes chemically bound water, while a high content of chlorides and organic matter remains.

To determine the functional groups of drilling sludge treated by various cleaning methods, FTIR spectroscopy was carried out, the results of which are presented in Fig.3. In all absorption spectra, stretching vibrations of the Si–O–Si and Si–O–Al bonds characteristic of aluminosilicate materials are observed in the broad mid-frequency regions of 1200-900 and 550-500 cm^{-1}. The presence of symmetric stretching carboxylate groups in the 1350-1530 cm^{-1} region indicates the presence of both nonpolar hydrocarbons and residual carboxylic acid salts, which also correlates with the chromatography results. Their retention is due to the fact that polar organic salts do not dissolve in gasoline, since it is a nonpolar solvent. It should be noted that the intensity of the carboxylate group peaks is identical for washing with gasoline and with water, which in turn is a polar solvent. This is probably due to the fact that washing with water was carried out once without prolonged mixing. In the opposite case, the use of prolonged mixing would reduce the content and degree of absorption of the carboxylate groups similarly to calcination (composition 4).

A narrow absorption band at 2300-2400 cm^{-1}, corresponding to atmospheric carbon dioxide CO_2, is also confirmed by the study of the authors [38] (CO_2 doublets at 2360 and 2340 cm^{-1}). In the region of 2800-2950 cm^{-1}, the spectra of the initial sludge (Fig.3, line 1) show stretching vibrations of C–H methylene bonds corresponding to n- and isoalkanes, which were also identified by GC-MS. For the treatment methods (Fig.3, lines 2-4), the intensity of the C–H bonds is lower, which also confirms the effectiveness of cleaning. The broad, intense band of O–H stretching vibrations at 3600-3300 cm^{-1} in sludges 1-3 includes structural (chemically bound) water of clay minerals (e.g., montmorillonite) or partially adsorbed water. The most significant changes in the IR spectra occur during calcination of the sludge: a decrease in intensity and an increase in the width of the shoulder of the Si–O–Si (1200-900 and 550-500 cm^{-1}), associated with the partial transition of silicates to the amorphous phase; a decrease in the intensity of the absorption bands of carboxylate groups (1350-1530 cm^{-1}) with the release of CO_2 or the formation of coke due to thermal decomposition of some carboxylates; as well as the removal of the C–H and O–H bands associated with the decomposition of most hydrocarbons and the removal of chemically bound water from the crystal lattice of clay minerals.

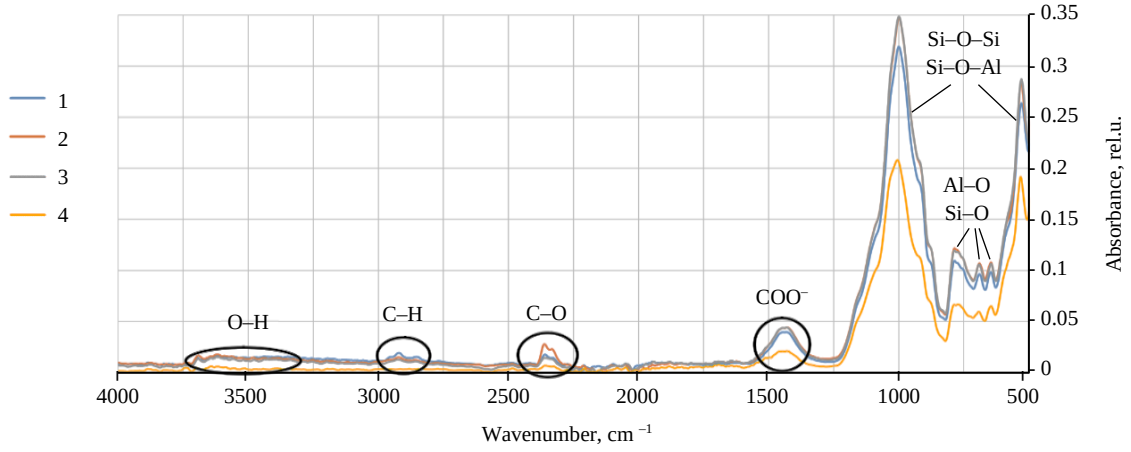


Fig.3. IR spectra of drilling sludge

1 – initial sludge without treatment; 2 – sludge washed with water; 3 – sludge washed with gasoline; 4 – sludge calcined at 400 °C

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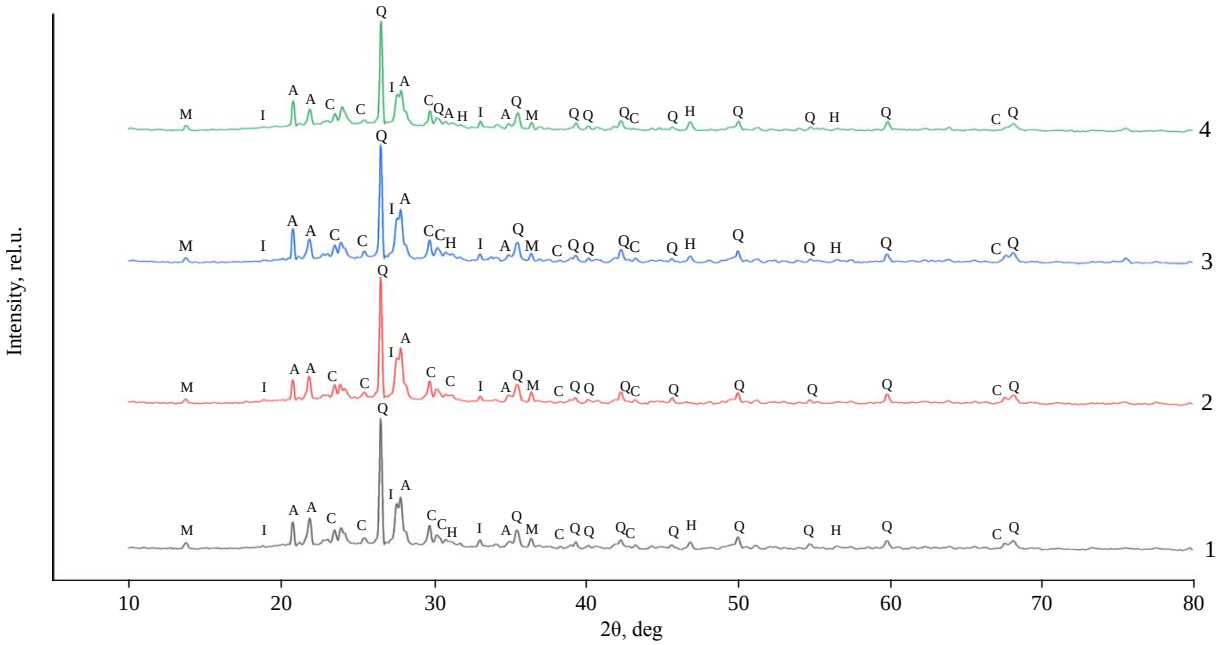


Fig.4. X-ray diffraction patterns of drilling sludge

1 – initial sludge without treatment; 2 – sludge washed with water; 3 – sludge washed with gasoline; 4 – sludge calcined at 400 °C
Q – quartz (SiO_2); M – montmorillonite $(Na,Ca)_{0.33}(Al,Mg)_2(Si_4O_{10})(OH)_2 \cdot nH_2O$; H – halite (NaCl); A – albite ($Na[AlSi_3O_8]$); I – illite $(K_{0.75}(H_3O)_{0.25})Al_2(Si_3Al)O_{10}((H_2O)_{0.75}(OH)_{0.25})_2$; C – calcite ($CaCO_3$)

To identify the structure-forming minerals of the drilling sludge, X-ray phase analysis was carried out, the results of which are presented in Fig.4. The X-ray diffraction patterns obtained by qualitative X-ray phase analysis show similar results. Compositions 1-3 have a relatively equal degree of crystallinity and peak positions. The only difference is the absence of halite peaks in composition 2, which confirms the effectiveness of hot water washing. It should be noted that the identification of halite was recorded at the sensitivity limit of the diffractometer. Fundamental changes are characteristic of composition 4: calcination of drilling sludge reduces the intensity of all peaks, with the greatest effect on the montmorillonite peak, which is absent at 20.1-20.3° 2θ. In addition, a slight increase in the amorphous halo is observed at 19-32° 2θ. Quantitative X-ray phase analysis obtained using the MAUD software is presented in Table 4.

A comparative analysis of the quantitative X-ray phase composition of the treated samples confirmed the effectiveness of halite removal by washing with water without affecting the other phases, which correlates with the results of previous studies. Washing with gasoline also partially reduces the amount of halite and the amorphous phase, but the decrease in the latter is more likely due to an error of the calculation method than to its washing out, since organic solvents are not capable of interacting with the silicate or aluminosilicate structure. Calcination of the sludge at 400 °C leads to severe destruction of clay layered minerals – montmorillonite and partially illite, which contain chemically bound water. The increase in the amorphous phase content is also associated with their decomposition and the partial transition of the decomposition products into the amorphous component. Quartz, albite, and calcite retain their crystalline structure, which confirms their thermal stability at 400 °C. In general, the qualitative and quantitative X-ray phase analysis carried out fully correlates with the results of previous studies.

Table 4

Quantitative X-ray phase analysis of drilling sludge, wt.%

Phase	1	2	3	4
Quartz	62.63	63.73	63.64	69.04
Montmorillonite	10.02	10.78	11.11	2.03
Halite	2.81	–	1.01	1.52
Albite	6.01	6.38	6.06	7.11
Illite	11.51	11.76	11.62	8.12
Calcite	4.01	3.92	4.03	4.06
Amorphous	3.01	3.43	2.53	8.12
∑	100	100	100	100

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Based on the technology and methods for calculating the physical and mechanical characteristics, model proppant samples were synthesized: 1p – based on untreated sludge; 2p – based on sludge washed with water; 3p – based on sludge washed with gasoline; 4p – based on sludge calcined at 400 °C. Their appearance before and after firing and their average properties are presented in Fig.5 and Table 5.

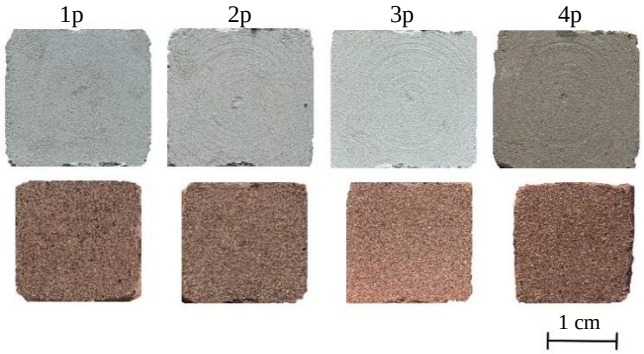


Fig.5. Appearance of model proppant samples before (upper row) and after (lower row) firing

Table 5

Physical and mechanical properties of model proppant samples

Composition	Density, kg/m^3	Ultimate compressive strength, MPa	Sintering coefficient
1	1771±40	91.17±2.53	1.27±0.05
2	1865±16	125.38±3.71	1.35±0.01
3	1884±48	99.51±4.67	1.39±0.05
4	1889±32	74.79±3.94	1.41±0.04

The obtained physical and mechanical properties of the model proppant samples fully correlate with the results of XRF, FTIR, and XRD analyses. The maximum compressive strength (125.38±3.71 MPa) corresponds to the model proppant samples synthesized using drilling sludge washed with hot water. The high mechanical characteristics are probably due to the removal of halite from the sludge structure while preserving the crystalline structure of all clay minerals, which is confirmed by the disappearance of NaCl peaks from the X-ray diffraction pattern (see Fig.4) and the quantitative phase content (Table 5). Washing with gasoline ensures the removal of hydrocarbons but retains halite, which leads to a decrease in the strength of the model proppant samples by an average of 26 % (99.5 MPa) relative to composition 2. At the same time, composition 3 shows a 9 % increase in strength relative to composition 1. The lowest strength is shown by the sample using calcined drilling sludge (74.79±3.94 MPa). The low strength characteristics are due to the retention of NaCl and the destruction of the clay mineral montmorillonite, which is necessary to ensure optimal plasticity of the ceramic mass. When synthesizing proppant granules, compliance with this condition is of great importance, which is confirmed by the study of the authors who add clay to a low-plasticity mixture based on bauxite and fly ash [39]. The change in color of the model proppant samples from light gray to a reddish hue is due to the formation of hematite (Fe_2O_3) as a result of the oxidation of iron to Fe^{3+} during firing.

To identify the structure-forming minerals of the model proppant samples, X-ray phase analysis was carried out, the results of which are presented in Fig.6. Quantitative X-ray phase analysis obtained using the MAUD software is presented in Table 6.

During high-temperature treatment of drilling sludge and glass cullet, new crystalline phases of mullite, anorthite, and hematite were formed. The formation of anorthite proceeds according to the reactions

$$CaCO_3 \rightarrow CaO + CO_2\uparrow;$$

$$CaO + Al_2O_3 + 2SiO_2 \rightarrow CaAl_2Si_2O_8.$$

Calcite and aluminosilicate minerals interact in the reaction of anorthite formation. When heated to 700-850 °C, calcite decomposes into calcium oxide and carbon dioxide. At the same time, clay minerals also decompose, releasing silicon and aluminum oxides, which then react with calcium oxide to form anorthite.

Hematite is formed as a result of the sequential oxidation of iron compounds through the intermediate formation of magnetite (Fe_3O_4) according to the reactions

$$6FeO + O_2 \rightarrow 2Fe_3O_4;$$

$$4Fe_3O_4 + O_2 \rightarrow 6Fe_2O_3.$$

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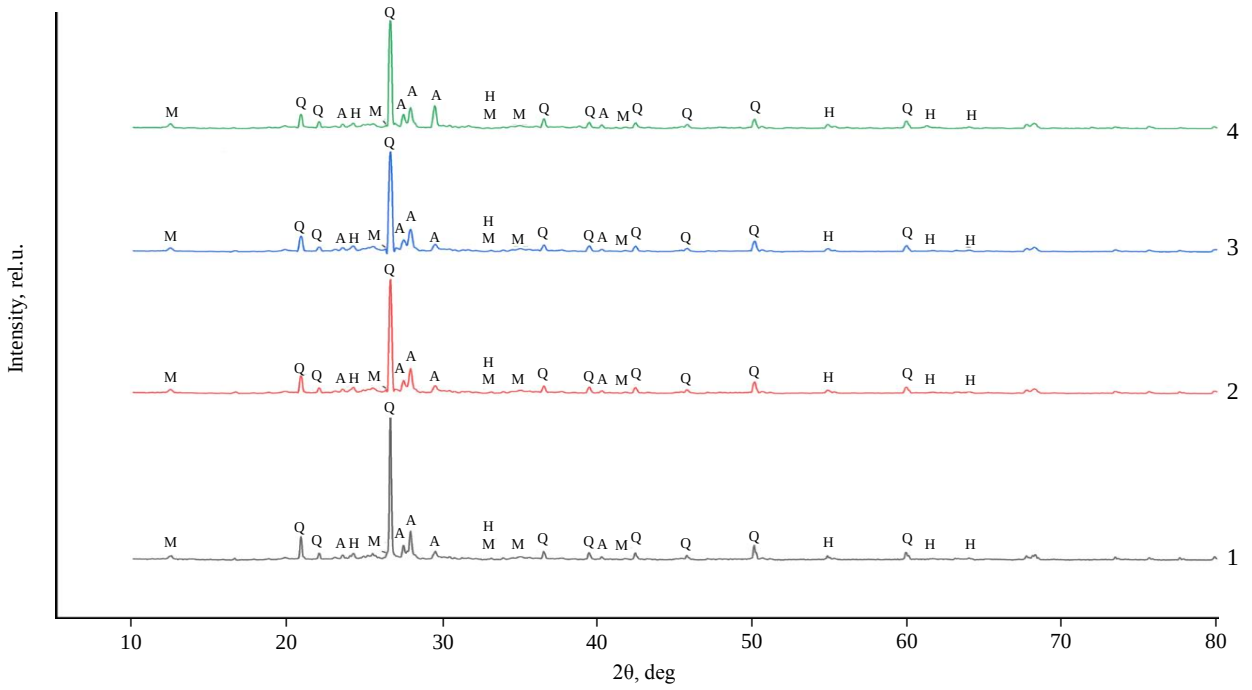


Fig.6. X-ray diffraction patterns of model proppant samples
1 – 1p; 2 – 2p; 3 – 3p; 4 – 4p
Q – quartz (SiO_2); M – mullite ($Al_6Si_2O_{13}$); A – anorthite ($CaAl_2Si_2O_8$); H – hematite (Fe_2O_3)

Table 6

Quantitative X-ray phase analysis of model proppant samples, wt.%

Phase	1p	2p	3p	4p
Quartz	63.78	57.03	60.13	66.86
Mullite	15.82	23.43	18.67	10.06
Anorthite	7.65	9.27	8.34	6.41
Hematite	5.1	6.78	7.13	7.59
Amorphous	7.65	3.49	5.73	9.08

The obtained X-ray phase analysis results revealed a direct dependence of the ultimate compressive strength of model proppant samples on the mullite content. Composition 2p is characterized by the maximum mullite content (23.43 %) and the minimum amorphous phase content (3.49 %), which ensures maximum strength. A similar dependence is confirmed by studies of other authors [40, 41]. The removal of chloride compounds and the preservation of clay minerals probably create optimal conditions for mullite formation. Composition 3p occupies an intermediate position in terms of mullite and amorphous phase contents, as well as ultimate compressive strength. A dependence has been established: an increase in mullite content and a decrease in the amorphous phase make it possible to obtain proppants with the highest strength.

The mechanism of mullite formation in the synthesized model proppant samples includes several stages. When heated above 900 °C, clay minerals are dehydroxylated and destroyed to form a metastable amorphous aluminosilicate phase according to the simplified formulas

$$\text{Montmorillonite} \rightarrow Al_2O_3{\cdot}2SiO_2\ \text{(amorphous)} + R_2O + XO + H_2O\uparrow;$$

$$\text{Illite} \rightarrow Al_2O_3{\cdot}2SiO_2\ \text{(amorphous)} + K_2O + H_2O\uparrow,$$

where XO are alkaline earth metals (Ca, Mg); R_2O are alkali metals (Na, K).

The introduction of additional Al_2O_3 (5 wt.% over 100 %) shifts the local chemical composition toward a region favorable for the crystallization of primary mullite by a solid-phase mechanism

$$Al_2O_3 + 2SiO_2 \rightarrow 3Al_2O_3{\cdot}2SiO_2.$$

Glass cullet containing amorphous SiO_2 and Na_2O acts as a flux. At a temperature of 1100 °C, it forms a silicate melt that promotes the dissolution of quartz and residual aluminosilicates, accelerating

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the diffusion of Al^{3+} and Si^{4+} and lowering the activation energy of mullite crystal nucleation. Sodium fluoride additionally reduces the sintering temperature by reacting with SiO_2 to form gaseous SiF_4. At the same time, the released Na_2O additionally reduces the viscosity of the melt. The combined action of the fluxing components (glass cullet and NaF) makes it possible to nucleate mullite crystals at 1100 °C, whereas temperatures of 1300-1400 °C are usually required for its formation.

In composition 2p, the preservation of the crystalline structure of clay minerals and the absence of chlorides ensure maximum reactivity of the aluminosilicates, which is confirmed by the high mullite yield (23.43 %) and the minimal amorphous phase content (3.49 %). In composition 4p, preliminary calcination at 400 °C leads to partial dehydration and amorphization of the clay minerals even before the high-temperature sintering stage. During subsequent firing, they do not fully recrystallize into mullite but remain in the form of inert aluminosilicate glass, which is confirmed by the low mullite yield (10.06 %) and the high amorphous phase content (9.08 %).

The effect of residual hydrocarbons on the structure and mechanical properties of model proppant samples is diverse. In composition 1p, firing causes pyrolysis of organic matter with the release of gases (CO_2, H_2O, CH_4), which leads to the formation of porosity and microdefects due to local overstress. This is confirmed by the decrease in compressive strength (91.17 MPa) compared to composition 2p (125.38 MPa). Partial removal of hydrocarbons by water reduces gas release, but the remaining organic matter is probably localized in large pores and does not have a critical defect-forming effect, provided that gases can be freely released. Conversely, washing with gasoline almost completely eliminates gas release, but the strength (99.51 MPa) does not reach the level of composition 2p due to the remaining chlorides (0.48 %), which can cause partial destruction during firing. In composition 4p, coking of heavy hydrocarbons occurs – the solid carbonaceous residue hinders sintering and mullite formation and also creates stress centers that lead to the minimum strength (74.79 MPa). Thus, optimal sludge preparation must ensure not only maximum removal of hydrocarbons but also complete removal of chlorides, as well as preservation of the crystalline structure of clay minerals and the absence of thermal degradation, which together are necessary conditions for the successful synthesis of proppants.

Washing drilling sludge with water is recommended as the optimal method for preparing drilling sludge, ensuring the removal of chlorides, the preservation of clay minerals, and the production of high-strength (up to 125 MPa) ceramic proppants for their further use as propping agents. Future research is planned in the following areas: the study of mechanochemical activation of the main precursors and its effect on the properties of proppants; the selection of more widespread auxiliary raw materials similar to glass cullet; the reduction of proppant density; and granulation and assessment of the compliance of their properties with regulatory documents.

Conclusion

As a result of the studies carried out, it has been established that the method of cleaning drilling sludge from the Urengoy oil and gas condensate field significantly affects the physical and mechanical properties of the synthesized model ceramic proppant samples. It has been shown that the initial drilling sludge contains 2.2 wt.% residual hydrocarbons and chlorides (1.43 wt.% Cl), and is also a polymineral aluminosilicate raw material with a predominance of quartz, montmorillonite, and illite.

The following selectivity of cleaning methods was revealed:

• washing with water heated to 75 °C most effectively removes chlorides (reduction of Cl to 0.05 wt.%) while preserving the crystalline structure of clay minerals; the degree of hydrocarbon removal is 45 %;

• washing with gasoline ensures maximum removal of hydrocarbons (cleaning degree up to 97 %), but is less effective with respect to chlorides (0.48 wt.% Cl);

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• calcination at 400 °C not only fails to remove hydrocarbons completely (residue1.0 wt.%) due to the coking of heavy fractions, but also leads to the destruction of clay minerals and an increase in the amorphous phase to 8.12 % (versus 3.01 % in the initial sludge).

By quantitative X-ray phase analysis of model proppant samples, a direct correlation was established between the mullite content and the ultimate compressive strength. The maximum strength (125.38 MPa) was achieved for composition 2p based on water-washed sludge. In this case, the mullite content was 23.43 % with a minimal amorphous phase content of 3.49 %. The minimum strength (74.79 MPa) was recorded for composition 4p based on sludge calcined at 400 °C, due to the low mullite content (10.06 %) and the high amorphous component content (9.08 %).

The practical significance of this work lies in the scientific justification of the choice of water washing as the optimal method for preparing drilling sludge for the synthesis of high-strength (up to 125 MPa) proppants suitable for use as propping agents. This makes it possible not only to dispose of class III and IV hazardous drilling waste but also to involve it in the production process and obtain an economically valuable product, while reducing the load on sludge pits and soil horizons.

Future research directions include the selection of more affordable and widespread analogues of glass cullet as an auxiliary additive; the development of methods for reducing proppant density; granulation of the resulting compositions and assessment of their compliance with the regulatory requirements for proppants.

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