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RESEARCH ARTICLE

Environmentally safe sorbent from ash-and-slag waste of heat power engineering

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Abstract

Objectives. *To determine the physical and chemical properties (bulk density, ash content, total pore volume, abrasion, humidity, sorption capacity) of sorbent based on ash-and-slag waste from heat power engineering, calcined and modified with a Tiprom K organosilicon water repellent.*
Methods. *The physicochemical properties of the modified sorbent were determined using an experimental method according to the methods of regulatory documents on equipment verified and certified in the prescribed manner.*
Results. *Ash and slag taken from the ash dump of the Novocherkasskaya GRES power station were dried, then calcined at a temperature of 600°C for 30 min and modified with a Silor hydrophobizing silicon-containing liquid (HSL). The modifier/ash ratios (by weight) were 1:20, 1:10, 1:5, 1:3, and 1:2. The optimal ratio was 1:5 at a sorption capacity with respect to hexane of 0.86 g/g. The modification temperature was optimized in the temperature range of 110–200°C. The optimal approach it to dry samples at 160°C to constant weight. At a temperature of 200°C, sintering of the material was observed. The analysis of HSL modifiers was carried out in terms of the price/sorption properties ratio. The following were considered as HSL: Silor, HSL-11BSP, HSL 136-157M, PROFILUX, Tiprom K, Tiprom U. The optimal modifier Tiprom K was selected. The physicochemical properties of the modified sorbent obtained at a ratio of 1:5 (by weight) and dried at 160°C were experimentally determined. The sorption properties were studied on the water surface with respect to various oil products: fuel oil, kerosene, AI-92 gasoline, nefras, oil sludge, and n-hexane. The smallest sorption capacity was obtained with respect to n-hexane, amounting to 0.86 g/g. During the experiment, it was found that half of the sorption capacity*

was filled with oil in the first minutes of contact. Complete sorption time was 30–40 min for relatively light hydrocarbons (n-hexane, AI-92 gasoline, kerosene, nefras), 40–60 min for oil sludge, and more than 60 min for fuel oil. Experiments established that the sorption process does not depend on the matrix (salinity) of water. A visual assessment of the color intensity of the residual spot of oil sludge allowed a conclusion to be made about a significant content of oil products in the case of sorption of oil sludge by quartz sand based on the residual yellow layer of oil sludge. In the case of sorption of oil sludge by calcined and modified sorbents, the residual oil products were insignificant. A comparative analysis of data on the effectiveness of the developed sorbent and currently available analogues based on sludge and slag is presented.

Conclusions. *The next physicochemical properties of the sorbent modified with HSL Tiprom K were determined: bulk density was 0.621 g/mL, ash content was 97.1%, total pore volume by water was less than 0.05 mL/g, attrition was 8.8%, humidity was less than 0.5%; sorption capacity, in g/g: for n-hexane, 0.86; for AI-92 gasoline, 0.89; for nefras, 0.93; for kerosene, 0.99; for oil sludge, 1.18; for fuel oil, 1.46. The efficiency of cleaning a solid surface from oil sludge with a calcined sorbent was 97%, and with a modified sorbent 95%. The modified sorbent has high buoyancy when saturated with oil products and the ability to retain them for a long time.*

Keywords: *sorbent, ash-and-slag waste, thermal power engineering, oil products, water-repellent silicon-containing liquids*

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НАУЧНАЯ СТАТЬЯ

Экологически безопасный сорбент из золошлаковых отходов теплоэнергетики

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Аннотация

Цели. *Определение физико-химических свойств (насыпной плотности, зольности, суммарного объема пор, истираемости, влажности, сорбционной емкости) сорбента на основе золошлаковых отходов теплоэнергетики, прокаленного и модифицированного кремнийорганическим гидрофобизатором Типром К.*

Методы. *Физико-химические свойства модифицированного сорбента определены экспериментальным методом по методикам нормативных документов на оборудовании, поверенном и аттестованном в установленном порядке.*

Результаты. *Золошлак, отобранный на золоотвале Новочеркасской ГРЭС, высушен, прокален при температуре 600°С в течение 30 мин и модифицирован гидрофобизирующей кремнийсодержащей жидкостью (ГКЖ) марки Силор. Исследованы соотношения*

модификант/зола (по массе) 1:20, 1:10, 1:5, 1:3, 1:2. Оптимальным принято соотношение 1:5 при сорбционной емкости по отношению к н-гексану 0.86 г/г. Проведена оптимизация температуры модификации в интервале температур 110–200°С. Наиболее оптимальным является высушивание образцов при 160°С до постоянной массы. При температуре 200°С наблюдалось спекание материала. Выполнен анализ модификаторов ГКЖ по соотношению цена/сорбционные свойства. В качестве ГКЖ рассмотрены марки: Силор, ГКЖ-11БСП, ГКЖ 136-157М, PROFILUX, Типром К, Типром У. Выбран оптимальный модификатор марки Типром К. Экспериментально определены физико-химические свойства модифицированного сорбента, полученного при соотношении 1:5 (по массе) и высушенного при 160°С. Изучены его сорбционные свойства на водной поверхности по отношению к различным нефтепродуктам: мазуту, керосину, бензину марки АИ-92, нефрасу, нефтешламу и н-гексану. Наименьшая сорбционная емкость получена по отношению к н-гексану, которая составила 0.86 г/г. В ходе эксперимента установлено, что половина величины сорбционной емкости заполнена нефтепродуктом в первые минуты контакта. Время полной сорбции составило 30–40 мин для относительно легких углеводородов (н-гексан, бензин АИ-92, керосин, нефрас), 40–60 мин для нефтешлама и более 60 мин для мазута. Экспериментально выявлено, что процесс сорбции не зависит от матрицы (солености) воды. При визуальной оценке по интенсивности окраски остаточного пятна нефтешлама сделан вывод о значительном содержании нефтепродуктов в случае сорбции нефтешлама кварцевым песком на основе остаточного желтого слоя нефтешлама. В случае сорбции нефтешлама прокаленным и модифицированным сорбентами остаточные нефтепродукты незначительны. Приведен сравнительный анализ данных по эффективности разработанного сорбента и имеющихся в настоящее время аналогов на основе шламов и шлаков.

Выводы. *Определены физико-химические свойства сорбента, модифицированного ГКЖ Типром К: насыпная плотность 0.621 г/см^3, зольность 97.1%, суммарный объем пор по воде менее 0.05 см^3/г, истираемость 8.8%, влажность менее 0.5%; сорбционная емкость, в г/г: по н-гексану 0.86, по бензину АИ-92 0.89, по нефрасу 0.93, по керосину 0.99, по нефтешламу 1.18, по мазуту 1.46. Эффективность очистки твердой поверхности от нефтешлама прокаленным сорбентом составила 97%, а модифицированным – 95%. Модифицированный сорбент обладает высокой плавучестью при насыщении нефтепродуктами и способностью их длительного удержания.*

Ключевые слова: *сорбент, золошлаковые отходы, теплоэнергетика, нефтепродукты, гидрофобизирующие кремнийсодержащие жидкости*

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INTRODUCTION

Ash and slag (ash residue) is formed by mixing water with captured fly ash and solid slag waste generated by the combustion of coal in the furnaces of power plants. It is sent for storage in the form of a pulp outside the territory of the thermal power station (TPS) to an ash dump, also called a hydroash dump.

Currently, the quantity of accumulated ash-and-slag waste (ASW) amounts to billions of tons. The area occupied by them is thousands of hectares of land. Seepage into the soil and dusting of ASWs have a negative impact on the environment and the health of people living in urban areas near TPSs, combined heat and power stations (TETS in Russian abbreviation), and state district power stations (GRES in Russian abbreviation) [1, 2]. Recycling of ASWs in Russia is no more than 8% [1].

This waste is cheap, accessible, non-explosive and non-flammable.

The review [2] analyzed research by scientists on the impact of ASWs on the environment. The results of the analysis are summarized in a table which

shows that the storage of ash waste leads to dust from the surface of the ash dump, migration of pollutants along the soil profile and alienation of the territory. The first two factors contribute to the deposition of pollutants on the surface, pollution of atmospheric air, surface, ground and underground waters. Data from the literature regarding the content of isotopes in the ash of thermal power plants enabled a conclusion to be made about the danger to public health and the threat to flora and fauna due to the leaching of radionuclides and heavy metals into the soil and groundwater. The following areas for processing and disposal of ASWs were identified: use in agriculture; extraction of metals; rare and valuable components; firefighting; alumina production; as well as wastewater treatment and isolation of various wastes. The analysis of the chemical composition of the ashes of the *Khabarovskaya TETS-3* power station showed that the waste is classified as a technogenic mineral raw material which accumulates over time.

Many research and review works have been devoted to the areas of recycling of ASWs [3–6]. In [3], the composition of ASWs of the *Kazanskaya TETS-2* power station was studied. It can be concluded that the main promising areas for the utilization of ASWs, in addition to the construction industry, can be the extraction of titanium from them, the synthesis of zeolites from the aluminosilicate components of ash and slag. In order to maximize the level of ASW recycling, it is recommended that the ASW removal system be switched to the dry method [4]. In order to reduce the negative impact of ASWs on the environment and involve them in economic turnover, the issue of regulating this problem is proposed to be returned to the legislative level [6].

ASWs from the combustion of coal from TPS is generally considered to be non-hazardous to the environment. In [7] the chemical composition of the ASWs of the *Khabarovskaya TETS-3* is given. Based on the calculation method and the phytotesting method, the waste is classified as hazard class V.

Our quantitative chemical [8] and toxicological (biotesting) analyzes of the ashes of the *Novocherkasskaya GRES* power station showed that the dry ash-and-slag mixture collected by an electric precipitator, cyclone and bag filter belongs to hazard class IV [9], while the ash-and-slag mixture accumulated at the ash dump according to the hydraulic ash removal scheme, belongs to hazard class V [10].

Every year, the pollution of water bodies with oil products (OPs) increases as a result of the discharge of partially treated or untreated wastewater into them. This in the future may lead to an environmental disaster. Research by scientists confirms the presence of adsorption properties of ASWs. The paper [11] proposes the use of waste water from an energy generating enterprise (Russia) as a sorbent (filter media) to clean the drainage of a storm drainage system. The characteristics of the proposed ash-and-slag sorbent (Russia) are given. A device for drainage of storm and snowmelt runoff, including a filter media, has been developed. It is recommended that purified water be used for household needs (irrigation, construction activities, etc.) in order to save drinking water.

In [12], the authors used fly ash from coal-fired power plants (India) as a sorbent for the treatment of domestic wastewater. Waste water was purified after preliminary settling for 24 h to precipitate coarse mechanical impurities. Wash water samples were placed in flasks with a capacity of 250 mL, a weighed portion of the adsorbent was added and kept in a thermostat with a shaker at a temperature of 25°C for 7 h. No significant adsorption was observed. Next, the effect of ash/wastewater ratio on the removal efficiency of pH, surfactants, and suspended solids was studied. As a result, it was proposed that the sorbent be used for the treatment of household wastewater at a dosage of 40 g of sorbent per 1 L of treated water for 6 h.

In [13], the ash and slag of the *Taichung Power Plant* (Taiwan) was studied from the point of view of its use as a sorbent for the purification of synthetic (model) wastewater. Based on chemical oxygen demand (COD), it was found that model wastewater contains copper in a quantity from 10 to 40 mg/L and potassium hydrophthalate in a quantity from 250 to 1000 mg/L. Wastewater (leachate) from the Taichung City landfill with known contents of ammonium nitrogen, total nitrogen, iron, phosphates, zinc, and manganese was also tested. Sorption conditions were optimized, such as solution pH, temperature, sorbent dosage (sorbent/purified water ratio). When using ash and slag as a sorbent, the purification efficiency for COD was 47.0%, for ammonium nitrogen, 39.4%; for total nitrogen, 31.1%; for zinc, 82.2%; for manganese, 94.3%; for iron, 96.5%; for phosphates, 92.9%.

Indian scientists have conducted studies on the treatment of wastewater with fly ash from phosphates and fluorides [14], suspended solids, COD and biochemical oxygen demand (BOD) [15], heavy metals and organic substances [16].

Wastewater with a volume of 1.0 L was filtered through a layer of sorbent (dynamic sorption) 10 cm thick. The free filtration process took about 1.3 h. As a result, the degree of purification of the wastewater sample in terms of suspended solids was 69.02%, in terms of BOD—71.48% and in terms of COD—66.59% [15].

In [16], fly ash was used as a sorbent, in order to purify wastewater from a number of heavy metals: nickel, zinc, lead, iron, manganese, and aluminum. The purification efficiency of the wastewater sample was more than 80% for all metals except manganese. Its cleaning efficiency was in the range of 70–80%.

In [17], fly ash was used as a sorbent to purify water from 2,4-dimethyl phenol. The sorption properties of the material were studied in model aqueous solutions containing 2,4-dimethylphenol, depending on the temperature of the solutions, the dose of the sorbent, and the initial content of 2,4-dimethylphenol. The authors managed to achieve a degree of extraction of 2,4-dimethylphenol from an aqueous solution at a level of about 90%. In addition, a method was proposed for regenerating the sorbent with a 2% aqueous solution of hydrogen peroxide.

In [18], fly ash was used as a sorbent for the removal of 2-chlorophenol (2-CP) and 2,4-dichlorophenol (2,4-DCP) from aqueous solutions. The effect of pH solution on the removal process was studied (the pH value is lower than the pK_a value of 2-CP and 2,4-DCP). An experimental setup was assembled with a filter filled in layers from the edges with quartz sand and in the middle layer with fly ash. Sorption was mobile in nature of the solution being purified (dynamic sorption). The sorption capacity of fly ash reached its maximum stable value already at a passing time of about 60 min and was about 2.0 g/g and 1.3 g/g for 2,4-DCP and 2-CP, respectively. The mutual influence of 2,4-DCP and 2-CP on the sorption process was studied.

This paper presents the results of studies of an environmentally friendly sorbent based on ASW thermal power engineering, calcined and modified with the Tiprom K organosilicon water-repellent agent. It has a high buoyancy when saturated with OPs and the ability to retain them for a long time. The modified sorbent was studied on model mixtures containing fuel oil, kerosene, AI-92 gasoline, nefras, oil sludge and *n*-hexane and is intended for cleaning wastewater from free-floating and emulsified petroleum products. The sorption of oil sludge on a water surface was carried out under laboratory conditions. A real sample of sea water (the Black Sea) was studied as a water body.

METHODS

The physicochemical properties of the modified sorbent are determined according to Russian regulatory documents: bulk density according to GOST R 51641-2000[1], ash content according to PND F 16.2.2:2.3:3.32-02[2], total pore volume according to GOST 17219-71[3], abrasion strength according to GOST R 51641-2000, mass concentration of OPs according to PND F 14.1:2:4.5-95[4], humidity according to GOST 5180-2015[5]. The sorption capacity of the modified sorbent in relation to OPs was determined experimentally, as described in this article.

To calcinate the samples, a LM-312.11 laboratory electric furnace (*VEB ELEKTRO BAD FRANKENHAU-ZEN*, 1990, German Democratic Republic) was used, maintaining a temperature range from +50 to +1200°C. The study of the OP content in the samples was carried out using a KN-3 concentration meter (*SIBEK-OPRIBOR*, 2018, Russia). To dry reagents and samples and prepare laboratory glassware, a drying cabinet of the LOIP LF-60/350-GG1 series (*Laboratornoe Oborudovanie i Pribory*, 2012, Russia) was used, maintaining a temperature range from +50 to +350°C. To weigh the samples, Adventurer electronic laboratory scales of AR214 modification (*OHAUS Europe*, 2004, Switzerland) were used. Extraction of solutions was carried out on an ES-8000 rotary extraction unit (*Ekokhim*, 2015, Russia), extraction of solid samples was conducted on a universal laboratory shaker of WU-4 type (*PREMED*, 1985, Poland). Abrasion tests of materials were carried out using a KP-131 set of sieves (*Meridian*, 2015, Russia) with a lid and tray. All measuring instruments are included in the Russian State Registry of Measuring Instruments[6] and verified in accordance with the procedure established by the legislation of the Russian Federation. When conducting laboratory

[1] GOST R 51641-2000. State Standard of the Russian Federation. Filtering granular materials. General specifications. Moscow: Gosstandart Rossii; 2000.

[2] PND F 16.2.2:2.3:3.32-02. Measurement procedure of the content of dry and calcined residue in solid and liquid waste of production and consumption, sediments, sludge, activated sludge, bottom sediments by gravimetric method. Moscow: NTF “Khromos”; 2002 (publ. 2017).

[3] GOST 17219-71. State Standard of the USSR. Active carbons. Method for determination of summary pore volume by the moisture capacity test. Moscow: Ordena “Znak Pochyota” Izdatelstvo stantartov; republication October 1987.

[4] PND F 14.1:2:4.5-95. Quantitative chemical analysis of waters. Method of measuring the mass concentration of petroleum products in drinking, surface and wastewater by IR spectrometry. Moscow: FTsAO; 1995 (publ. 2011).

[5] GOST 5180-2015. Interstate Standard. Soils. Methods of laboratory determination of physical characteristics. Moscow: Standartinform; 2015.

[6] https://fgis.gost.ru/fundmetrology/registry. Accessed October 03, 2022.

studies, volumetric glassware was used: cylinders, burettes, volumetric flasks, and pipettes in accordance with GOST 29228-91[7].

RESULTS AND DISCUSSION

The object of the study is ASW taken from the 3rd section of the ash dump of the *Novocherkasskaya GRES* power station (Novocherkassk, Rostov oblast), which is a liquid-solid mass. A sample of ASW was homogenized and placed in a drying oven, where it was kept at 110°C for 30 min to remove free moisture. The dried ASW had a lumpy shape. As a result of calcination in a muffle furnace at 600°C and holding at this temperature for 30 min, a loose crumbly material was obtained, called calcined sorbent, the physicochemical properties of which were determined by us in [19] and amounted to: bulk density was 0.666 g/mL; ash content, 99.5%; total pore volume, 0.506 mL/g; abrasion, 8.5%; humidity, less than 1%. According to the results of granulometric analysis, 95.2% of the mass of the calcined sorbent falls in the fraction from 0.25 to 0.5 mm.

In order to impart buoyancy when saturated with OPs and ability to retain them for a long time on the water surface, the calcined sorbent is modified with an organosilicon water repellent (hydrophobizing silicon-containing liquid, HSL) and is called a modified sorbent.

At the first stage of research, the optimal mass ratio of HSL and prepared calcined sorbent was determined. Five samples were prepared based on Silor/ASW HSL in mass ratios of 1:20, 1:10, 1:5, 1:3, and 1:2. The samples were left open for a day at room temperature until completely dry. Next, the samples were ground in an agate mortar to break up the lumps formed during the process of wetting the ASW by HSL, and the sorption properties of the resulting samples with respect to *n*-hexane were studied.

Distilled water of 1.0 L was placed in an open cylindrical glass container with a capacity of about 3 L. An aliquot of 2.00 mL was placed into the same container using a microdispenser and, in a separate experiment, 4.00 mL of chemically pure (99.9% purity) *n*-hexane (density at 20°C is 0.6548 g/mL), which corresponds to 1308 mg and 2617 mg in terms of pure substance. *n*-Hexane was placed on the surface of the water in the region of the geometric center so that the *n*-hexane spot did not touch the walls of the container. Next, an accurate weighed portion of the prepared sample of the modified sorbent weighing about 1.0 g was selected (the mass value was recorded with an accuracy of 0.0001 g and was used in further calculations) and was scattered in a uniform thin layer on the surface of the *n*-hexane spot. The samples were left in this form for 30 min. After this, the sorbent was collected with ash-free blue ribbon filter paper, the sample with the filter paper was placed in a Petri dish and dried open in air for about a day to evaporate water and residual *n*-hexane. The air-dry sample together with filter paper was placed in a conical flask with a ground stopper with a capacity of 100 mL, 10 mL of carbon tetrachloride was added, the flask was closed with a lid and the sample was shaken vigorously on a universal laboratory shaker for 1 h. The resulting extract was filtered through an ash-free blue-ribbon filter, installed in a glass funnel, in a flask with a ground stopper with a capacity of 50 mL. Extraction of the sample followed by filtration was repeated 2 more times with new portions of carbon tetrachloride, 10 mL each. All extracts were combined in the same flask with a ground stopper with a capacity of 50 mL, into which the first portion of the extract was collected, and the contents of the flask were mixed with rotational movements.

The resulting combined extract was passed through a previously prepared chromatographic column, which was a cut burette with a capacity of 25 mL. A small piece of glass wool was placed in the lower part of the column, previously kept in a 1:1 solution of sulfuric acid for 12 h, washed with distilled water and, after drying, washed with carbon tetrachloride and dried in air. Then 6 g of aluminum oxide, activated by calcination in a muffle furnace at a temperature of 600°C for 4 h, was poured into the column, followed by the addition of distilled water with a volume of 15 mL per 500 g of calcined aluminum oxide, followed by stirring and holding for 24 h in a desiccator. The top layer of aluminum oxide was also fixed in the column with a small piece of glass wool. Then 8 mL of carbon tetrachloride was poured into the column for wetting. After absorbing a portion of carbon tetrachloride, the column tap was opened and the eluate passing through the column was collected into a measuring cylinder with a volume of 10 mL. As soon as the upper limit of carbon tetrachloride passed through the chromatographic column reached the top layer of glass wool, the column tap was closed and the first portion of the sample extract with a volume of about 10 mL was added to the column, after which the column tap was opened and the eluate continued to be collected into a measuring cylinder with a capacity of 10 mL. As soon

[7] GOST 29228-91. State Standard of the USSR. Laboratory glassware. Graduated pipettes. Part 2. Graduated pipettes without a set waiting time. Moscow: Standartinform; 1991.

as the quantity of eluate in the cylinder reached 8 mL, the column tap was closed and the collected eluate was discarded, then the column tap was opened and the eluate continued to be collected in a conical flask with a ground stopper with a capacity of 50 mL. In this way, the entire volume of the sample extract was passed through the sorption column and as soon as the top layer of the extract touched the top layer of glass wool, the column was closed and 5 mL of carbon tetrachloride was added, after which the stopcock was opened and the eluate continued to be collected in a conical flask until the liquid in the column sank to the level of the top layer of glass wool. The resulting eluate with a volume of 30 mL was thoroughly mixed in a flask by shaking, the eluate was successively diluted 1000 times, the diluted eluate was poured into a measuring cuvette, and the OP content was measured using a KN-3 concentrator previously prepared in accordance with the operational documentation. The OP content in samples X, g/g, was calculated using the expression

$$X = \frac{C_m \cdot B \cdot K}{M \cdot 1000} \quad (1)$$

where C_m is the measured quantity of OP in the extract, mg/L; B is the eluate volume, L (equal to 0.03 L); K is the eluate dilution factor (equal 1000); M is the sample weight, g.

The obtained values of OP content in the samples are expressed in g/g and are the values of the sorption capacity of the modified sorbent samples. The results are shown in Tables 1 and 2 and in Fig. 1.

Based on the analysis of the data obtained, it was concluded that the sorption capacity of the samples does not depend on the amount of *n*-hexane excess. The highest value of the sorption capacity of the studied samples was taken as the result. The optimal weight ratio of HSL to ASW was 1:5, while the sorption capacity with respect to *n*-hexane reached a maximum of 0.86 g/g.

At the second stage of the research, the optimal conditions for drying the samples were determined. The sample masses were subjected to forced drying in an oven at temperatures of 110–200°C to constant mass. At temperatures above 180°C, non-uniform drying of the sample, adhesion and enlargement of grains were observed, which in turn led to a decrease in porosity and a deterioration in sorption properties. At a temperature of 200°C, sintering of the material was observed. The most optimal method is to dry samples at 160°C. For samples weighing 1 g, the drying process took no more than 10 min. Optimization of modification temperature is given in Table 3.

At the third stage of research, the optimal HSL modifier was selected based on the price/sorption properties ratio. Silor, HSL-11BSP, HSL 136-157M, PROFILUX, Tiprom K, and Tiprom U modifiers were considered as HSL. The sorbents obtained on the basis of HSL 136-157M and Tiprom K have the highest oil capacity, but Tiprom K has the lowest price, due to than chosen as the optimal modifier. Figure 2 shows the values of sorption capacity (oil capacity), in g/g, in relation to *n*-hexane, indicating the price of HSL in RUR/kg. The price is shown in figures around HSL in 2021 prices.

Tiprom K HSL was accepted as optimal; its price was 420 RUR/kg, and the sorption capacity of the resulting modified sorbent is 0.86 g/g.

At the fourth stage, the physicochemical properties of the Tiprom K modified HSL sorbent

Table 1. Sorption capacity in relation to *n*-hexane of hydrophobizing silicon-containing liquid (HSL) samples of the Silor brand—calcined sorbent according to the measured content of OPs in the samples when interacting with 1308 mg of *n*-hexane

Modifier/ash ratio (by weight)	Sample weight, g	Measured content of OPs in the extract, mg/L	Eluate dilution factor	Sorption capacity in relation to *n*-hexane, g/g
1:20	1.0008	20.7	1000	0.62
1:10	1.0016	23.7	1000	0.71
1:5	0.9999	28.3	1000	0.85
1:3	0.9968	28.2	1000	0.85
1:2	0.9972	28.3	1000	0.85

Table 2. Sorption capacity in relation to *n*-hexane of HSL samples of the Silor brand—calcined sorbent according to the measured content of OPs in the samples when interacting with 2617 mg of *n*-hexane

Modifier/ash ratio (by weight)	Sample weight, g	Measured content of OPs in the extract, mg/L	Eluate dilution factor	Sorption capacity in relation to *n*-hexane, g/g
1:20	0.9972	21.3	1000	0.64
1:10	0.9976	23.9	1000	0.72
1:5	1.0022	28.7	1000	0.86
1:3	0.9981	28.6	1000	0.86
1:2	0.9974	28.6	1000	0.86

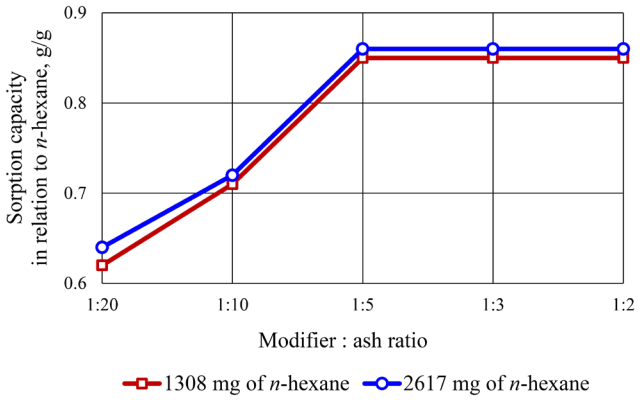

Fig. 1. Dependence of sorption capacity on the modifier/ash ratio (by weight).

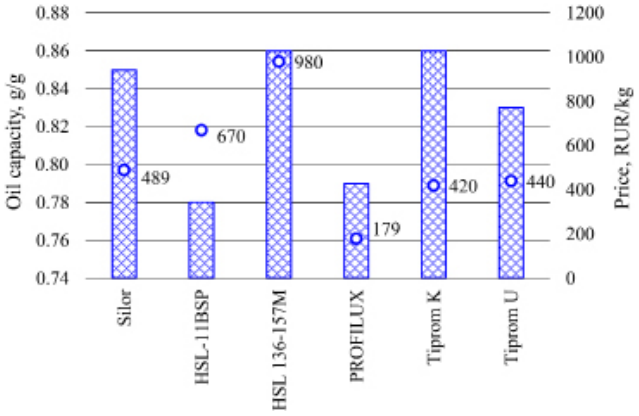

Fig. 2. Sorption capacity (oil capacity) in relation to *n*-hexane and price of HSL.

Table 3. Modification temperature optimization

Temperature °C	Sorption capacity in relation to *n*-hexane, g/g
110	0.72
120	0.75
130	0.78
140	0.81
150	0.85
160	0.86
170	0.84
180	0.78
200	0.60

were studied: bulk density, ash content, total pore volume, abrasion, humidity, and sorption capacity, which are given in Table 4. Methods for determining these indicators were described in detail by us in [19] when studying the properties of a calcined sorbent.

At the fifth stage, the sorption properties of the modified sorbent were studied in relation to various petroleum products, such as: fuel oil, kerosene, AI-92 gasoline, nefras, oil sludge, and *n*-hexane. OP samples were applied to the surface of distilled water at the rate of 2.00 g per 1 L of water, after which 1.00 g of sorbent was evenly scattered in a thin layer over the entire area of the OP spot. The time of interaction of the sorbent with the OPs was recorded. After a certain time, the samples were collected with blue ribbon filter paper, dried in Petri dishes in the open air for 24 h, and in the case of fuel oil and oil sludge, excess OPs were additionally blotted with filter paper and the sorption capacity of the samples was measured. The highest oil capacity was observed in the case of sorption of fuel oil, the lowest—with *n*-hexane. The increase in oil intensity can be explained by an increase in the density and molecular weight of the oil. The sorption time was optimized (Table 5).

For relatively light OPs, the optimal sorption time was at least 30–40 min, for heavy ones—at least 60 min. During the experiment, it was established that half the sorption capacity is filled with OPs

Table 4. Physical and chemical properties modified sorbent

Indicators	Units of measurement	Modified sorbent
Bulk density	g/mL	0.621
Ash content	%	97.1
Total pore volume	mL/g	Less than 0.050*
Abrasion	%	8.8
Humidity	%	Less than 0.5

*The total pore volume is determined by water; the resulting hydrophobized sorbent has hydrophobic properties.

Table 5. Optimization of sorption time

Sorption time, min	Fuel oil	Kerosene	AI-92 gasoline	Nefras	Oil sludge	*n*-Hexane
	Sorption capacity (oil capacity) g/g					
10	0.68	0.58	0.59	0.52	0.61	0.56
20	1.03	0.79	0.72	0.68	0.84	0.73
30	1.20	0.92	0.85	0.81	0.99	0.84
40	1.32	0.97	0.88	0.89	1.09	0.86
60	1.45	0.99	0.89	0.92	1.17	0.86
90	1.46	0.99	0.89	0.93	1.18	0.85

in the first minutes of contact. Conventionally, complete sorption can be considered 30–40 min for relatively light hydrocarbons (*n*-hexane, AI-92 gasoline, kerosene, and nefras), 40–60 min for oil sludge and more than 60 min for fuel oil (Fig. 3).

A laboratory study of the sorption of oil sludge on the water surface was carried out (Fig. 4). A real sample of sea water (the Black Sea) was studied as a water body.

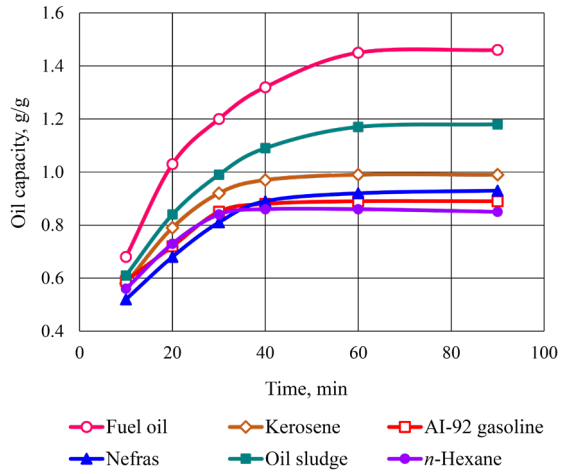

Fig. 3. Dependence of the oil capacity of OPs on the time of sorption.

(a)

(b)

Fig. 4. Sorption of oil sludge on the water surface: (a) applying oil sludge to the water surface with a microdoser; (b) sorption of oil sludge by a modified sorbent.

Oil sludge of 1.00 g was applied to the surface of water with a volume of 1 L using a microdoser. Then 1.00 g of sorbent was applied to the OP spot. After 30 min, the sorbent with oil sludge was removed and the water was analyzed for OP content using the infrared spectrometry.

The sea water sample was transferred to a separating funnel with a capacity of 2 L. 20 mL of a 1:9 sulfuric acid solution was added to the separating funnel with the water sample under study and mixed. Next, 20 mL of carbon tetrachloride was added to the funnel and the OPs were extracted with carbon tetrachloride by intensively mixing the contents of the funnel using a rotary extraction unit for 5 min. After extraction, the funnel with the sample was left alone for 10 min for phase separation. After phase separation, the lower organic phase was poured into a glass with a capacity of 50 mL. 10 mL of carbon tetrachloride was again added to the separating funnel with the sample and the OPs were re-extracted in the manner described above, and the lower organic layer was poured into the same beaker with a capacity of 50 mL that contained the first portion of the extract, thus combining the resulting extracts. Next, 5 g of sodium sulfate, previously dried in an oven at 110°C for 3 h, was added to the glass with the extract, the contents were thoroughly mixed with a glass rod and left for 30 min to completely dry the extract. After drying, the extract was passed through a pre-prepared chromatographic column, as described in the experiment to study the sorption capacity for *n*-hexane of Silor HSL samples.

The resulting eluate with a volume of 30 mL (0.03 L) was thoroughly mixed in a flask by shaking, the eluate was diluted 10 times, the diluted extract was poured into a measuring cuvette, and the OP content was measured using a KN-3 concentrator. The OP content in sea water sample X, mg/L, was calculated using the expression

$$X = \frac{C_m \cdot B \cdot K}{V} \quad (2)$$

where C_m is the measured quantity of OP in the extract, mg/L; B is the eluate volume, L (equal to 0.03 L); K is the eluate dilution factor; V is the volume of sample taken for analysis, L (equal to 1 L).

Table 6 shows the measured OP contents in extracts recalculated to the OP content in sea water contaminated with oil sludge after interaction with the sorbent, as well as the data necessary to establish the efficiency of sorption of surface OPs.

Thus, the content of residual OPs was 12.0 mg/L, which corresponds to a sorption efficiency of surface OPs of more than 95%. During the experiment, it was found that the sorption process does not depend on the matrix (salinity) of water.

At the final stage, a comparative experiment was carried out on oil spill response using quartz sand, calcined and modified sorbents (Fig. 5).

A thin film of 1.00 g of oil sludge was applied to the surface of the watch glass. Next, 1.00 g of calcined sorbent, modified sorbent, and quartz sand were applied. After 30 min, the mixture was collected with a spatula. When visually assessing the color intensity of the residual oil sludge stain, it was concluded that there is a significant content of OPs in the case of sorption of oil sludge by quartz sand; a residual yellow layer of oil sludge is visible. In the case of sorption of oil sludge by calcined and modified sorbents, residual oil residues are insignificant. Instrumental analysis of samples of spent modified and calcined sorbents showed the presence of OPs in 1.00 g of sorbent, equal to 0.97 g and 0.95 g, respectively. Thus, the efficiency of cleaning solid surfaces from oil sludge with a calcined sorbent was 97%, and with a modified sorbent—95%.

A comparative analysis of data on the effectiveness of the developed sorbent and currently available analogues based on sludge and slag is given in Table 7. Analysis of the sorption capacity for AI-92 gasoline shows that modifying sludge and slag with various HSLs can increase their sorption properties. The sorption capacity of such sorbents fluctuates at the level of 1 g/g.

The methods presented in [20], [21], [22], and [23] for studying the sorption capacity of sorbents are based on measuring the mass of OPs adsorbed by the sorbent provided they are kept in the thickness of the OPs under complete immersion, which shows the value of the total possible sorption capacity. This article experimentally studied the efficiency of sorption of the working volume of the sorbent during elimination of oil spills of various petroleum products on water and solid surfaces. The methodology for studying sorption capacity contains a stage of drying a sorbent sample with adsorbed OPs in the open air for about a day, in order to remove water and OPs not adsorbed by the sorbent. This demonstrates the ability of the sorbent to reta in adsorbed OPs for a long time.

Based on the research results, sorbent modified with Tiprom K HSL based on the ASWs of the *Novocherkasskaya GRES* power station is recommended for use in eliminating oil spills of various petroleum products on the surfaces of any type of water, including sea water.

Table 6. Efficiency of sorption of surface OPs in a sea water sample based on measured and calculated residual oil product contents

Mass of OPs added to the 1 L water, mg	Measured content of OPs in the extract, mg/L	Eluate dilution factor	Concentration of OPs after sorption, mg/L	Sorption efficiency, %
1000	40.0	10	12.0	98.8

Fig. 5. Oil sludge sorption on a hard surface: from left to right: oil product sample, quartz sand, calcined sorbent, and modified sorbent.

Table 7. Characteristics of sorbents based on sludge and slag (production waste)

Sorbent	Sorption capacity for AI-92 gasoline, g/g	Sorption capacity research method	Source
Activated carbon AG-3	0.48	–	[20]
Granular sorbent based on chemical water treatment sludge at *Kazanskaya TETS-1*:		Gravity method by immersing a sorbent sample in a sample of pure OPs	
impregnated with a 3% aqueous solution of HSL-11N	0.62		[20]
impregnated with a 5% aqueous solution of HSL-94N	0.68		
impregnated with a 5% aqueous solution of HSL-94N + Silor	0.648		[21]
Sorbent based on soda production sludge of *Bereznikovsky Sodovy Zavod* (formed at the boundary of the water surface, finely dispersed, pasty, white):		Ratio of the absorbed OP mass to the known mass of dry sorbent	
original	0.95		[22]
processed with HSL-11P	1.13		
treated with *Akvasila* solution	1.2		[23]
Sorbent based on ash and slag from *Novocherkasskaya GRES* power station (modified HSL brand Tiprom K)	0.89	Measurement of the mass content of OPs in the sorbent using infrared spectrometric methods	Current article

CONCLUSIONS

An environmentally friendly modified sorbent was developed based on thermal power ASWs accumulated in ash dumps. This has high buoyancy when saturated with OPs and the ability to retain them for a long time.

The physicochemical properties of sorbent modified with Tiprom K HSL were determined: bulk density was 0.621 g/mL; ash content was 97.1%; total pore volume in water was less than 0.05 mL/g, abrasion was 8.8%, humidity was less than 0.5%, sorption capacity, in g/g: for *n*-hexane, 0.86; for AI-92 gasoline, 0.89; for nefras, 0.93; for kerosene, 0.99; for oil sludge, 1.18; for fuel oil, 1.46.

A study of oil sludge sorption on a water surface showed an efficiency of more than 95%. The efficiency of cleaning solid surfaces from oil sludge with the calcined sorbent was 97%, and with the modified sorbent—95%.

The resulting modified sorbent is recommended for eliminating spills of OPs and oil-containing waste at production sites and soils. The use of a modified sorbent will make it possible to effectively purify wastewater before discharging it into a reservoir to acceptable quality standards with minimal economic costs.

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Authors' contribution

All authors equally contributed to the research work.

The authors declare no conflicts of interest.

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