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New sorbents for removal of petroleum-derived pollution on the basis of expanded perlite

Elżbieta Vogt[1], Otmar Vogt[2]*, Anna Papież[1]

[1] AGH University of Krakow, Faculty Energy and Fuels, al. Mickiewicza 30, 30-059 Cracow, Poland
[2] Cracow University of Technology, Faculty of Chemical Engineering and Technology, Warszawska 24, 31-155 Cracow, Poland

*** Corresponding author, e-mail:**
otmar.vogt@pk.edu.pl

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Abstract

New hydrophobic sorbents were developed to remove oil-derived pollutants. The process of hydrophobization was conducted by the application of stearic acid and the commercially available silicone agent Sarsil H. Three fractions of expanded perlite were studied, including the P100F fraction, which is a waste product during the processing of perlite. By determining the beneficial influence of the modifiers upon the selected sorption properties of various fractions of perlite one stated that there is a possibility to apply the modified materials, as sorbents for the removal of petroleum-derived pollution. The process of hydrophobization significantly enhanced the hydrophobic properties of all modified materials. All samples after modification were characterised by lesser absorbability and significantly greater capability of the adsorption of diesel oil from the soil and water surface in comparison with raw samples. One extended the range of the products to remove oil-derived pollutants not only in the context of various means of modification methods and modifying substances but one also demonstrated the possibility of the application of fine-grained fraction of P100F perlite as a sorbent for the removal of petroleum-derived pollutions.

Keywords
sorbents, perlite, hydrophobic, oil-derived pollutants

1. INTRODUCTION

In the current conditions of the extent of the use of oil-derived products they constitute one of the main sources of the pollution of the environment (Eurostat, 2025; Mahmoud et al., 2021), causing extensive contamination of soil, water and air. Oil spills and derived substances float at the surface of the water for a long period of time, blocking oxygen and light, whereas heavy petroleum-derived substances undergo sedimentation and they are deposited at the bottom. However, one should not forget about less spectacular, minor but common cases of spillage of these substances to the environment, caused by the use of machines, transport, fuel distribution stations and other elements of fuel-based infrastructure (Bhattacharjee and Dutta, 2022). A major problem is also associated with (military) training areas, where, due to the degradation of depots and transmission pipelines, harmful substances entered the soil. Compounds such as, e.g. toluene, benzene, phenol, xylene, polycyclic aromatic hydrocarbons (PAHs), considerable amounts of which occur in petroleum-derived substances, manifest strong carcinogenic, mutagenic and toxic properties (Gray et al., 2013; Shukla et al., 2022). Against the background of such conditions, the developing of new methods and new materials for removal of petroleum pollutants is a priority action for the benefit of the natural environment.

There are many methods of the removal of petroleum-derived pollution. They may be divided into mechanical, chemical and biological methods (Bala et al., 2022; Chen et al., 2016; Deschamps et al., 2003; Dong et al., 2016; Haque et al., 2022; Nnaji et al., 2016; Periasamy et al., 2017). Mechanical methods include the following: separators, mechanical devices, natural and synthetic sorbents (Bhardwaj and Bhaskarwar, 2018; Bigui et al., 2019; Han et al., 2021). In the pursuit of more and more efficient methods, one perceives particular potential for these types of processes in adsorption methods. The use of sorbents for the removal of oily substances allows the transition of pollutants from the liquid phase to the semi-solid, which makes it possible to remove them effectively from the environment (Abdel-Aty et al., 2020; Adebajo et al., 2003; Bai et al., 2020; Galblaub et al., 2016; Gupta and Tai, 2016; Karki et al., 2019; Kukkar et al., 2020; Li et al., 2019; Vogt and Płachta, 2017; Yao et al., 2019; Zamparas et al., 2020).

Mineral sorbents, in the form of granules, work well even in hard to reach places, both when removing pollutants from the water surface and from the soil. Sorption sleeves are an indispensable aid in the event of limiting the leak zone and oil adsorption as close as possible to the leak source. Promising potential is represented by work associated with the modification of the properties of natural sorbents, which, due to the lack of certain properties cannot be completely usable. Potential raw materials for such modifications may be constituted by waste materials from agriculture or the industry as well as natural materials, which are available in considerable amounts, such as: perlite, ash, bark, sawdust, leaves of trees, peat, straw, hay, reed, bran, chitin (Qi et al., 2013; Szerement et al., 2021; Tauanov et al., 2020). The advantage of these sorbents has to do with their considerable lightness and good adsorption of pollutants and, after they are subjected to the appropriate modification processes, they may be applied to the task of removing oil-derived pollution with the same efficiency as the expensive zeolite or coal materials (Szerement et al., 2021; Tauanov et al., 2020). A slight modification of selected properties (especially water absorbability) may render these substances a sorbent characterised by radically better usage-related parameters than the input raw material, and decreases the production costs (Bai et al., 2020; Bigui et al., 2019; Davoodi et al., 2019; Deschamps et al., 2003; Gupta and Tai, 2016; Han et al., 2021; PERLIPOL; Perlit Polska Sp. z o.o.; Roulia et al., 2003; Teas et al., 2001).

1.1. Perlite – a raw material for the production of sorbents

Raw perlite is transformed volcanic rock, whose chemical composition depends on the place of extraction, although the content of the main oxides varies only slightly. In Poland, the production of expanded perlite is about 40-50 thousand tonnes per year, in companies which use mainly the raw material imported from Hungary and Slovakia (PERLIPOL; Perlit Polska Sp. z o.o.). The process of the production of expanded perlite consists in the rapid heating of appropriately ground perlite ore in a specific kind of a furnace at a temperature of 900–1000 °C (PERLIPOL; Perlit Polska Sp. z o.o.), where the material is expanded from the size of a few dozen μm to that of a few mm (the final size of the grains determines the class of the product (PERLIPOL; Perlit Polska Sp. z o.o.). During production, also small amounts of perlite powder arise, which is difficult to be used due to its considerable fragmentation and small volume density (PERLIPOL). Considerable water absorbability is the basic disadvantage of expanded perlite (80–300%). Raw perlite manifests porosity within the range of ca. 45–55%, and after the process of expanding – even more than 90%. The water absorbability of perlite may be reduced in the process of hydrophobisation (Vogt and Płachta, 2017; Vogt and Topolska, 2023). Some producers of perlite offer a selected grain fraction of expanded perlite in the hydrophobic form, e.g. the Perlipol company (PERLIPOL, 2022), or the Perlit-Polska company. Hydrophobised expanded perlite is used particularly in the building industry, and also in environmental protection in order to remove petroleum-derived pollution (Bastani et al., 2006; EN 14316-1:2004; PERLIPOL; Roulia et al., 2003). However, the line of hydrophobic perlite which is currently used to remove oil-derived pollution does not utilise the full potential of the raw material. There is a possibility to increase the range of use of perlite by utilising various raw materials, modifiers and modification methods. The efficiency of pollutant sorption is also influenced by the size of perlite grains, for the sorption capacity increases along with fragmentation. The introduction of a greater amount of products which represent various costs of production and properties into the market will enhance the availability of the means of protection of this kind. Hydrophobisation increases the adsorption of oil, even in a water-based environment. Such perlite floats on the water for a considerable period of time and it may be conveniently collected by means of pumps (Bastani et al,. 2006; Muratorplus, 2008; PERLIPOL; Perlit Polska Sp. z o.o.; Qi et al., 2013; Roulia et al., 2003; Teas et al., 2001; Vogt and Płachta, 2017; Vogt et al., 2021).

The novelty of the conducted research is the demonstration of the usefulness of the proposed method for obtaining sorbents based on different kinds of perlite fractions, with a smaller grain size than currently produced sorbents for removing oil-derived contaminants.

2. EXPERIMENTAL

2.1. The research material

For the purposes of this research, three fractions of expanded perlite which differ according to granulation were used: expanded perlite: P100F (class 0), P150 (class 1), P180 (class 2) (PERLIPOL; PERLIPOL, 2025). The chemical composition of these materials is available on the website of the producer and it is as follows [% mass.]: SiO_2 – 65–73; Al_2O_3 – 10–18; $K_2O + Na_2O$ – 6–9; CaO – 2–6; Fe – 1–2. The amount of moisture determined by means of the drying oven method at a temperature of 105–110 °C, in the samples of materials, was the following, respectively [% mass.]: P100F – 0.996; P150 – 0.933; P180 – 1.067; Hydroperl – 0.797. Hydrophobised perlite, whose commercial name is "Hydroperl", was used in the work as a comparison-related material. According to the data provided by the producer, the maximum adsorption of oil for Hydroperl is 350 l/m^3 of the sorbent (PERLIPOL, 2022).

2.2. Conditions of the hydrophobisation process

The solutions of stearic acid in methanol and the commercial, silicone agent for the hydrophobisation of mineral of the ground, Sarsil H, were used as modifiers of hydrophobisation (Silikony Polskie). One determined, by means of an experiment, the supplement of stearic acid (97% w/w) to the sample of the material at the level of 8% mass concentration (Vogt and Płachta, 2017). The solution of stearic acid was being continuously added to the solid material, without interrupting the mixing of the components. The mixture was kept in room conditions for the period of 48 h. Previous studies were conducted using longer and shorter contact times of the reagents. The materials obtained after modification in a shorter time were characterized by worse properties. Extending the contact time beyond 48 hours did not improve the properties of the modified materials described in the work. After that time, the solvent was evaporated from the samples by means of a rotary evaporator. Then the materials were dried at a temperature of 105–110 °C. By conducting an experiment one determined that 10% vol. of the Sarsil H agent was a sufficient amount to cover a solid material thoroughly.

The agent was added in portions, mixing it thoroughly with perlite. It was kept in contact with the solid until the solvent was evaporated completely.

2.3. The study of the properties of hydrophobic materials

In a preliminary manner, the hydrophobic properties of the materials were determined on the basis of the results of the test associated with floating on the surface of water and water absorbability. The contact angles were determined with the use of Krüss DSA 100M apparatus (Alghunaim et al., 2016; Buckton and Newton, 1986; Tohry et al., 2020).

2.3.1. Floating on the water test

Small amounts of perlite (ca. 2 cm^3) were put into small beakers filled with water. After an hour, after 24 h and after a week, one observed which part of the material floated on the water. Figure 1 presents examplary photographs related to the test which was conducted.

2.3.2. Contact angles

The contact angles were measured on the surface of the compacted material. The average values of the contact angles for samples are presented in Table 1.

Table 1. Average contact angles.

Sample type		Average contact angles [°]
Raw	P 100F	20.5
	P 150	20.0
	P 180	23.8
Stearic acid	P 100F	66.9
	P 150	82.8
	P 180	75.8
Sarsil H	P 100F	105.3
	P 150	102.8
	P 180	93.1
Reference material	Hydroperl	62.3

2.3.3. Absorbability

The hydrophobic properties of materials were also evaluated by measuring the absorbability, which consists in the determining of the amount of water which was not absorbed by the sample during the flowing through the bad. In the majority of cases, research is conducted in a pipe which is filled by the material, at the end of which there is a sieve (EN 1097-6:2013; Szułczyńska et al., 2020). Another method, which is highly relevant in the industry, has to do with contacting a specific mass of a solid with water, and then in measuring the change of the mass after a specific period of time (CEN/TS 15366:2009; EN 1097-6:2013; Szułczyńska et al., 2020). Research was conducted both in reference to raw samples as well as the modified samples, and the comparative material (Hydroperl). 2 g of each material was contacted with 100 ml of distilled water over the period of one hour. After that time, the samples were moved to the sieves for the period of 15 min. The absorbability [$kg_{water}/kg_{perlite}$] of samples is presented in Table 2.

Table 2. The water absorbability.

	Modifier type		
Material	Raw	Stearic acid	Sarsil H
	Absorbability [$kg_{water}/kg_{perlite}$]		
P100F	5.58	3.10	1.51
P150	8.47	5.02	3.06
P180	5.02	2.41	1.10
Hydroperl		3.30	

2.4. The sorption of petroleum-derived compounds

2.4.1. The sorption of petroleum-derived substances from the water surface

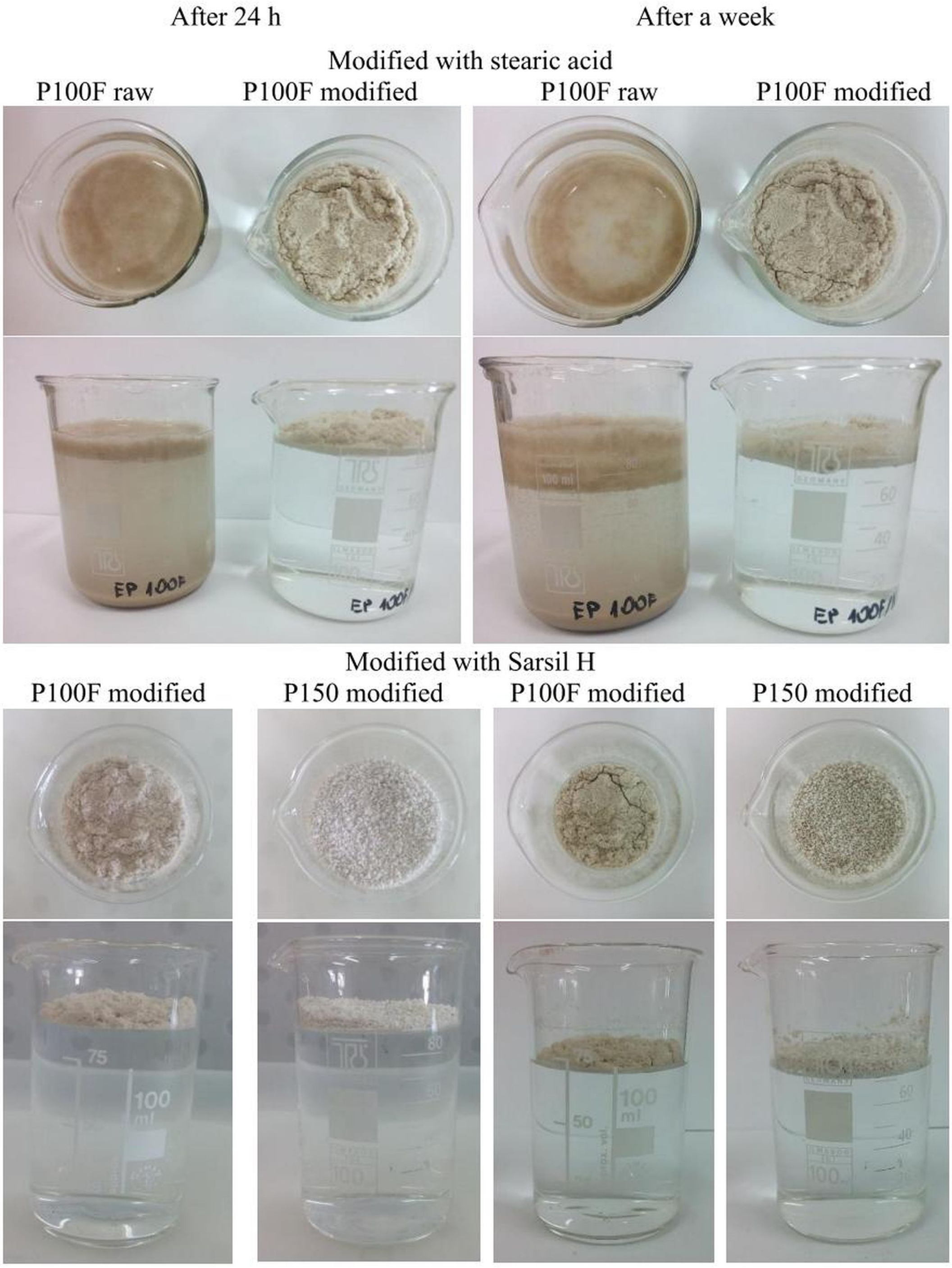
One conducted a study of the sorption of petroleum-derived substances from the surface of water (PERLIPOL, 2022). One placed 25 cm^3 of the perlite sample to a glass container and one added 30 cm^3 of distilled water. Then, one added 15 cm^3 of diesel fuel provided by the BP company (SPL2403 2006) which was adsorbed by shaking for 10 min. After that time, the material was separated by means of a porous plunger. The separated solution was poured to a measuring cylinder, and after the oil separated from the water one read the volume of the non-adsorbed oil. The adsorption of oil was calculated on the basis of a Equation (1) (PERLIPOL, 2022). The results are indicated in Table 3.

$$\mathrm{Ads} = \frac{15 - V_x}{25} \cdot 1000 \left[\frac{\mathrm{l}}{\mathrm{m}^3}\right] \tag{1}$$

where: V_x – the volume of non-adsorbed diesel oil [cm^3].

2.4.2. The sorption of petroleum-derived substances from the soil surface

The sorption of petroleum-derived compounds from the soil surface was conducted on the basis of research conducted by Łuksa et al. (2010). Portions of 200 g of soil were poured to bowls whose diameter was 12.5 cm. The soil was characterised by a humidity of 19.1%. The thickness of the layer of the soil



After 24 h
After a week
Modified with stearic acid
P100F raw
P100F modified
P100F raw
P100F modified
Modified with Sarsil H
P100F modified
P150 modified
P100F modified
P150 modified

Figure 1. The photographs of the floating test of P100F and P150 expanded perlite – modified and raw samples – on the surface of water.

was ca. 2 cm. On the surface of the soil one spread evenly, by means of a spray system, 10 cm^3 of diesel oil and one kept it for 30 minutes. After that time one poured 60 cm^3 of perlite on the soil. Such an amount of the sorbent yielded a 0.5 cm-thick layer on its surface. Subsequently, the samples were divided into three even parts (the division was made according to the size of the field), which were acquired for further research in specific intervals of time: after an hour (1), after 24 h (2) and after six days (3). At the beginning, one collected the sorbent from a given part of the soil, and then the soil was moved to a beaker. By means of hot distilled water one extracted non-adsorbed diesel oil from the soil. One read the volume of the extracted diesel oil which was not adsorbed by the sorbent.

The adsorption of diesel oil was calculated by means of Equation (2).

$$Ads = \frac{(V - V_{s1} - V_x)}{V_s} \cdot 1000 \left[\frac{\mathrm{l}}{\mathrm{m}^3}\right] \tag{2}$$

where: V – the total volume of oil in the soil [cm^3]

V_{s1} – the oil volume adsorbed by the soil (blank) [cm^3]

V_x – the non-adsorbed oil-volume [cm^3]

V_s – the sorbent volume on the soil [cm^3]

The results are presented in Table 3.

Table 3. The adsorption of diesel oil from the water and from the soil: (1) after one hour, (2) after 24 hours, (3) after six days.

	Modifier type										
	Raw		Stearic acid					Sarsil H			
	The adsorption of diesel oil from water [l/m^3]										
P100F	280		580					480			
P150	120		440					420			
P180	120		300					300			
Hydroperl	240		–					–			
	The adsorption of diesel oil from soil [l/m^3]										
	(1)	(2)	(3)	(1)	(2)	(3)	(1)	(2)	(3)	(1)	(2)
P100F	75	125	125	100	125	125	75	100	100	133	183
P150	50	75	100	100	100	100	–	–	–	133	167
P180	50	75	100	75	75	100	50	75	75	150	167
Hydroperl	50	75	100	–	–	–	–	–	–	–	–

2.5. The study of bulk density

One measured the loose bulk density (ρ_b) and tapped bulk density (ρ_{bp}). Considering the conditions of the application of the materials which are obtained, this property may greatly influence the efficiency of their use. A material which is too light will be more conveniently blown away from the area of application. A material which is too heavy will not float on the surface of water.

The study was conducted five times, by means of the Powder Characteristic Tester apparatus, in reference to each samples. One assumed three minutes as the time of the densification of the deposit during the determination of the tapped bulk density. On the basis of the results which were obtained one calculated the Hausner ratio (Carr, 1965; Hausner, 1967; Hosokawa Micron Corporation; Saker et al., 2019) (coefficient of dynamic compressibility, IH) Equation (3), a parameter applied for the characterisation of fine dispersive materials in terms of their flow-related properties.

$$\mathrm{IH} = \frac{\rho_{bp}}{\rho_b} \tag{3}$$

The results are presented in Table 4.

Table 4. Hausner ratio.

	Modifier type		
	Raw	Stearic acid	Sarsil H
	Hausner ratio		
P100F	1.49	1.60	1.80
P150	1.16	1.24	1.21
P180	1.16	1.15	1.17
Hydroperl	1.13		

3. DISCUSSION OF THE RESULTS

The process of hydrophobisation was conducted by the application of two substances: stearic acid dissolved in methanol and the commercially available silicone substance – Sarsil H. One studied three fractions of expanded perlite whose granularity varied, including the P100F fraction, which is a waste product during the processing of perlite. The hydrophobic properties of samples which were obtained in the work were compared with the properties of trade hydrophobic perlite Hydroperl, on the basis of the results obtained from the test associated with floating on the surface of the water and the study of absorbability. The contact angles were measured on the surface of the compacted material. One evaluated the usability of modified perlite in the removal of petroleum-derived compounds from the surface of the water and the soil.

One may observe (see Figure 1 – the testing associated with floating on the surface of water) which materials are subject to moisturisation. A considerable part of the finest-grained, raw fraction of perlite P100F instantly settled at the bottom of the beaker, and the rest of the particles which were floating on the surface were clearly moist. After a week elapsed, one noticed that almost the entire material settled at the bottom. In the case of P100F perlite modified by stearic acid, one observed that even after a week the entire material was floating on the surface of the water and it was not moist. Similarly behaved the greater grains of the raw fraction of expanded perlite: P150 and P180. They were clearly moist. It was observed that perlite P150 and P180 modified by stearic acid even after a week was floating on the surface of the water and it was not moist. Perlite hydrophobised by means of the Sarsil H silicone agent floated on the surface of the water during the entire period of the observation (one week) and one did not observe surface moisturisation of the material.

All samples after modification were characterised by lesser water absorbability in reference to the raw samples and some of them had lesser absorbability than that of the commercial Hydroperl. The tested contact angles of all modified materials were higher than in the case of raw materials.

After modification, all samples of expanded perlite were characterised by much better capacity of the adsorption of diesel oil as compared to raw samples. The greatest level of adsorption was manifested by the sample of P100F perlite (which is a waste product during the processing of perlite), which was subjected to modification with stearic acid, this value was 580 l/m^3. The greatest increase of the adsorption capabilities after modification was manifested by P150 perlite, regardless of the modifier which was applied. The samples of perlite: P100 F and P150, which were subject to modification with a silicone substance manifested slightly lesser values of the adsorption of diesel oil than the samples modified with stearic acid. Perlite which represented the greatest grain size, P180, modified with Sarsil H, manifested the same capability for adsorption as in the case of the perlite modified with stearic acid. All samples after modification manifested greater capability of diesel oil adsorption from the surface of water than the comparative material – Hydroperl. By studying the adsorption of another diesel oil from water on the same perlite fractions but the ones which were modified with stearic acid dissolved in ethanol and petroleum ether (Vogt and Płachta, 2017) one stated that the adsorption capability was also the greatest in reference to P100F perlite and it was 520 l/m^3.

As far as the samples which were modified with stearic acid are concerned, one may observe that already after an hour the adsorption of oil from the surface of the soil increased in comparison with raw samples. The values of adsorption which were acquired after 24 h and after six days were the same as those of the raw samples after the same period of contact. The best adsorption of diesel oil from the soil was represented by samples of perlite which were modified by means of a silicone substance. In the case of these samples the adsorption was not measured after six days because satisfactory results were obtained in a lesser amount of time. Considering that the comparative material, Hydroperl, was characterised by the same adsorption capability as the raw samples P150 and P180, one may state that modification with Sarsil H enhances adsorption properties. The results which were obtained may carry errors resulting from small amounts of oil used in the research, although they do enable us to notice differences between the particular materials during the sorption of oil from the soil.

On the basis of the measurements of the tapped bulk density one may characterise the flow properties of the materials under study on the basis of the values of the Hausner ratio: $1.00 \div 1.10$ free flowing; $1.10 \div 1.25$ relatively unimpaired flowing; $1.25 \div 1.40$ poorly flowing (semicohesive); > 1.40 very poorly flowing/non-flowing (strongly cohesive). The fractions of P150 and P180 perlite after modification, in comparison with raw materials, did not change their flow properties. Similarly as in the case of Hydroperl, they are materials which flow relatively well. This accounts for the fact that the probability of them being blown away from a contaminated area is the same like for raw material. The finest-grained fraction of P100F perlite, both in the raw form and after the process of modification, is a material which is very poorly flowing. The process of hydrophobisation reduces the susceptibility of the perlite to being blown away.

4. CONCLUSIONS

The process of hydrophobisation which was conducted significantly enhanced the hydrophobic properties of all modified materials. During the entire period of observation, samples modified with Sarsil H and stearic acid, floated on the surface of water and they did not become moist, similarly as in the case of Hydroperl.

The study of the absorbability of water enables one to conduct a quantitative evaluation of the hydrophobic properties of the modified materials. All samples after modification were characterised by lesser absorbability, and the fractions of perlite which were modified by Sarsil H are characterised by lesser absorbability than the commercial Hydroperl. This is particularly manifest in the case of P100F and P180, whose absorbability constitutes ca. 30% of the value obtained for Hydroperl. In the case of these two materials, also after modification with stearic acid, one obtained, in the measurements, lesser values of absorbability than in the case of Hydroperl.

The tested contact angles of all modified materials were higher than in the case of raw materials.

The process of hydrophobisation also enhanced the sorption properties in reference to petroleum-derived substances. All samples were characterised by significantly greater capability of the adsorption of diesel oil from the surface of water in comparison with raw samples. It is worthwhile to emphasise that the greatest adsorption was manifested by samples of perlite whose grain was the finest (P100F). Samples modified with Sarsil H were characterised by slightly lesser adsorption of oil from water, in reference to samples modified with stearic acid. However, practically all the samples manifested greater capability of the sorption of diesel oil than the comparative material, Hydroperl.

In the case of the sorption of diesel oil from the soil, the greatest capability of adsorption was characterised by samples modified with Sarsil H.

The study of bulk density of samples after hydrophobisation indicates that the proposed processes of hydrophobisation does not unfavorably influence the increase of the volatility of the materials and they do not increase their weight. Thus, they will not sink to the bottom during the removal of pollution from the surface of water, and they will not be blown away from the area of application, to a greater extent than the perlite sorbents which were used before.

By determining the beneficial influence of the hydrophobic modifiers that were used upon the selected sorption properties of various fractions of expanded perlite one stated that there is a possibility to apply the materials, after modification, as sorbents for the removal of petroleum-derived pollution. One extended the range of the products of this type not only in the context of various means of modification and modifying substances but one also demonstrated the possibility of the application of fine-grained fraction of P100F perlite as a sorbent for the removal of petroleum-derived pollutions.

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