

A magnetic hydrophobic sorbent for removal of oil products from water surface

Věnceslava TOKAROVÁ^a, Stanislava STIBOROVÁ^a, Pavel BĚLECKÝ^a,
Aleš KAŠPÁREK^a, Joanna E. OLSZOWKA^b

^a Unipetrol výzkumně vzdělávací centrum, Revoluční 84, 400 01 Ústí nad Labem, Czech Republic;

^b Jerzy Haber Institute of Catalysis and Surface Chemistry, Polish Academy of Sciences, 30239 Kraków, Poland

E-mail: venceslava.tokarova@unicre.cz, pavel.belecky@unicre.cz,
ales.kasperek@unicre.cz, joanna.olszowka@jh-inst.cas.cz

Abstract

In this article, a development of a hydrophobized sorbent with a silica gel matrix and incorporated magnetite particles is described, porosities and surface areas are measured by nitrogen sorption and desorption isotherms both at pure silica gels and sorbents with incorporated magnetite particles. For a precipitation of silica gel from potassium water glass, four different diluted acids were used (sulphuric, nitric, hydrochloric and acetic). Nitric and sulphuric acids showed the best results, because silica gels from both these acids contained about 1 cm³ of pores per 1 g with a size in the mesopore range. Moreover, the precipitation of silica gel with nitric acid provided the silica gel with surface area above 900 m²/g, which is almost 1¾ times more than in the case of the silica gel prepared from sulphuric acid. Silica gels prepared from all four acids were formed by extrusion with 10, 20 and 30 g of magnetite per 100 g of silica gel dry matter. All silica gel extrudates with magnetite particles were also measured by nitrogen sorption and desorption isotherms to determine their surface area and porosity data. A correlation between magnetite contents and porosities was not obvious. Anyway, for needed magnetic properties to collect the sorbent from water surfaces, 10 g of magnetite per 100 g of silica gel dry matter were sufficient. Silica gel extrudates were hydrophobized by silanization with hexadecyltrimethoxysilane and then tested for a sorption of Diesel fuel spills from water surfaces. The extrudates caught roughly 1.3 g of Diesel fuel per 1 g of the sorbent with a sorption efficiency 90%.

Keywords: hydrophobic magnetic sorbent, silica gel, Diesel fuel

Introduction

Oil spills on water surfaces belong to serious ecological disasters involving immediate and cheap solutions with regard to large volumes of oil products penetrating to waters from damaged tankers, ships and other accidents. Among various methods of ecological disposal of such contaminations, mechanical methods by using special devices take place¹, various barriers such as booms made of fences and other fire resistant tools² can help very effectively, too. In the case of large contaminated areas, burning can be the cheapest method. Anyway, not always such combustion processes are proper or even possible. Chemical treatment of oil spills by various dispersants is utilized as well, but this method is not very cheap³, especially when synthetic polymers are applied⁴. Moreover, these polymers are not very useful for larger and thicker spills. For oil removal, many sorbents of various natures have been utilized for many years. For sorption of fuels and oil products, both organic and inorganic materials are used⁵⁻⁷. Inorganic sorbents are based on natural zeolites, clays and similar materials, silica, alumina etc.⁸. Synthetic polymers based on polypropylene or polystyrene foams are advantageous for their natural hydrophobicity and higher sorption capacity than inorganic sorption materials have⁶. Anyway, their drawback lies in prices, non-biodegradability and incompatibility with environment⁹.

Nowadays, various cheaper natural materials as cellulose, keratin, agricultural waste, and similar materials become topical and promising sorbents for oil and fuel contaminations^{10, 11}. These biopolymeric sorbents interact with oil and fuels by several mechanisms including van der Waals forces, interaction with hydrogen atoms, polar interactions and also chemical bonds. Both adsorption on surfaces of sorbents and absorption into their pores take place¹²⁻¹⁴. Firstly, the contaminant reaches the surface of the sorbent and then it penetrates into its pores, where hydrophobicity and lipophilicity of the sorbent play a very important role^{15, 16}.

Among biopolymers, cellulose with glucose as its main structural element, usually organized into fibrous matrix with hemicellulose and lignin¹⁷, became popular probably for its availability due to its large production in the world. Wastepaper, fibres from Kapok tree, banana tree, cotton and various agricultural waste can be promising available sources of cellulose sorbents¹⁸⁻²³. On the other hand, these materials involve further modification to become oleophilic and hydrophobic²⁴. Anyway, low hydrophobicity of cellulose remains its main drawback for sorption of oil and fuel contaminants. To overcome this problem, porous cellulose aerogels were developed. Their advantage lies in low density and low price. For sorption processes, also keratins with collagen, contained in animal bodies and available as waste from poultry farms and industrial plants for a treatment of animal residues, can be utilized as natural biopolymers²⁵⁻²⁷.

To assume advantages and drawbacks of various sorption materials, comparative tests are needed. When several biomass sorbents (kapok and cattail fibre, rice and coconut husk and bagasse) were tested in comparison with commercial synthetic polyester fibre in bench-scale experiments, polyester provided lower sorption capacities for oil than most biomass sorbents and released more oil than all biomass sorbents²⁸.

Sorption properties of not only biological sorption materials are also influenced by their particle size as a main factor of their external surface. Goat hairs and coconut husk were utilized for capture of oil and fuel spills with a contact time 90 min. Besides particle size, molecular weight (chain length) and activation were investigated, too. Activation and small particle size had a big importance in sorption features. Oil and fuel contaminants were sorbed in the order crude oil > diesel > kerosene > petrol. Activated keratine coming from goat hairs sorbed more contaminants than coir²⁹.

Furthermore, other natural polymers provide also promising capability of oil capture, if they are hydrophobic and porous enough. Very good results were reached with porous rubber gel, based on liquid natural rubber³⁰.

To compare various natural, organic and non-conventional, but cheap inorganic materials for capture of various contaminants, a study with peat, wood chips, cement mixtures, road gritting materials, lime, and limestone crumbling was done. Investigated contaminants included diesel, gasoline, alkali hydroxide, ammonia and acids. Among studied sorbents, every material found its application for some of the wide series of contaminants³¹.

Today's trend turns from synthetic sorption materials (e.g. Corexit and similar dispersants) for oil removal to eco-friendly sorbents based on biomass, various sponges and aerogels etc.³². Among natural, non-toxic, cheap and biodegradable sorbents, a porous dendrimer structured sorbent (HF-D) from orange peels belongs to noticeable³³, despite it is not suggested for oil removal. Another example is a modified chitosan with oleoyl-carboxymethyl groups which bring about amphiphilic features³⁴. Besides bioorganic substances, also hydroxylapatite as an utterly non-toxic and biocompatible inorganic material can be beneficial as a sorbent of toxic organic substances from water (e.g. parabens) for its naturally porous structured agglomerates consisting of nanocrystals. Hydroxylapatite can be modified by γ -Fe₂O₃ for easy magnetic separations³⁵. Hydrophobized aquatic plants (eichhornia crassipes) with magnetic nanoparticles were also prepared as a promising sorbent for removal of oil spills³⁶. Plant waste (e.g. sawdust) were also utilized for a manufacture of hydrophobic carbon foam with a honeycomb structure with applications in oil capture from water³⁷. Anyway, for cleaning environment from oil spills, propoxylated glycerol as a lipophilic gel is efficient and reusable³⁸, too. Altogether, various materials have assorted sorption capacities for oil spills: hollow carbon spheres 10 g/g, lignin modified aerogel 20 – 40 g/g, polylactic acid 14 – 34, and some sophisticated sorbents can catch even about 100 g/g³⁶.

In this article, a preparation method of a magnetic hydrophobic sorbent with silica gel as an active component is described and sorption properties for capture of Diesel fuel are tested. This sorbent is suggested for a capture of oil spills from water surfaces.

Experimental

Note: In this chapter, % always stands for weight percent.

Chemicals

Potassium water glass containing 26.5% of SiO_2 and 24.0% of K_2O made in Vodni sklo, jsc, Czech Republic

Acids for precipitation (all made in Lach-Ner, Ltd., Czech Republic):

sulphuric acid p.a. (96% of H_2SO_4)

hydrochloric acid p.a. (35% of HCl)

nitric acid p.a. (65% of HNO_3)

acetic acid p.a. (99.8% of CH_3COOH)

Magnetite Fe_3O_4 FEPREN B630 by Precheza, jsc, Czech Republic

Preparation of silica gel

Silica gel was prepared by precipitation of potassium water glass with several acids (all made in Lach-Ner, Ltd., Czech Republic): sulphuric acid (96% of H_2SO_4), hydrochloric acid (35% of HCl), nitric acid (65% HNO_3), and pure anhydrous acetic acid. Before use, the acids for precipitation were diluted with water as it is given in the Table 1.

Table 1: Dilution of acids for potassium water glass precipitation to silica gel

Silica gel	Precipitating acid, its final concentration	Weights of acid and water to mix
2V	24% H_2SO_4	25 g of 96% H_2SO_4 + 75 g of water
3V	23.33% HCl	200 g of 35% HCl + 100 g of water
4V	21.67% HNO_3	100 g of 65% HNO_3 + 200 g of water
5V	50% CH_3COOH	501 g of 99.8% CH_3COOH + 499 g of water

Precipitation of silica gel

Potassium water glass (200 g) was diluted with distilled water (1000 g), a pH electrode was immersed and a solution of one of the precipitating acid, prepared according to Table 1, was added dropwise down to pH 5 while stirring. Arisen silica gel was separated on a filter, rinsed with water to neutral pH and let on the filter overnight. Then the cake was saved in a waterproof bag and a content of dry matter (given in the Table 2) was determined in a small sample by drying at 105 °C overnight and then 2 hours at 135 °C. In this dry sample, physical sorption and desorption isotherms of nitrogen were measured to determine surface area, a total volume of pores and a volume of mesopores. In this way, four types of silica gel were prepared from potassium water glass precipitated by four different acids. Surface areas of silica gels were calculated by BET method, total volumes of pores and volumes of mesopores by NLDTF analysis. It is appropriate to note, that the content of dry matter in wet silica gel, determined by drying in two steps as above described, was also assumed equal to the content of SiO_2 in the wet silica gel, as other impurities were present only in negligible trace amounts after thorough washing.

Preparation of silica gel extrudates with magnetite

From silica gels precipitated by various acids, extrudates with various magnetite doses (10, 20, and 30 g per 100 g of silica gel dry matter) were prepared (see Table 2). Silica gel wet cake amounts containing 100 g of dry matter (dry matter contents are given in the Table 2) were blended in a liquidiser with magnetite Fe_3O_4 (made in the Czech Republic, Precheza company, under a trade mark FEPREN B630) in amounts given in the Table 2. Then this mixture was dried at 60 °C in a laboratory dryer to evaporate 65 – 70 g to make the paste thicker. After that the paste was cooled, 2 g of 25% tetramethylammonium hydroxide solution (TMAOH) were added and after blending this paste was pushed in a snail extruder through a matrix with circular slots of 2.5 mm diameter. Arisen extrudates were dried overnight at 90 °C and then at 135 °C for 2 hours. After drying the diameter of extrudates decreased to 2.2 mm, their average length was in the range 0.6-1.2 cm. All these extrudates were collected by a magnetic bar to test their magnetic properties.

Table 2: Doses of magnetite to silica gels with various dry matter contents

Silica gel	% of dry matter	$\text{Fe}_3\text{O}_4/\text{silica}$	added Fe_3O_4
2V	9.5	0.10	0.95 g
		0.20	1.90 g
		0.30	2.85 g
3V	8.8	0.10	0.88 g
		0.20	1.76 g
		0.30	2.64 g
4V	8.8	0.10	0.88 g
		0.20	1.76 g
		0.30	2.64 g
5V	11.2	0.10	1.12 g
		0.20	2.24 g
		0.30	3.36 g

Hydrophobization of magnetic silica gel extrudates by silanization

Silica gel magnetic extrudates were silanized in a round bottom flask under reflux. Inside the flask, a stainless steel perforated basket with extrudates was stirred in a solution of hexadecyltrimethoxysilane (1.0 g) in methanol (100 mL) by a magnetic stirrer (500 rpm). The flask was immersed in oil bath and heated. After reaching 72 °C in oil bath and 65 °C in the flask, silanization ran 4 hours. After that silanized extrudates were let dry in the air and saved in a plastic bag.

Sorption tests of Diesel fuel spills

Silanized magnetic sorbent containing 10 g of Fe_3O_4 per 100 g of silica (2.0 g) was dried at 60 °C for 2 hours to evaporate residual methanol from its preparation. Meantime, beakers of 50 mL volume with 25.0 g of distilled water and Diesel fuel spills on water surface were prepared. In three pairs of experiments, 2, 3, and 4 g of Diesel fuel were added to create spills. Then the whole dose of dry sorbent (2 g) was thrown on the surface of liquid inside every beaker, the beaker was covered with parafilm to prevent its content from evaporation and let for 24 hours (sorption time) at laboratory temperature. For the whole time the sorbent was on the surface and no particle dropped down, so silanization was fully successful. After the sorption time all magnetic sorbent was collected by a magnetic rod and immersed into a vial with 10 mL of acetone, the vial was closed and thoroughly agitated. Then the liquid was poured away through a filtration funnel into a 100 mL volumetric flask, new acetone (10 mL) was added to the sorbent remaining still in the vial, after capping the vial was agitated again and the liquid poured into the same 100 mL flask. The whole process was repeated once more to extract all captured Diesel fuel in the sorbent. Finally, all the sorbent was poured on the funnel, washed with acetone into the 100 mL flask, the flask was filled with acetone and agitated. Diesel fuel in acetone solution in the flask

was analysed by gas chromatography to determine sorption capacity and efficiency of the magnetic silica gel sorbent. Besides that, a full sorption capacity of the sorbent was determined. The sorbent of a precise weight was immersed in Diesel fuel overnight, then it was separated by magnetic rod, let free of redundant fuel on a frit for 15 min without vacuum and weighed again.

Besides that, a full sorption capacity was determined. Silanized magnetic sorbent containing 10 g of Fe_3O_4 per 100 g of silica (2.0 g) was dried at 60 °C for 2 hours to evaporate residual methanol from its preparation. Then the dry sorbent was transferred into a beaker with 10 g of Diesel fuel, covered with parafilm and let overnight at laboratory temperature. After that, the sorbent was collected by a magnetic rod and transferred into a vial with 10 mL of acetone. After short shaking, all the content of the vial was poured on a frit and the liquid was taken into a 100 mL volumetric flask. The sorbent on a frit was washed with acetone and finally the flask was filled with acetone.

Results and Discussion

Results of surface areas and porosity data for all pure silica gels are in the Table 3. Both total pore volume and volume of mesopores reach the highest values for silica gels precipitated from sulphuric (2V) and nitric acid (4V). Anyway, the silica gel from nitric acid has significantly higher surface area and therefore is the best.

Table 3: Surface areas, total pore volumes and volumes of mesopores in silica gels

Precipitating acid, its concentration	Silica gel	Surface area, m^2/g	Total pore volume, cm^3/g	Volume of mesopores, cm^3/g
24% H_2SO_4	2V	539.9	1.117	1.069
23.33% HCl	3V	476.1	0.675	0.674
21.67% HNO_3	4V	935.7	1.015	0.995
50% CH_3COOH	5V	755.9	0.875	0.680

In the Figures 1 – 3 (bellow) dependences of surface areas, total volumes of pores, and volumes of mesopores of all silica gels on magnetite concentrations are given. Incorporation of magnetite into silica gel causes a significant decrease in surface area to very similar values. All the differences in surface area among individual silica gels diminish even with 10 g of Fe_3O_4 per 100 g of SiO_2 and remain very similar also with 20 and 30 g of Fe_3O_4 per 100 g of SiO_2 . Porosity data provide slightly bigger differences. In the case of two silica gels (4V from nitric acid and 5V from acetic acid), total volumes of pores and also volumes of mesopores are higher with 20 g of magnetite per 100 g of silica than with a half of magnetite (in relative values about 15% higher) and rather close to corresponding values of pure silica gels.

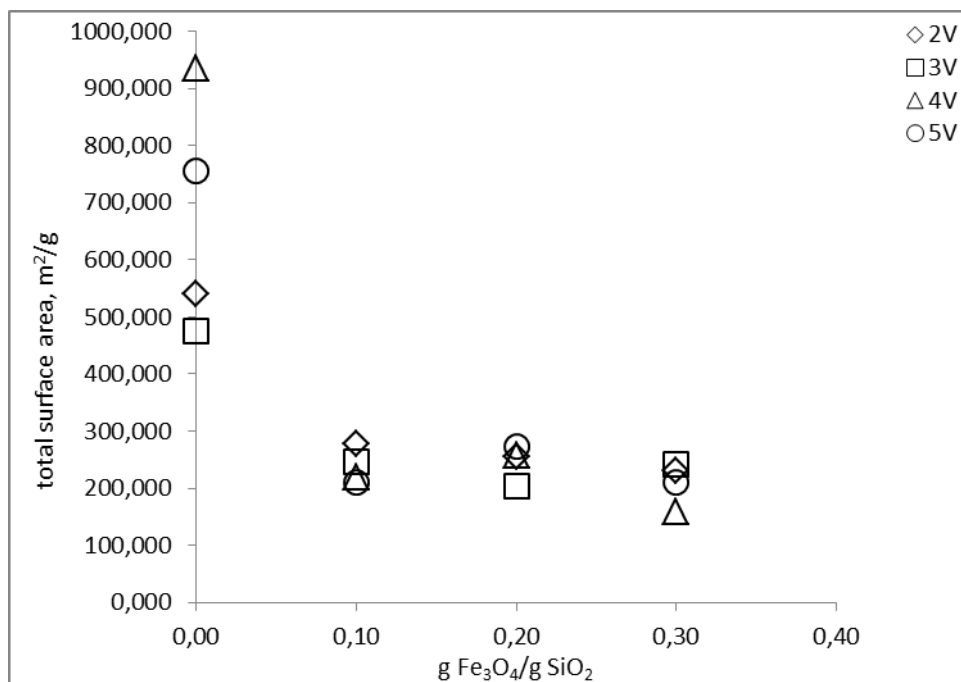


Figure 1: Dependence of surface area of all silica gels on magnetite concentration

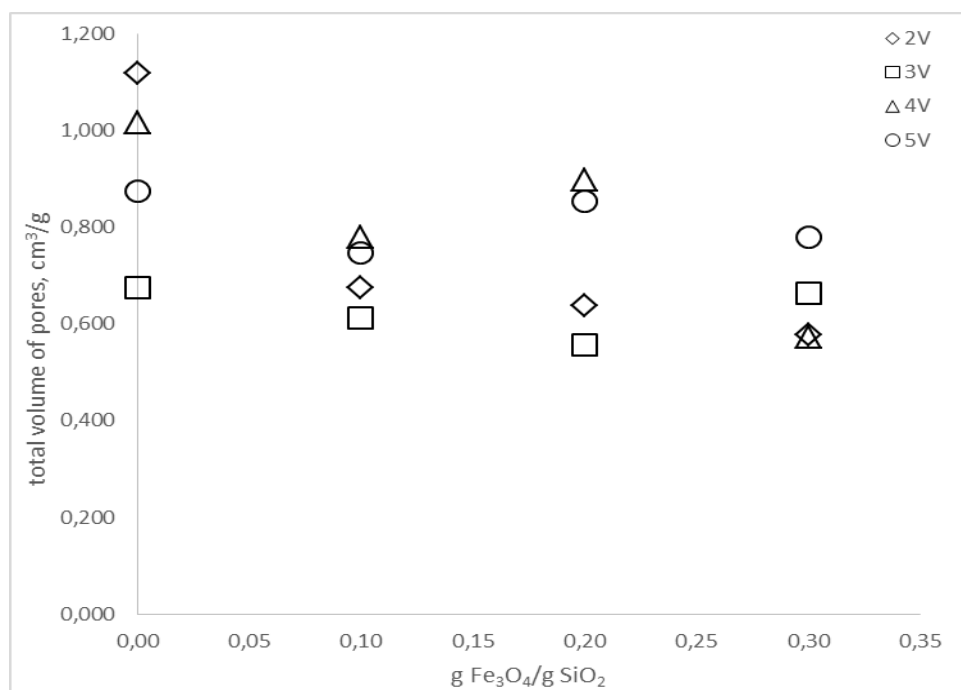


Figure 2: Dependence of total volume of pores of all silica gels on magnetite concentration

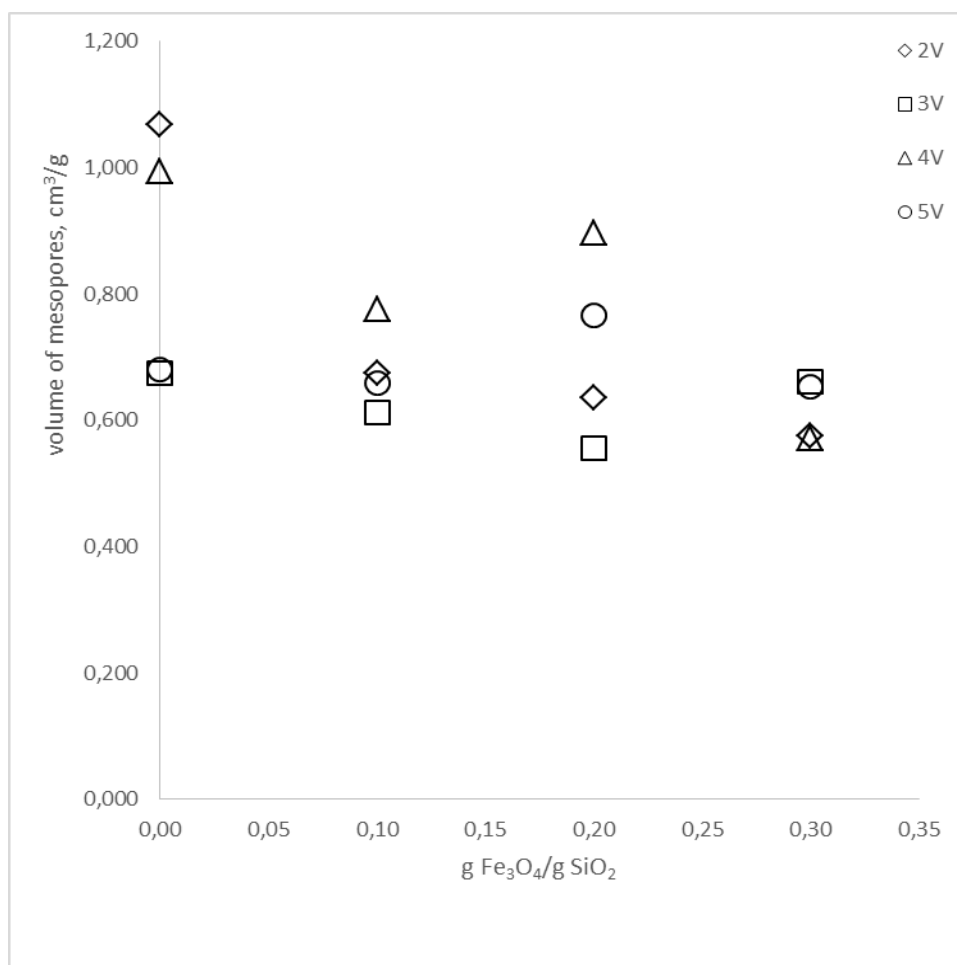


Figure 3: Dependence of volume of mesopores of all silica gels on magnetite concentration

With respect to all results, extrudates prepared from silica gel precipitated by nitric acid with 10% of magnetite were chosen for silanization and capture of Diesel fuel spills. The results given in the Table 4 were achieved with this sorbent, containing silica gel precipitated by nitric acid, extruded with 10% of magnetite and silanized.

Capacity and efficiency of silanized magnetic silica gel for sorption of Diesel fuel spills from water surface

A full sorption capacity of the sorbent for Diesel fuel was 1.435 g/g of the sorbent. In the Table 4, results of sorption experiments are given. Each of three pairs of sorption tests, with 2, 3 and 4 g of the fuel and 2 g of the sorbent, was done with a different saturation of the sorbent with respect to its capacity. From these pairs, average values and relative deviations of individual values from them (%) were calculated. Average values, given under each pair of tests, show, that when the amount of the fuel corresponds to 70% of the sorbent capacity, about 97% of all the fuel is caught. If the quantity of the fuel exceeds 100% of the sorbent capacity only a bit (104%), still nearly 90% is sorbed. Only when the fuel amount exceeds the sorbent capacity significantly, (about 140%), less than 60% of the fuel spill from water surface can be removed. To conclude, if 90% of Diesel fuel spill shall be sorbed, at least 1 g of the sorbent per 1.5 g of the fuel is needed. This dependence is illustrated in the Figure 4 and the experimental points are interpolated by a polynomic curve of the 2nd degree.

Table 4: Weights of sorbents and added Diesel fuel, sorbed amounts per 1 g of the sorbent and sorption efficiency

m_{sorbent} , g	m_{fuel} , g	% of capacity	m_{sorbed} /g of sorbent	% of sorption	rel. deviation, %
2.013	2.025	70.1	0.974	96.8	0.16
2.014	2.020	69.9	0.968	96.5	- 0.16
average % of capacity		70.0	average % of sorption	96.65	
2.011	3.006	104.2	1.353	90.5	1.44
2.056	3.014	102.2	1.289	87.9	- 1.44
average % of capacity		103.2	average % of sorption	89.2	
2.002	4.003	139.3	2.270	56.7	1.11
2.028	4.040	138.8	2.342	58.0	- 1.11
average % of capacity		139.1	average % of sorption	57.3	

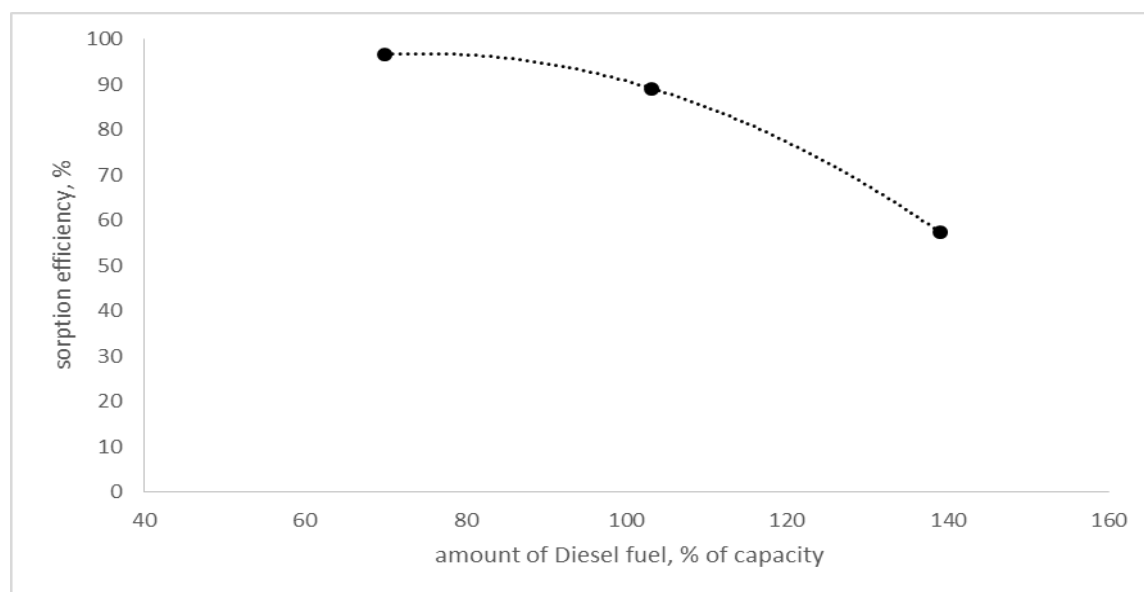


Figure 4: Dependence of Diesel fuel sorption efficiency by magnetic hydrophobic silica gel sorbent on amounts of Diesel fuel, expressed as % of the sorbent capacity

Conclusions

A new hydrophobic magnetic sorbent, for capture of oil or fuel spills from water surfaces, was developed. Its sorption active component is a highly porous silica gel, incorporation of magnetite provides magnetic properties and silanization enables floating on a water surface without immersing even a part of the sorbent. The sorbent was tested in Diesel fuel spill capture from water surface with efficiency in fuel sorption about 90%. Nevertheless, if roughly 90% of Diesel fuel spill shall be sorbed, at least 1 g of the sorbent per 1.5 g of the fuel is needed. Such a sorption capacity is not very high, especially in comparison with published materials mentioned in the penultimate paragraph of Introduction. On the other hand, this sorbent is made from non-toxic materials, environmentally friendly, and can be made rather simply from cheap raw materials, so its drawback can be overcome.

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Magnetický hydrofóbní sorbent pro odstraňování ropných produktů z vodní hladiny

Věnceslava TOKAROVÁ^a, Stanislava STIBOROVÁ^a, Pavel BĚLECKÝ^a, Aleš KAŠPÁREK^a,
Joanna E. OLSZOWKA^b

^a Unipetrol výzkumně vzdělávací centrum, Revoluční 84, 400 01 Ústí nad Labem;

^b Jerzy Haber Institute of Catalysis and Surface Chemistry, Polish Academy of Sciences, 30239 Kraków, Poland

e-mail: venceslava.tokarova@unicre.cz, pavel.belecky@unicre.cz, ales.kasperek@unicre.cz,
joanna.olszowka@jh-inst.cas.cz

Souhrn

Je popsán vývoj hydrofobizovaného sorbentu se silikagelovou maticí a zabudovaným magnetitem, byly proměřeny porozimetrické údaje i měrný povrch metodou izoterm fyzikální sorpce a desorpce dusíku, a to jak u připravených silikagelů, tak i s přidavkem různých množství magnetitu. Pro srážení silikagelu z draselného vodního skla byly použity čtyři různé naředěné kyseliny (sírová, dusičná, chlorovodíková a octová), z nichž se nejlépe osvědčily kyselina dusičná a sírová, neboť silikagel z nich připravený v obou případech obsahoval zhruba 1 ml pórů/g téměř výhradně o velikosti mezopórů. Srážení kyselinou dusičnou nadto poskytlo silikagel s měrným povrchem nad 900 m²/g, tedy téměř 1³/₄ násobek povrchu silikagelu připraveného z kyseliny sírové.

Silikagely připravené ze všech čtyř kyselin byly tvarovány extrudací s přidavkem 10, 20 a 30 g magnetitu na 100 g sušiny silikagelu, u vzniklých extrudátů s magnetitem byly rovněž proměřeny porozimetrické údaje a měrný povrch. Závislost na množství přidaného magnetitu nebyla jasně patrná, nicméně pro dosažení potřebných magnetických vlastností pro sběr sorbentu z vodní hladiny postačoval přídavek 10 g magnetitu na 100 g sušiny silikagelu. Silikagelové extrudáty s magnetitem byly hydrofobizovány silanizací hexadecyltrimethoxysilanem a poté vyzkoušeny k sorpci motorové nafty pro Dieselovy motory z vodní hladiny. Sorbent zachytil zhruba 1,3 g nafty na 1 g sorbentu s účinností sorpce 90 %.

Klíčová slova: hydrofóbní magnetický sorbent, silikagel, nafta do Dieselových motorů