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MODIFICATION OF NATURAL OIL SORBENT FOR IMPROVEMENT OF HYDROPHOBICITY

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Highlights

- Natural organic sorbent (moss) modification methods to increase its hydrophobic–oleophilic characteristics were experimentally studied.
- The highest diesel sorption capacity was equal to 8.99 g · g^{-1} when the moss is modified with 5% alkaline solution (NaOH), water sorption capacity was also high – 13.53 g · g^{-1}.
- The lowest water sorption capacity was equal to 2.49 g · g^{-1} when moss is coated with oil, diesel sorption capacity was also low – 1.09 g · g^{-1}.
- The selection of modification method should depend on the application of sorbent – from water or a solid surface.

Abstract. A sorbent made of natural materials (moss) was selected for the sorption of diesel from the surface of the water. The sorbent was modified to increase its hydrophobic–oleophilic characteristics. Typical natural organic sorbent from moss was selected and modified in three different ways: processed with hot water (80 °C and 100 °C), mercerized and coated with oil–water (10% and 50%). Water, diesel sorption capacity, and oil retention tests were performed. Tests showed that simple treatment with hot water and alkali can change surface properties and improve sorption capacity. Modification with hot water at 80 °C enhanced sorbents' oil sorption capacity and showed the best results but meanwhile, this method readily increased hydrophilicity. This method of sorbent treatment could not be applied in cases where sorbents are used to clean oil spills from water surfaces. Meanwhile, better sorption results would be obtained in cases where cleaning operations take place on soil and other solid surfaces.

Keywords: sorption, oil spills cleanup, natural sorbent, sorbent modification methods, hydrophobicity.

Introduction

Accidental oil spills or discharges occur mainly during transportation, production and refilling activities. These spills are very dangerous to ecosystems (Galblaub et al., 2016; Annunciado et al., 2005).

Extraction, transportation and accidents are the main reasons of oil and its products entering the environment (Juteau et al., 2003).

Six sources of oil pollutants entering the soil could be identified:
- industrial waste (37%);
- mining, oil production (33%);
- former military zones (15%);
- oil and chemical storage (10.5%);
- transportation spills (4%);
- other sources (0.5%).

Even if a spill occurs on the surface of the land, not only the soil but also the pollution of the groundwater should be considered. The spread of oil products accounts for 53% of total groundwater pollution, and with groundwater pollutants, it also enters the human body (Panagos et al., 2013).

The negative effect of chemical compounds depends on their solubility, bioavailability and cancerogenicity. Hydrocarbons are pollutants that accumulate in human tissues. There is data that show the impact of these pollutants on an increase of incidence of liver cancer and leukemia (Badawi et al., 2000).

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The light fraction volatile hydrocarbons are readily biodegradable, but the less volatile hydrocarbons persist in the soil for a long time and that's why they need to be removed.

Many methods, such as chemical degradation, combustion *in situ* and mechanical extraction are used to remove oil from the environment. Among all of the used oil removal methods, sorption is a popular technique because it is an effective method to collect and capture spilled oil.

There are many natural, synthetic and modified sorbents used for oil-polluted territory treatment: carbon aerogels, graphene or carbon nanotubes (Gupta & Nyan-Hwa, 2016), cellulose sorbents prepared from corn straw (Li et al., 2012), treated polyurethane sponges with gasoline and silica sol (Wu et al., 2014), nanoporous sorbents from polystyrene (Lin et al., 2012), polypropylene sorbents, produced by a needleless melt device (Li et al., 2014), compositions of granulated polystyrene and carbonized sunflower husk (Baiseitov et al., 2015), polyurethane and polyimide (Barry et al., 2017), modified non–woven polyethylene terephthalate sorbents (Atta et al., 2013), biocompatible porous monoliths produced from polylactic acid with graphene oxide (Liu et al., 2015). Lignocellulose, cellulose and hemicellulose are the main components of sorbents made from natural organic materials (Aqsha et al., 2017).

Highly efficient oil sorbent must correspond not so easily combinable characteristics, such as good hydrophobicity and oleophilicity. Meanwhile they must have a large sorption capacity, high buoyancy, fast oil sorption rate and low cost (Wang et al., 2013).

Lignin and cellulose determine the hydrophilic characteristics of the sorbent. That's why besides sorbing oil products sorbents from natural materials also absorb water. It decreases collection efficiency and has an unfavorable impact on cleanup because water occupies a large part of the sorbent pores and initiates sinking of them (Galblaub et al., 2016).

Sorbents that show hydrophobic–oleophilic characteristics have been found suitable for oil/water separation (Aydin & Sonmez, 2015). Hydrophobic–oleophilic characteristics determine parameters such as the chemical composition of the sorbent, the physical configuration of the fiber, the amount of surface wax, the porosity of the surface and the roughness. These characteristics also depend on the type of oil, quantity, temperature, specific conditions and temperature (Abdullah et al., 2010; Hasanzadeh, 1993). Because of their high selectivity for oils, these sorbents can be used for the removal of oil spills from the surface of water. It decreases the total amount of liquids handled, which later must be treated on board or onshore (Avila et al., 2014). The hydrophobic origin of the sorbent is necessary for effective hydrophobic pollutants like oil removal from surface water. It is not easy to find or synthesize single material with mentioned properties. Therefore, there is still an actual demand to develop new materials or modify existing ones that will absorb oil quickly and exclude water. These materials should also be renewable in the long term. Therefore, various modification methods have been tested to change the surface properties of sorbents with the aim to increase hydrophobic–oleophilic characteristics (Galblaub et al., 2016; Aydin & Sonmez, 2015). Angelova et al. (2011) obtained the composite material containing C/SiO_2 by pyrolysis of rice husks at a temperature of 480 °C. This sorbent showed very good buoyancy and high hydrophobicity. However, carbonization is a costly and time-consuming process (Wahi et al., 2013).

Different chemical and physical methods could be used for surface modification of sorbents.

Thermal processing at low temperatures, such as drying and pretreatment, is not enough effective method to significantly increase sorbent hydrophobicity (Rengasamy et al., 2011). Chemical treatment involves mercerization, benzoylation, acetylation treatment, grafting etc.

Mercerization by alkaline is one of the most popular chemical treatment methods used to change the properties of natural-origine sorbents. This method is used due to its simple application and effectiveness (Wang et al., 2012). The main methods for the modification of cellulose–based sorbents are etherification, esterification, halogenations, mercerization, peroxide, oxidation, benzoylation, acetylation and coupling agents with or without heat (Hokkanen et al., 2016; Kabir et al., 2012). Although these hydrophobizing treatments are effective, acceptable in diversified applications and implemented on an industrial scale, they have a negative side. This type of modification adds chemical substances and handling, disposal and utilization of sorbents become unattractive. Therefore, surface modification of natural sorbents can be done by the application of alternative methods and avoiding use of chemical treatment (Kalia et al., 2013).

Physical modification methods such as pyrolysis require a lot of energy and are expensive. New methods should be adopted for modification of sorbents surface and environmentally friendly methods are an excellent alternative for this.

Ecofriendly methods such as hot water treatment, bacteria treatments, lignin coating and modification with plant triglycerides can be applied for the modification of the surface of natural sorbents (Dankovich & Hsieh, 2007; Morent et al., 2008; Yang et al., 2017). An increasing interest has been noticed in literature about application of ecofriendly and biodegradable reagents. To foster use of sorbents from natural materials, development of their properties using ecofriendly techniques should be precedence. In this study natural sorbent – moss (Rhytidiadelphus triquetrus) was used as sorbent. Rhytidiadelphus triquetrus, the big shaggy-moss or rough goose neck moss, is a species of moss in the family Hylocomiaceae. It is often the dominating moss species in moderately rich forest habitats in the Boreal regions and use of it will not harm the environment.

The purpose of this study is to compare different natural sorbent (moss) modification methods impact on their hydrophobicity increase.

Method should be as much as it is possible environmentally friendly.

1. Methodology

1.1. Materials and methods

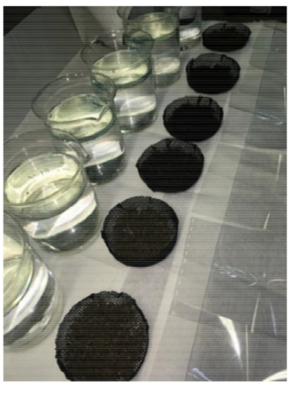
Different methods and solvents were used for the hydrophobicity increase of the sorbent from natural organic material, moss (*Rhytidiadelphus triquetrus*) hydrophobicity increase.

Three modification methods were selected: moss treatment with alkaline solution (5% NaOH); treatment with deionized hot water (80 °C and 100 °C); coating with 10% and 50% concentrations oil–water emulsion.

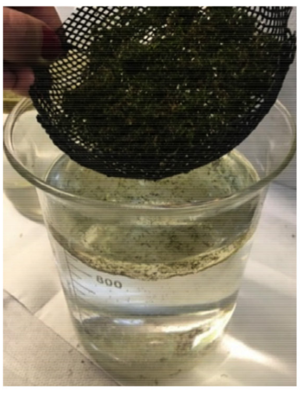
The treated and untreated samples were air dried (in 20 °C approximately for 24 hours) and then dried at 105±2 °C till a constant mass. Sorbents were placed in plastic zipped bags.

1.2. Water and diesel sorption capacity test

Standard Test Method for Sorbent Performance of Adsorbents (ASTM F726-12) was used for evaluation of water and diesel products sorption capacity. In this experiment loose–form of sorbent (size 3–10 mm) were placed in mesh basket and immersed into a beaker filled with water and diesel (Figure 1a). In the oil sorption capacity test, the beaker was filled with 800 ml of deionized water. In this experiment as oil product diesel of 0.826 g/cm^3 density at 20 °C and viscosity 4.46 cSt was used. 80 ml of diesel was poured on top of it. This quantity of diesel was chosen trying to ensure that the entire area of the sorbent surface is accessible to the diesel. For the water sorption capacity test, deionized water was used. The same amount of 800 ml was used and filled into the beaker. The amount of sorbent was selected under visual estimation – to cover the surface area in the beaker. In each experiment the weight of the sorbent after treatment was different because of different treatment techniques were used. The mesh with the sorbent after 1 h was removed from the beaker, excess liquid was taken away by keeping the mesh above the beaker for 2 min (Figure 1b). The experiment was tripled and the average value was calculated.



a)



b)

Figure 1. Diesel/water sorption test: a – beakers filled with liquid and sorbent in the mesh, b – liquid excess elimination (author's photo)

Sorption capacity is the mass of chemical compound absorbed by 1 g of dry mass of sorbent and is calculated using Equation (1):

$$M_{H_2O/\text{Diesel}} = \frac{\left(M_A - M_B\right)}{M_D}, \tag{1}$$

where: $M_{H_2O/\text{Diesel}}$ – sorption capacity, g/g; M_B – a weight of the beaker before immersion, g; M_A – a weight of the beaker after immersion, g; M_D – a weight of the dry sorbent, g.

Water/Diesel absorbency tests were performed at room temperature (20±2 °C).

1.3. Diesel retention test

Diesel products retention describe capability of sorbent to maintain and store them. It's very important feature after sorbent application and further transportation into disposal site. After sorbent application on diesel, mesh is taken out from the beaker, placed on the plate and left for 24 h. After that, the mesh with the sorbent is removed and the plate mass is determined. Amount of diesel released by the sorbent is calculated using Equation (2).

$$M_{\text{Diesel}} = \frac{(M_R - M_P)}{M_D}, \tag{2}$$

where: M_{Diesel} – the amount of diesel released by the sorbent g diesel/g sorbent; M_P – a weight of the plate, g; M_R – a weight of the plate after 24 h, g; M_S – a weight of the dry sorbent, g.

1.4. Quality control

All chemical reagents used in experiment were obtained from Sigma Aldrich (Germany). They were used without further purification i.e. as received. The solutions were prepared by mixing reagents with deionized water. Deionized water was prepared in water purification system – Demiwa 3 roi (Watek, Czech Republic). Deionized water meets the requirements of standard LST EN ISO 3696:1996 (Water for analytical laboratory use – specification and test methods). All beakers and flasks used in the experiments were only of the highest accuracy class. They were cleaned before experiments. Analytical balance of high accuracy Radwag AS 60/220.R2 was used for weighting. Three measurements were carried out for an average calculation.

2. Statistical procedures

Microsoft Excel was used for statistical evaluation of experimental data. Statistical parameters were estimated at $p < 0.05$. Mean ± standard deviation are presented in graphs.

3. Results

Diesel retention, water and dieselsorption tests were made to compare efficiency of the different methods used for

modification of sorbents. 7 types of sorbent modification methods were investigated:
- treatment with hot water at 100 °C (HW100);
- treatment with hot water at 80 °C (HW80);
- treatment with oil–water emulsion, 10% concentration (Oil10);
- treatment with oil–water emulsion, 10% concentration (Oil50);
- treatment with oil without water (Oil);
- treatment by mercerization (NaOH);
- untreated sorbent (Untreated).

3.1. Diesel sorption capacity test

As oil diesel was used in all oil sorption capacity tests. ASTM F726-12 method was used for diesel sorption capacity evaluation. Results of tests are presented in Table 1.

Table 1. Diesel sorption data

Sorbent treatment type	Mass of the sorbent, g	Mass of absorbed diesel, g
HW100	0.85	6.70
HW80	0.81	6.67
Oil10	2.13	6.18
Oil50	4.42	7.18
Oil	4.48	4.86
NaOH	0.65	5.87
Untreated	0.78	5.71

Figure 2 shows calculated diesel sorption capacity results. The highest diesel sorption was of sorbent modificated with a solution of NaOH (5%) and we can see that mercerization had positive effect on absorption capacity.

Thermal modification by hot water – 80 °C and at 100 °C temperatures, increased sorption of diesel. When the sorbent was modified with hot water at 100 °C, the diesel sorption capacity increased by 6.7% compared to the untreated one.

Effect of sorbent sorption capacity increase by 12.4% was observed when it was treated with the 80 °C temperature water and it was greater than with 100 °C. Increasing the temperature of water to 100 °C resulted the reduction of absorption characteristics of the sorbents. This coarser and undulant surface cannot only increase the fiber surface area but also improve the adhesion property of oil on the sorbent surface (Wang et al., 2013).

Reduction of diesel sorption capacity of the sorbent was observed after coating of the sorbent with oil. Immersion of sorbent in mixture containing oil and water at concentrations of 10% and 50% showed a decrease in diesel absorption by 60.6% and 78.0%, respectively.

Typically adsorption capacity of selected sorbent is presented as sorbed mass of pollutant divided by mass of sorbent. Other properties of selected sorbents – density and volume should be considered. Data of sorption capacity by its volume are presented in Figure 3.

Effectiveness of sorbent diesel sorption capacity by its volume and effectiveness of sorbent diesel sorption capacity by its mass are different.

Comparing sorption capacity by its volume shows very good result of hot water at 80 °C and mercerization impact on increase of the diesel sorption capacity.

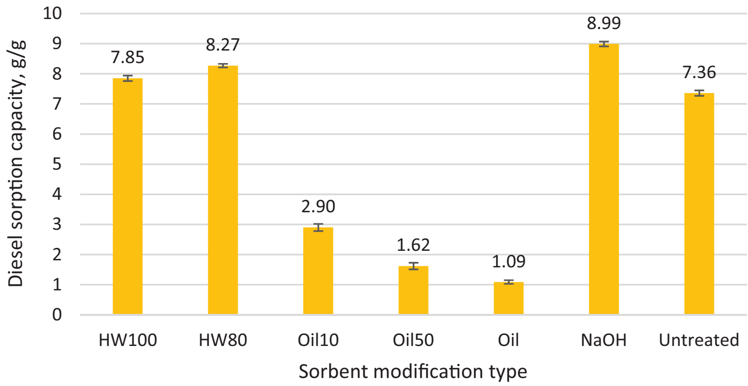


Figure 2. Diesel sorption capacity by mass

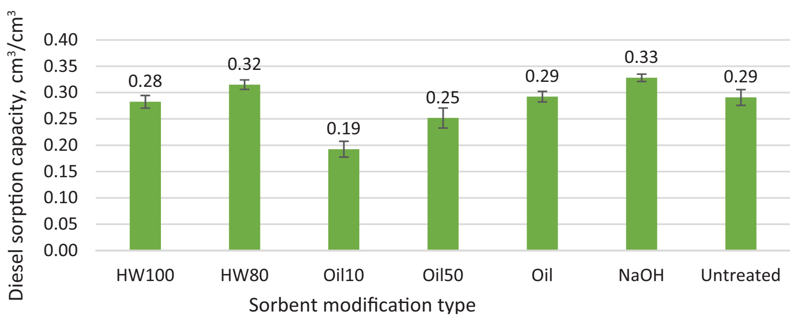


Figure 3. Diesel sorption capacity of sorbent

3.2. Water sorption capacity test

Deionized water was used for performing of water sorption capacity test of selected adsorbents.

ASTM F726-12 method was used for water sorption capacity analysis. Results of experimental research are presented in the Table 2. M_w is the mass of water absorbed by the sorbent, g; M_S – a mass of the sorbent, g.

Results of water sorption capacity research are presenting in Table 2 for each type of treated sorbent.

Table 2. Results of water sorption experiment

Sorbent	M_S, g	M_w, g
HW100	0.81	14.72
HW80	0.71	11.36
Oil10	2.05	12.36
Oil50	4.01	11.53
Oil	3.74	9.29
NaOH	0.81	11.09
Untreated	0.76	11.00

As seen in Table 2, when sorbent is coated with the oil the amount of water absorbed by the sorbent is reduced. Hydrophobity of the investigated sorbent is increasing with increasing of its coating with oil.

Coating of moss with oil at 10% and 50% concentrations was the reason of water sorption reducing by 58.5% and 80.2%, respectively. Moss coating by pure oil increased the resistance to water by 82.9%. High surface area is typical for such sorbent.

Water cannot penetrate the sorbent, due to the coating of the moss surface and its pores with oil.

Results of water sorption capacity are presented in Figure 4.

Coating of sorbent with oil and mercerization enhanced sorbent hydrophobicity.

Modification of sorbent with water at 100 °C, increased its sorption capacity by 26.5% compared with the untreated one. Smaller hydrophobicity reduction (increased its sorption capacity by 10.9% compared with the untreated one) was observed under the application of cooler water 80 °C.

Experimental results of water sorption by modified sorbents calculated by its volume are presented in Figure 5.

Sorbent water capacity by its volume and sorbent water capacity by its mass are different.

Mercerization and coating with oil in water emulsions cause increasing of sorbent hydrophobic properties. Increasing of water sorption was observed when sorbent was coated with oil not in aqueous emulsion. It could be concluded that all the modifications of selected sorbents changed the diesel and water sorption properties of moss.

Enhancing of sorbent oleophilicity and reducing of its hydrophobicity was observed after sorbent treatment with hot water.

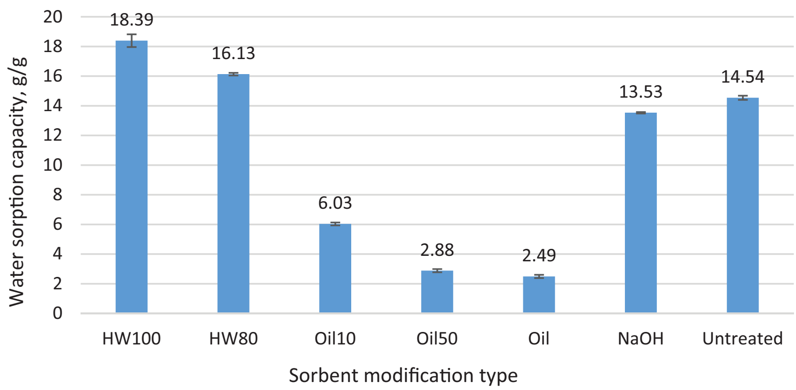


Figure 4. Sorbent water sorption capacity

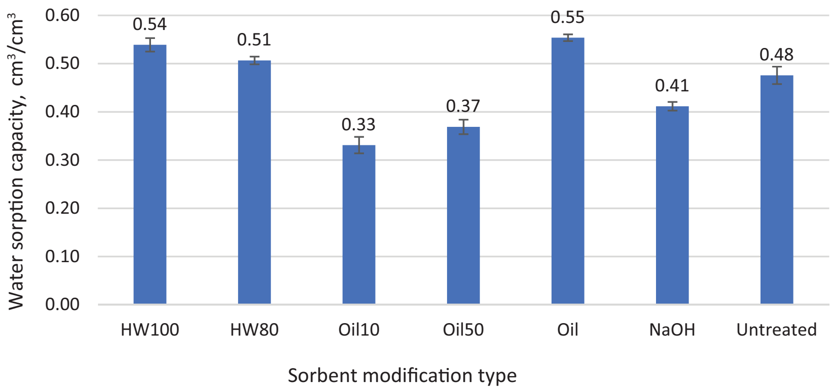


Figure 5. Water sorption capacity of sorbent

Hot water modification process removes volatile compounds, extractives and waxy coatings from the cellulose surface, making it more accessible for the absorption medium. Sorbent surface area is increasing due to impact of hot water on sorbent fibers swelling. Moss sorption capacity have increased not only for diesel, but also for water due to these changes in the structure of the fibre.

This environmental friendly modification type of sorbent modification can be used for removal of oil products from the solid surfaces but not from the water surface due to possible increasing of water absorption capacity. Treating of selected sorbent with 80 °C water is optimal.

Effect of significant improving of sorbent hydrophobicity and reducing of sorption capacity of diesel is registered after sorbent surface covering with oil.

Increasing of sorbent hydrophobicity and oleophilicity is observed after application of its chemical modification – e.g. alkalization of sorbent. Alkali treatment of the sorbent is a popular modification method used by scientists. Environmentally friendly modification of selected sorbents with hot water at 80 °C and alkalization did not show a significant difference in the sorption capacity of sorbents for diesel.

Mercerization increased the absorbance of diesel by sorbent at the level of 22.0% and its treatment with hot water at 80 °C increased it by 12.4%.

3.3. Diesel retention test

Collecting of used sorbent and handling operations can be the reason of additional pollution of environment and that is why is very important to use effective sorbents capable to retain the absorbed oil. Due to mentioned reason oil retention rate is very important parameter.

The oil retention rate could be defined as percent of released oil after 24 h. Experimental results of diesel retention by selected sorbents are presented in Figure 6.

As shown in Figure 6, the oil product release rate from raw moss is the highest. All types of treatments increased the diesel holding capacity.

The most effective treatment methods of sorbent for improving of its sorption capacity is rinsing with hot water at 80 °C and mercerization: the sorbent releases 0.75% and 0.88% of absorbed diesel fuel, respectively.

Treatment of sorbent did not increased the sorption capacity of diesel as much as the above mentioned methods. The result was still positive.

4. Discussion

Moss can store large amounts of water within their cells. The adsorption capacity of water can be 16–26 times higher than their dry weight, depending on the species of moss (Bold, 1967). High amount of water – 14.54 g absorbed by 1 g of unmodified sorbent is due to this property of plant. Mercerization of the moss was the reason for decreased water absorption by almost 7%. Different concentrations of alkali were used in experiments by scientists and even low concentrations of NaOH for treating of cellulose fibers. Reducing of fiber diameter and decreasing of surface area can be observed as a result of fibers swelling (Wong et al., 2016).

Swelling can be observed as effect of alkali on the cellulose component of the sorbent and the natural crystalline structure of the cellulose relaxes. The degree of swelling and the degree of lattice transformation into cellulose depends on the type of alkali and its concentration.

One of most popular chemicals containing Na+ and widening the smallest pores between the lattice planes for effective penetration is sodium oxide. New Na–cellulose lattice is formed after removal of excess of NaOH.

Conversion of the –OH groups of the cellulose into ONa– groups is observed. New crystalline structure – cellulose is formed after conversion of cellulose. New product contains a lattice that is more stable than cellulose (Weyenberg et al., 2006). Hot water under 100 °C and 80 °C temperatures increased moss hydrophilicity.

The highest mass of diesel was sorbed by 1 g of sorbent modificated with a solution of NaOH (5%). Mercerization had positive effect on absorption capacity. This method enhanced it up to 22%. This could be explained by alkali treatment impact on the surface roughness of moss fibers. It caused swelling and incensement of surface area that led to improved diesel absorption.

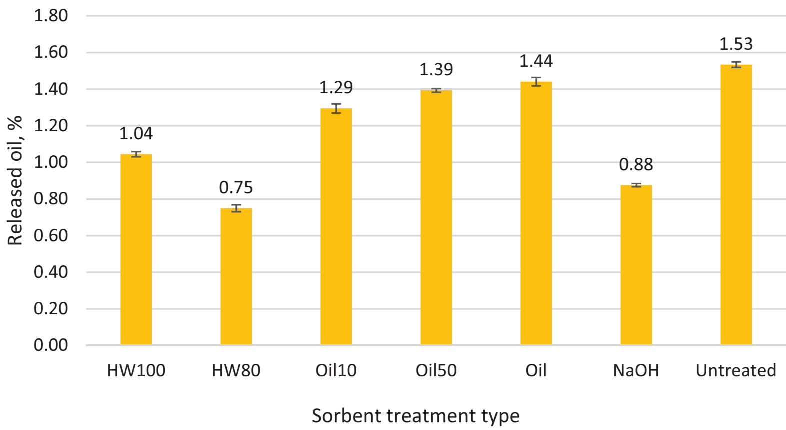


Figure 6. Results of dieselretention test

Smaller hydrophobicity reduction was observed by application of cooler water 80 °C. According to results of experiment, the lowest acceptable temperature for removing of volatile organic compounds (VOC) is 80 °C. At this temperature wax coating could also be removed from the surface of moss and the surface is becoming more accessible to the sorption. In higher temperature the external surface area of the fibers is becoming more accessible to water due to its swelling by hot water. It also refers to the larger dissolving part of lignin included in the moss fibers (Amer et al., 2007).

The lowest diesel penetration capacity was observed when moss was coated with oil not in aqueous emulsion. It was reduced by 85%. It could have happened due to covering of the largest part of the sorbent area and filling of the pores of the sorbent with oil. Sorbent treatment with hot water at 100 °C hardly changed sorbent properties. It could be concluded that diesel absorption by oil coated sorbents according to its volume was higher comparing to diesel absorption by sorbent mass. This kind of moss treatment had minimal effect on increasing of the adsorption capacity of diesel.

A proportional decreasing in the sorption capacity of the sorbent for diesel and water was found in this study with increasing the degree of the sorbent coating with oil. Therefore, it can be assumed that the oil sticks to the surface of the selected sorbent and does not pass water or diesel. The larger the area of the sorbent covered with oil, the lower the observed sorption capacity of the sorbent.

The moss coated with oil had better water resistance comparing with case using the chemical treatment – the alkali treated sorbent absorbed 13.53 g/g and the oil treated sorbent absorbed only 2.49 g/g. Oleophilicity of the sorbent was registered up to 8.99 g/g for diesel using alkalization and 1.09 g/g using coating with oil. Therefore, when using a sorbent coated with oil, a low concentration of oil should be used on the sorbent surface.

It was observed that evaluation of sorbents should be done not only by their sorption capacity by weight but also by volume. Taking into account how much 1 cm^3 of sorbent absorbs diesel, it can be concluded that its treatment with hot water at a temperature of 80 °C and mercerization increase the absorption of diesel.

Depending on the amount of dieselabsorbed by 1 cm^3 of sorbent, it's treating with oil in water emulsions and alkali may increase its hydrophobicity.

Conclusions

1. Evaluating of the influence of different sorbent treatment methods for the adsorption capacity of diesel showed that the maximum absorption capacity was achieved using mercerization (8.99 g/g), treatment with hot water at 80 °C (8.29 g/g) and at 100 °C (7.85 g/g). The use of these methods has led to swelling of the cellulose fibers. Sorption capacity of the sorbent due to an increasing of its surface area was a result of this effect.

2. Diesel penetration decreased due to sorbent surface coating with oil. This effect may have been occurred due to the coating of the sorbent surface area and the filling of its pores with an oil–in–water emulsion. As a result, the adhesion of diesel and water to the sorbent surface was reduced.

3. Water absorption was found to be reduced by almost 7% due to sorbent mercerization.

4. It was found the difference between sorbent water capacities expressed by volume from its capacity expressed by mass unit.

5. Depending on the dieselabsorption by 1 cm^3 of sorbent, treatment of sorbent with hot water at 80 °C and mercerization are capable to improve the absorption of diesel.

6. It was observed that all of the selected sorbents modification methods increased the retention capacity of the diesel. Best retention show sorbents modificated by hot water treatment at 80 °C and mercerization. Under these conditions sorbent released only 0.75% and 0.88% of the absorbed diesel.

7. Sorbent treatment with hot water and alkali is responsible for changing of moss surface and fibers characteristics and improves diesel absorption. Treatment of sorbent with 80 °C hot water is perspective because of low treatment costs.

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Conflict of interest

The authors declare that they have no conflict of interest.

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