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OIL REMOVAL FROM REFINERY WASTEWATER THROUGH ADSORPTION ON LOW COST NATURAL BIOSORBENTS

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Abstract

The performance, in terms of sorption properties, of the two natural sorbents were compared with the commercial synthetic sorbent Fibroil. The experiments were conducted using two continuous setups of filtration under dynamic conditions. Experimental results revealed that the removal of chemical oxygen demand (COD) was 89%, 87% and 37% for Fibroil, *Schoenoplectus lacustris* and *Acorus calamus* respectively in a down-flow filtration, while with the up-flow filtration the removal efficacy of COD was 96% and 89% for Fibroil and *Schoenoplectus lacustris* respectively. *Schoenoplectus* lacustris presents a great potential to be used as an inexpensive and easily available alternative bio sorbent for the removal of oil from wastewater.

Keywords: adsorption; filtration, natural sorbent, oily wastewater

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1. Introduction

Petroleum based products are one of the most important energy sources for the industrialized countries. During the production, transportation, storage, utilization and disposal of oil products can induce accidental and operational environmental pollution. Furthermore, the petrochemical industries generate significant amounts of oily wastewaters, which are heavily loaded with toxic organic compounds. Toxic wastewaters are causing comprehensive environmental and health problems (Mciwem et al., 2010). The treatment of said wastewater has been a substantial challenge for various industrial companies, which has to adhere to stringent environmental regulations. The oil wastewater constitutes a large amount of the chemical oxygen demand (COD) in the effluent, they seem to be a good indicator of the organic matter content. The COD is commonly used to indirectly measure the amount of organic compounds in oily wastewater.

In order to reduce these adverse effects on the environment, several treatment technologies have been used for the reduction of COD (Ayhan et al., 2017), such as coagulation-flocculation (Fu, 2017; Ighilahriz et al., 2018), flotation (Santo et al. 2012), advanced oxidation technologies (Bustillo et al., 2018), and adsorption (Sirotkina and Novoselova, 2005). All of these treatment technologies are available with a different degree of success to minimize this pollution. Adsorption or filtration using an inexpensive and available porous sorbents (Sabir, 2015; Yanhong et al., 2018) provides an attractive alternative treatment, due to its simplicity of design

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and relatively lower processing cost in comparison to other wastewater treatment technologies (Ho and Mckay, 2003). The commercial sorbents such as activated carbon and Fibroil are the most widely used sorbents in many industrial processes because of their high adsorption capacity to remove organic matter, petroleum products (Arroyo et al., 2019; Mažeikienė et al., 2014) from landfill leachate (Chaouki et al., 2017), industrial wastewater (Asenjo et al., 2011), pig manure digestate (Carney et al., 2016). However, the use of these sorbents is being limited by their high operating costs, potential safety issues and other problems associated to their regeneration and recycling. From this point of view, effective and low-cost alternative sorbents are being sought after.

In order to replace commercial sorbents used in the treatment of wastewater from the refining industries, many researchers recently have focused on the preparation of sorbent from a natural waste made from fruit trees, banana peels (El-din et al., 2018), cattail fiber (Shengbin et al., 2017), wool and reeds (Mažeikienė et al., 2014). Natural materials could be used as a low-cost, environmentally friendly sorbents in wastewater treatment. The main objective of the study is to research the characteristics and efficacy of natural sorbents collected from Lithuanian natural bodies of water (lakes) to remove the COD from oily wastewater, and to compare their efficacy with a synthetic sorbent alternative. The novelty of the research is related with the additional value of waste biomass utilization. Additional advantages are that natural bio sorbents are inexpensive materials and can be used with small amount of processing for wastewater treatment.

2. Material and methods

2.1. Preparation of wastewater

Two types of mixtures of oily wastewater were prepared with different initial COD concentration. Mixtures were prepared by mixing 5 ml of commercial oil (diesel, class C) per liter of a domestic wastewater - First mixture (I) (COD concentration=790-900 $mgO_2 \cdot L^{-1}$, pH = 7.4) and Second mixture (II) (COD =1700-1800 $mgO_2 \cdot L^{-1}$, pH = 7.4). Two different levels concentrations of COD were chosen to better evaluate and understand the variations when treating different levels of contamination of wastewater.

The effluent samples used in this study were collected from the small wastewater treatment plant (WWTP), located in suburban area of Vilnius, Lithuania. Their COD concentrations were kept in the constant range of 120-160 $mgO_2 \cdot L^{-1}$ and 1000-1120 $mgO_2 \cdot L^{-1}$. The COD characteristics of each aquatic mixtures (AM) used in this study are presented in Table 1.

2.2. Preparation and characterization of sorbent

The materials investigated as sorbents in the study, namely *Schoenoplectus lacustris* and *Acorus calamus* (collected from the lakeshore) were compared with the synthetic sorbent Fibroil with the aim of evaluating their potential as sorbents for the removal of oil from wastewater. The first investigated sorbent was *Schoenoplectus lacustris*, a species of *Cyperaceae* plant family. *S. lacustris* is a tall rhizomatous perennial herb that grows up to 3.5 m tall, with stems of 5–15 mm thick. The second investigated material was *Acorus calamus*. It is also an aquatic plant. About one meter high, with erect linear leaves.

Both sorbents were acquired from the lakeshore of a natural water body located near Vilnius, Lithuania. Plant stems were cut into 1.0 - 1.5 cm^2 segments and washed with deionized water to remove any adhering substances and soluble pigments. Afterwards plant samples were dried at 95°C for 24 hours duration.

The third sorbent (Fibroil) was selected for a baseline comparison of the results. The synthetic sorbent Fibroil constitutes of polypropylene and polyethylene fibers with a calcareous stone additive. It is a popular choice for oil separation. Fibroil can only be disposed of by burning it. Morphological features of the sorbents were analyzed by MOTIC B1 microscope.

2.3. Batch sorption and filtration experiments

Oil sorption process was investigated in the continuous tubular and batch experiments. The capacity of sorption of each sorbent has been studied both in water and oil. Dynamic sorption experiments were carried out using one of two setups: down-flow and up-flow. Sorption efficacy was determined by the measure of COD of the samples taken at outlet of each setup at regular time intervals. Finally, booms of sorbents were evaluated to determine their capacity to remove and limit oily wastewater.

2.3.1. Batch sorption

Oil and water sorption experiments were carried out using the batch method according to the standard*: Standard Test Method for Sorbent Performance of Adsorbents* (ASTM F726-12, 2012).

Table 1. Characterization of aquatic mixtures (AM) and comparison to reject requirements

Mixture type	*Mixture No.*	*COD $mgO_2 \cdot L^{-1}$) (Wastewater)*	*COD ($mgO_2 \cdot L^{-1}$) (Wastewater + 5ml·L^{-1} diesel)*
	AM 1	158	834
I	AM 2	126	912
	AM 3	125	791
II	AM 4	1120	1804
	AM 5	1097	1719

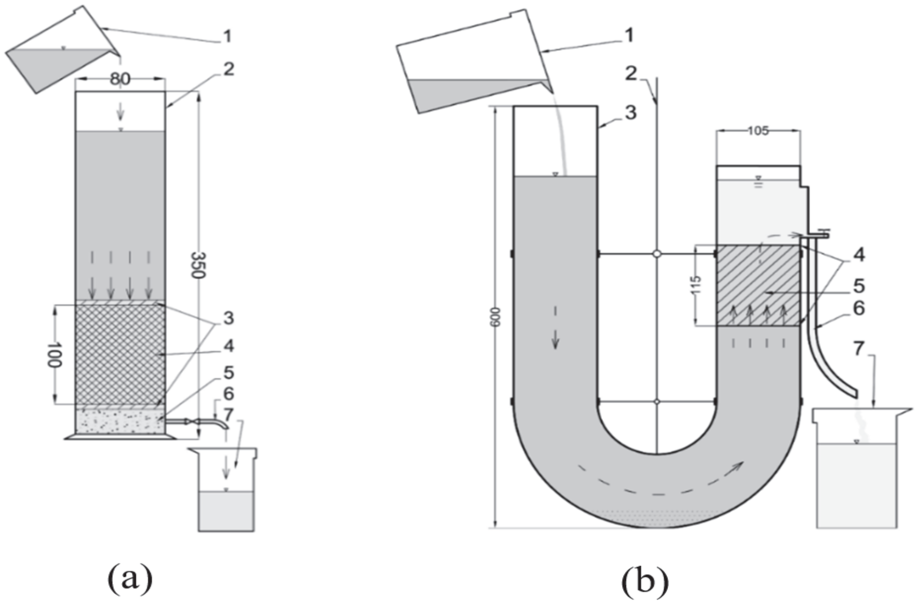

Fig. 1. Filtration setup - **Setup (a):** (1) beaker of aquatic mixture (2) filter column (3), (4) perforated disks (5) sorbent media (6) flexible hose for filtrate samples (7) filtrate recovery beaker; **Setup (b):** (1) beaker of aquatic mixture (2) support (3) filter setup, (4) perforated disks (5) sorbent media (6) flexible hose for filtrate samples (7) filtrate recovery beaker

The sorbent sample (2 g) was placed in a fine mesh basket and was immersed inside a beaker filled with 100 cm^3 of oil or water for 15 min, followed by a step in which the sorbent is removed from the bath and allowed to drain for 2 minutes (dripping time). One the excess of the liquid was removed; the sorbent was weighed.

The same experiment for oil is conducted, but for 24 hours contact time instead of 15 minutes. Experiments were performed at room (25°C) temperature and every test was conducted in triplicate. The sorption capacity (*SC*, $g_{sorbate}\cdot g^{-1}{}_{sorbent}$) was determined according to Eq. (1):

$$SC = \frac{S_i - S_0}{S_0} \quad (1)$$

where S_0 (g) and S_i (g) are the weight of the sorbent sample before and after of oil sorption test, respectively, and (S_i-S_0) (g) is the net oil adsorbed.

2.3.2. Filtration experiments

Filtration tests were carried out using two setups installed in the laboratory of the Environmental Protection and Water Engineering at Vilnius Gediminas Technical University. The experimental setups are presented in the Fig. 1.

Dynamic sorption experiment was carried out using the first setup (Fig. 1 (a)). It is composed of a polyvinyl chloride (PVC) column (external diameter of 7.8 cm, and a height of35 cm) and was loaded with the known masses of sorbents (see setup device (Fig. 1 (a)). During each experiment, the selected height of filter filling in the cylinder was 10 cm. Two perforated disks encapsulated the sorbent media to keep it in place. The mass of the sorbent corresponding to the bed height of 10 cm was equal to 28.35 g, 145 g and 31 g for *Schoenoplectus Lacustris*, *Acorus calamus* and Fibroil, respectively. The oily wastewater (I) was prepared by continuously mixing domestic water with an oil at 5 $ml\cdot L^{-1}$ concentration. This concentration was chosen to achieve the value of COD~800 $mgO_2\cdot L^{-1}$. The mixture was introduced into the filtration setup on a down-flow stream. Analysis of the filtration performance was based on the experimental data recorded when the filtration was performed at the flow rate of 2.4 $L\cdot h^{-1}$.

The second study was conducted using the U-shape setup: 10.5 cm external diameter and 60 cm height. This form allows utilization of the up-flow stream, which is considered to be the best for filtration of a highly contaminated wastewater. Also, up-flow allows the experiment to be performed without using a peristaltic pump. The wastewater was filtrated through an 11 cm high sorbent media, packed between two perforated disks in order to prevent loss of the sorbent. For this experiment 62 g of *Schoenoplectus Lacustris* and 67.8 g of Fibroil were used (Fig. 1 (b)).

The effluent II used in the experiment was prepared by mixing the domestic wastewater loaded on activated sludge with a COD (~1000-1100) $mgO_2\cdot L^{-1}$ and an oil concentration of 0.5 $mL\cdot L^{-1}$. COD of this aquatic mixture was ~1700 $mg\cdot L^{-1}$. The filtration flow was fixed at 2.7 $L\cdot h^{-1}$.

Filtration experiments were conducted at room temperature, in the pH range of 6.8. - 7.1. Samples were collected at the outlet of each treatment unit, at a regular time intervals and were further analyzed for COD removal efficacy.

2.4. Sorbent booms

Sorbent booms constitutes a major part of equipment used in marine oil spills response and treatment as they are efficient in absorbing oil floating on the surface of the water. Two sorbents booms were made from Fibroil and *Schoenoplectus lacustris* respectively. They were evaluated in terms of the buoyancy, retention and limitation of oil in an artificial marine environment made of a mixture of 1 L of water and 20 cm^3 of oil.

2.5. Analytical methods

Chemical oxygen demand (COD) measurement is used to quantify the organic matter contained in oily wastewater. All samples were characterized before and after the treatment. Analysis of chemical oxygen demand (COD) is performed using the titrimetric method in accordance with the standard methods for wastewater examination (APHA, 2012). The COD removal efficacy (*% COD*) was obtained using the following equation (Eq. 2):

$$\%COD = \frac{(COD_0 - COD_t)}{COD_0} x100 \qquad (2)$$

where COD_0 refers to COD of wastewater before and COD_t refers to the metric after treatment.

3. Results and discussion

3.1. Sorbents microscope image

Fig. 2 illustrates the rough surfaces of the three sorbents as seen under a microscope MOTIC B1 (magnitude ×40) with achromatic objectives. It is noticeable that Fibroil and *Schoenoplectus Lacustris* sorbents have a fibrous structure, an irregular surface and a heterogeneous porosity. This structure facilities oil entering into the internal parts of the material for easier sorption process. However, the *Acorus calamus* structure seems to be different than structure of the other two materials. It presents a non-fibrous surface, and the presence of the holes, which appears clearly in the image.

3.2. Sorption capacity

Fig. 3 shows the amount of sorbed oil into *Schoenoplectus lacustris*, *Acorus calamus* and Fibroil sorbents using a dose of 2 g of each sorbent in ether 100 mL of water or oil solution at room temperature. As it is evident in Fig. 3, the sorption capacities of Fibroil, *Schoenoplectus lacustris* and *Acorus calamus* for oil in batch system were 7 $g_{oil}/g_{sorbent}$, 4.05 $g_{oil}/g_{sorbent}$ and 1.39 $g_{oil}/g_{sorbent}$ respectively. Fibroil showed highest sorption capacity compared to the other two sorbents.

However, the highest water sorption capacity value found was using *Acorus calamus* with 3.76 $g/g_{sorbent}$ followed by 2.39 $g/g_{sorbent}$ and 1.37 $g/g_{sorbent}$ for *Schoenoplectus lacustris* and Fibroil, respectively. Hydrophobic materials are considered to be the most efficient adsorbents for organic compounds from water solutions, as their adsorption is mainly based on the dispersion force (Rotar et al., 2014).

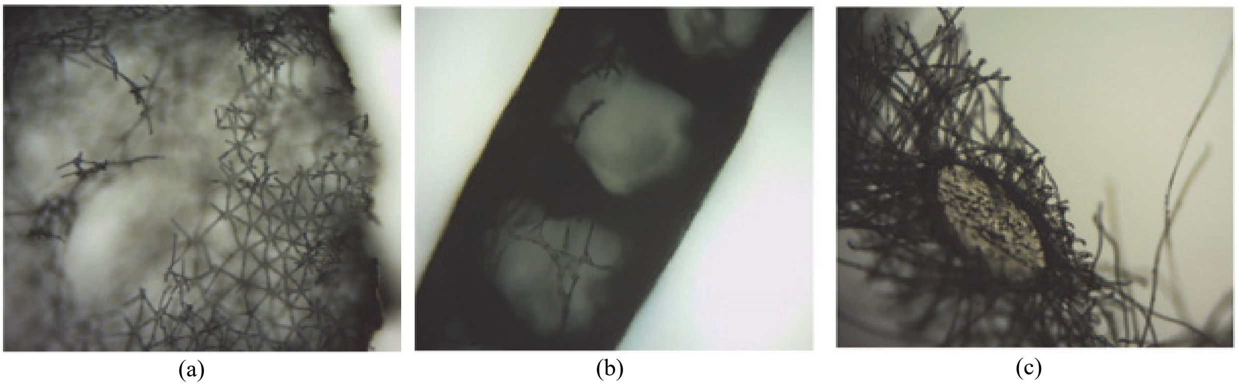

Fig. 2. Microscope MOTIC B1 images of the sorbents: (a) *Schoenoplectus Lacustris*, (b) *Acorus calamus* and (c) Fibroil

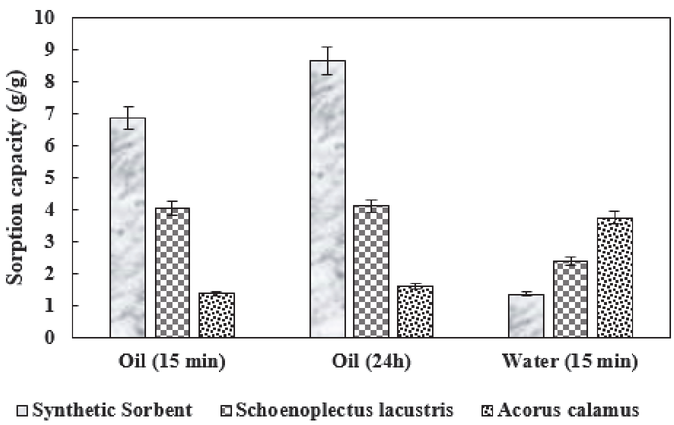

Fig. 3. Sorption capacity of oil for different sorbents

This proves Fibroil's higher hydrophobic and oleophilic proprieties than other sorbents, followed by *Schoenoplectus lacustris, Acorus calamus* sorbent which presents low selectivity. From results of oil sorption capacity after 24 hours presented in Fig. 3, it can be remarked that the sorption capacity for Fibroil increases, as opposed to the two natural sorbents (*Schoenoplectus lacustris* and *Acorus calamus*) which remains constant and is not influenced by contact time.

In order to show the advantages of the application of sorbents used in the study in the removal of organic compounds from wastewater, the amount of adsorbed oil was compared to the previous records of various low-cost sorbents efficiency under similar conditions. Previous records: kapok fiber – 38.9 $g_{oil}/g_{sorbent}$ (Wang et al., 2013); sunflower pith – 9.56-12.67 $g_{oil}/g_{sorbent}$ (Knapik and Stopa, 2018); saw dust – 4.5-8.5 $g_{oil}/g_{sorbent}$ (Galblaub et al., 2015); bagasse – 3.0-6.0 $g_{oil}/g_{sorbent}$ (Bayat et al., 2005); spent malted barley – 4.91 $g_{oil}/g_{sorbent}$ (Tontiwachwuthikul et al,. 2016); white rice husk ash – 2.8 $g_{oil}/g_{sorbent}$ (Annunciado et al., 2005); onion peels – 0.45 $g_{oil}/g_{sorbent}$ (Sayed and Zayed, 2006). The oil adsorption capacity of *Schoenoplectus lacustris* and *Acorus calamus* were at 4 $g_{oil}/g_{sorbent}$ and 1.5 – 1.75 4 $g_{oil}/g_{sorbent}$ respectively, which is considerably better when compared to natural sorbents such as onion peels, white rice husk ash, spend malted barley.

3.3. Filtration results

The dynamic filtration experiments were conducted to evaluate the performance of the sorbents' (*Schoenoplectus lacustris*, *Acorus calamus* and Fibroil) retention rate of oil from solution. Experiments were conducted using the aquatic mixtures (AM1, AM2 and AM3). A predetermined flow rate of 2.4 $L\cdot h^{-1}$ was maintained throughout the experiment. All filters were 10 cm high and occupied the same volume. The experiment was carried out under the same conditions for proper evaluation of the three sorbents. Highest efficacy of COD removal (98 %) was achieved using Fibroil filament. Efficacy was improving for the first hour of experiment until it leveled out and stayed at 89 % for the remaining 5 hours of the experiment. Conducting the same experiment with *Schoenoplectus lacustris* yielded COD removal efficacy of 86%, while using *Acorus calamus* amounted to only 38 %. Quite poor results of the *Acorus calamus* might be due to its non-fibrous structure.

Over the duration of the experiment 15.46 g O_2 of organic material was introduced to the filtration column. Water after treatment using Fibroil, *Schoenoplectus lacustris* and *Acorus calamus* contained 0.83, 1.56 and 5.16 g O_2 (COD) respectively. Therefore, highest level of COD removal properties was demonstrated by Fibroil (initial level of contamination was decreased 18 times). *Schoenoplectus lacustris* demonstrated a slightly lower result (initial level of contamination was decreased 10 times), while *Acorus calamus* performed the worst (initial level of contamination was decreased 3 times).

Up-flow filtration

Two sorbents, *Schoenoplectus lacustris* and Fibroil were tested under dynamic conditions of up-flow filtration. The experiment was conducted using wastewater (II) which contained COD concentration of 1800 $mgO_2\cdot L^{-1}$. In order to evaluate the performance of the sorption process of each sorbent used, the COD concentrations of the oily wastewater (II) were measured at the outlet of the filtration setup (up-flow filter) at equal increments of time (every 4 hours). The COD removal efficacy of oily wastewater after the sorption on *Schoenoplectus lacustris* and Fibroil are shown in Fig. 4. The up-flow filtration process achieved a very high removal of COD from wastewater levels. Using Fibroil as a sorbent, COD removal level reached 93 % after 2 hours of filtration and stayed constant (Fig. 4 (b)). While using *Schoenoplectus lacustris* as a sorbent yielded a COD removal level of 53 % (Fig. 4 (a)) in the first hours of the test and kept increasing until reaching 84 - 89 % of COD removal after 6 hours. From the Fig. 4 it is evident that for the first hours of filtration, the Fibroil demonstrated a higher efficacy than *Schoenoplectus lacustris* After first hours of filtration the efficacy becomes very close, within a 5 % tolerance. It is observed that after filtering 24 liters of the aquatic mixture, Fibroil sorbent demonstrated a higher efficacy in reducing COD levels (factor of 17 – initial 41.77 gO_2 were reduced to 2.55 gO_2) as compared to *Schoenoplectus lacustris* (factor of 4 – initial 41.77 gO_2 were reduced to 9.94 gO_2).

Filtration is a complex set of phenomena, with the hydraulic resistance influenced by several factors: material density, sorption properties, hygroscopicity and water absorption. As can be seen in the results of both experiments, the filtration efficacy with *Schoenoplectus lacustris* depends on the effluent load, because when its COD increases the sorption takes more time to achieve a proper removal of COD. On the other hand, the effectiveness of the treatment with Fibroil remains almost constant across the experiments. In addition, many researchers have been observing a decrease in sorption efficiency in experiments using an oil separator with sorbents during the filtration (Deschamps et al., 2003; Paruch and Roseth, 2008; Rajakovic et al., 2007) by closing the filtration system construction due to the clogging of the filter substrate, which led to a decrease in the permeability of the filters.

3.4. Experimental test of sorbent booms

The commercial sorbent booms from synthetic sorbents are usually made from polyurethane, polyethylene and different polymeric sorbents.

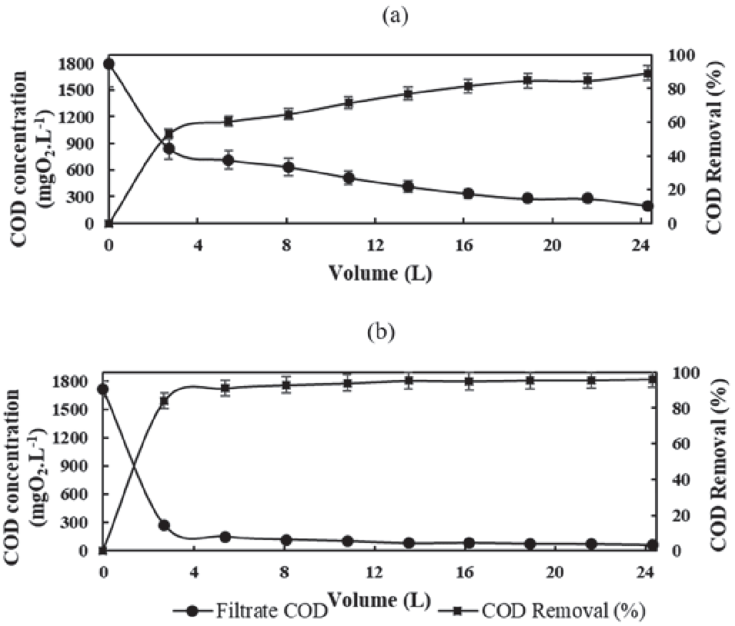

Fig. 4. Removal efficiencies of COD in Up-flow filtration from aquatic mixture using *Schoenoplectus lacustris* (a) and Fibroil (b)

These materials usually have a high hydrophobic and oleophilic characteristics, such as Fibroil used in our study. These sorbents are often expensive and non-biodegradable. The downsides of bio-based waste materials are low hydrophobicity that results in low oil sorption capacity, and buoyancy properties (Doshi et al., 2018). Since the plant *Schoenoplectus lacustris* has exhibited hydrophobicity and oleophilic properties as a result of the sorption capacity experiment, it was decided to investigate its efficiency as a sorbent boom filling. Fig. 5 shows a brief experiment where sorbents booms made from Fibroil (a) and *Schoenoplectus lacustris* (b) were evaluated.

As seen from the Figure, the two booms possess the ability to float. It appears that Fibroil adsorbs nearly the totality of the oil and contains it in the inner circle of the boom. *Schoenoplectus lacustris* also contains oil, but it's less adsorbent than the booms made out of Fibroil. Results indicates that *Schoenoplectus lacustris* can be used, not only as a sorbent, but also to make a buoyant sorbent booms to be used in the marine environment to combat oil pollution. More profound study to determine maximum of its oil retention capacity would be required.

Today, due to ecological and economic reasons, organic and inorganic natural materials used as oil sorbents include rice straw, corn cob, peat moss, wool, cotton, cotton vinegar, cattail fiber, rice husks, sawdust, bark, bagasse, milkweed, zeolite, clinoptilolite and vermiculite, mercerized pineapple leaves, bagasse and corn husk are becoming increasingly important as alternatives to synthetic sorbents (Abdullah et al., 2010; Cheu et al., 2016; Isam et al., 2016; Pachathu et al., 2016; Radetić et al., 2003; Rajaković-Ognjanović et al., 2008; Sánchez et al., 2013; Srinivasan and Viraraghavan, 2010). Traditional sorbents derived from bio-based waste materials are generally cost-effective and environmentally-friendly due to their abundance and biological origin (Doshi et al., 2018).

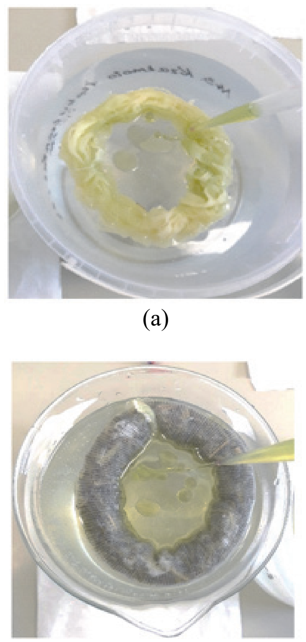

Fig. 5. Pass or fail buoyancy and oil-retention response of booms sorbent in water with oil: (*a*) Fibroil and (*b*) *Schoenoplectus lacustris*

The cost aspect has been considered: *Schoenoplectus lacustris* and *Acorus calamus* plant materials prices are around 60 €/t, which is considerably lower than alternatives. Oakum, which is 150 €/t, Reindeer moss is standing at 120 €/t and significantly (24 times) more expensive Coconut carbon sold for 1460 €/t and even 82 times more expensive Fibroil (~5000 €/t).

The use of the tested plant-based sorbents in the treatment of oily wastewater is relevant not only economically, but also ecologically. About 3000 lakes in Lithuania are in the maturing and ageing phases. The shores of lakes are plumed by a dense plants' cultures, which spoils the landscape. This can be mitigated by harvesting the water plants, which can be used for absorbing petroleum and oil products, furthermore, after absorbing the oil. The stems of the plants can be easily disposed of by burning and used for energy production. Contrary to the Fibroil, which has to be burned in the safe manner and can present a risk for the environment.

4. Conclusions

A sustainable approach towards the environment has introduced low-cost, non-toxic and biodegradable materials for the removal and recovery of oil from water resources. The aim of the study was to investigate the feasibility of using Lithuanian naturally occurring, plant-based sorbents to remove organic matter from oily wastewater. Two plant-based and one synthetic commercial sorbent were examined to determine the oil sorption capacity and the potential of investigated sorbents being utilized for oil spill cleanup.

Use of these plants as a sorbents follows the principles of sustainable development: easily available, inexpensive, recyclable or easy to recover, and non-toxic. Sorption experiments were carried out under simulated conditions and the efficiency of the process was evaluated in terms of COD. Experimental results confirm that *Schoenoplectus lacustris* and *Acorus calamus* can be used as a low-cost natural sorbents for the removal of oil sorption. The sorption capacity of Fibroil, *Schoenoplectus lacustris* and *Acorus calamus* was 7 $g \cdot g^{-1}_{sorbent}$, 4.05 $g \cdot g^{-1}_{sorbent}$ and 1.39 $g \cdot g^{-1}_{sorbent}$, respectively.

The efficacy 89 %, 87 % and 37 % of COD removal was achieved during a down-flow filtration experiment with Fibroil, *Schoenoplectus lacustris* and *Acorus calamus* respectively. In the up-flow experiment Fibroil and *Schoenoplectus lacustris* demonstrated similar results - 96 % and 89 % respectively. In addition to minute efficacy differences, the disadvantages of Fibroil is that it's more expensive, non-biodegradable and not reusable nor recyclable. Due to its abundance and low cost the *Schoenoplectus lacustris* is likely to make a strong candidate as a sorbent for removing oil from water.

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