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Mats for Removing Technical Oil Contamination

Abstract
The aim of the investigation was to design and prepare a fibrous mat containing chicken feathers as an active filling to absorb spills of petroleum derivatives. The fibrous mat used to remove petroleum contamination consists of an active layer from chicken feathers (the main waste from poultry plants), placed between two layers of hydrophobic material of PP or PES in the form of nonwovens. The mata designed are characterised by excellent sorption of a wide range of hydrophobic petroleum substances, selectivity (sorption of liquid immiscible with water) and subdivision even at the highest saturation of the mat. Oil binder mats based on chicken feathers have been found to be highly effective in absorbing spilled oil from water surface. The material can be used, directly on a water surface regardless of weather conditions, as well as on other surfaces such as concrete or asphalt to fight leakages of oily substances. The mats are light, easy to store and use, and inexpensive. Apart from sorptive proprieties of the mats designed, the possibility of biological utilisation of petroleum contamination by means of a bioremediation method was introduced in the article.

Key words: *mat, chicken feathers, oil spills, bioremediation, sorption.*

Introduction

Environmental pollution of petroleum compounds is one of the most serious environmental problems. Neutralisation of leakage caused by improper storage during transport or oil processing is still a real problem [1, 2]. Penetration of hydrocarbons to soil, groundwater and water reservoirs forced a search for solutions to reduce drastic and long-term changes in ecosystems caused by the interaction of these compounds. [3]. Oil slick floating on the surface of a water reservoir blocks the access of light and also causes significant reduction in the oxygen concentration in the environment, which in turn leads to the death of flora and fauna living in the dirty water tank.

There are many different kinds of solutions for removing petroleum substances from water and soil, depending primarily on the environment in which the spill occurs. Among the different methods to combat oil spills on open water reservoirs (burning, melting, sorbents, dispersion and collection of the oil), the collection of oil from the surface of the water is the most advantageous method [4].

Technologies conducted using sorbents require highly efficient materials which capture hydrophobic compounds with a strength significantly higher than the one which associates hydrocarbons in soil or matrix and keeps them in an aqueous medium.

Further treatment of the sorbent and absorbed contamination can take place in different ways.

Synthetic sorbents are usually regenerated and the pollution is sent for incineration. In the case of natural sorbents, both sorbent and pollution are subjected to combustion. Proposed modification of the technology uses the opportunities provided by natural sorbents, which are placed in suitable, permeable material and formed into a mat or sleeves that can be put into the place where the leak occurred. After the uptake of the appropriate amount of contaminants the sorbent can be recovered and /or, if necessary, replaced with another. There are methods of removing spills of petroleum substances which use active materials in the form of pellets, obtained from natural minerals [5 - 10]. They are harmless to the environment, they quickly and effectively absorb petroleum substances, do not reflect the substance absorbed, are non-flammable and easy to use. The disposal of this material may be performed by burning in incinerators [11, 12].

It is much more difficult to remove oily, petroleum substances from the surface of water. The U.S. Patent describes materials based on cellulose [13]. These materials are both hydrophilic and oleophilic, and thus tend to absorb oil and water, which inhibits the degree of removal of the oil spills. Known mats are made of coal silicone granules placed in envelopes which are effective material for absorbing oil from the surface [14].

Furthermore, for removing oily substances the amorphous silica placed on an inert organic or inorganic support (clay, perlite, vermiculite, glass, crushed, volcanic ash, sand, peat, straw, sawdust, corn cobs and coconut) are used. The carriers may also be synthetic, including polyurethane, polyethylene or polypropylene [15 - 22].

The Textile Research Institute in Lodz launched a moulded polypropylene nonwovens produced by pneumotermal technique used for the removal of oil and petroleum substances from the surface of soil and water. PP microfibres with a high oil absorption are produced in the form of sheets (80 × 100 cm) or strips (width 80 cm), loose fibres, and also mats, cushions and dams devoted for use in environmental protection. The dangerous pollution of the environment caused by petroleum products forces inspiration to design innovative solutions in order to increase the efficiency of remediation the contaminated sites.Environmental pollution caused by spills of petroleum products is an incentive to design innovative solutions increasing the efficiency of remediation of contaminated sites [23 - 25].

One promising treatment technology modifying the environment is the use of active fibre mats filled with feathers, in which the negative phenomenon of permanent deposition of oily substance on the feathers of birds were used. Fulfilment of specific properties allows applying this material as a sorbent used for spills of petroleum products. However, despite the high sorption capacity of the product, the disposal of mat containing adsorbed contamination is still a problem. One solution is the use of bioremediation technology, which uses natural biological activity, in order to destroy and /or neutralize contaminants [26]. This technology does not require high costs or complex equipment, may be used in place of contamination and also is widely accepted in the society [27].

The authors, whose results are presented in this article have been trying to develop a new, so far unknown types of materials in the form of absorbing mats for collecting oil from the water surface, using waste material in the form of feathers. For many years non-biodegradable waste has been a nuisance to the environment [28, 29] due to its characteristics. It is a novel proposal to apply the material in the form of feathers to remove technical oil contamination from the environment. In addition, the study attempts to use biological methods of utilisation of technical oil contamination adsorbed on feathers contained in the sorption mats.

Scientific goal

The negative phenomenon of permanent deposition of oily substance on bird feathers which have been observed during ecological disasters, was used by the authors to develop a fibrous sorption mat to purify the water environment contaminated with petroleum pollutants. The aim of the study was to design a fibre mat for the removal of spills of oil technical impurities. The mat consists of an active layer of comminuted chicken feathers, placed between two layers of a hydrophobic material e.g. PP or PES in the form of nonwovens or woven fabrics. The efficiency of absorption of oily substances was evaluated. It was decided to assess the possibility of biological utilisation of oil contaminants deposited on the feathers.

Materials

1. White chicken feathers, from poultry slaughterhouse (Wróblew 51, province Lodz), after physico-mechanical processing (described in the section "**Pre-treatment of chicken feathers**") are characterised by the following:
   - sulphur content 2.9%
   - nitrogen content 15.5%
   - ash content approx. 1%
   - lipid content 15.8%
   - apparent density 3.5 × 10^{-2} g/cm^3.

2. Materials for mat preparation

For the purpose of the investigation hydrophobic materials from PP and PES were used

- PP nonwovens of surface mass 50 g/m^2, produced in IBWCh with melt-blown technique
- PP nonwovens of surface mass 100 g/m^2, produced in IBWCh with the melt-blown technique
- PES nonwovens Novitex of surface mass 50 g/m^2, produced with the hydrodynamic method by NOVITA S.A., Poland
- PES woven fabric, surface mass 50 g/m^2, produced by Fako S.A., Poland (commercial product).

The evaluation of selected physico-mechanical properties was made in IBWCH Metrological Laboratory according to the following standards:

- Mass per area PN-EN 12127:2000
- Thickness of the nonwovens and woven fabric PN-EN ISO 9073-2:2002
- Tensile strength PN-EN 29073-3:1994.

Method of manufacturing fibrous sorptive mats to remove technical oil contamination

Pre-treatment of chicken feathers

Waste chicken feathers from a slaughterhouse were pre-treated by washing 3 times with warm water at 40 °C in the presence of a surfactant for 1 hour. After filtering of the water, the feathers were treated with 15% of hydrogen peroxide (H_2O_2) and filtered. Wet feathers were cut into pieces of 4 mm length and dried.

For the evaluation of the appearance of cut feathers, scanning electron microscopy was used. Measurement of the thickness of cut feathers was based on the software analySIS Docu Soft Imaging System. The average thickness of feathers was 5.0 µm.

Preparation of mats

Samples of mats were prepared in the form of 10 × 10 cm envelopes. The mat used for removing technical oil contaminations (***Figure 2***) consists of an active layer made of fragmented, pre-treated chicken feathers, placed between two layers of hydrophobic polypropylene or polyester material in the form of nonwovens or woven fabric, size 10 × 10 cm. The envelopes were stitched, pierced along the length of parallel lines spaced at equal distances in order to maintain the stability of the position of the sorbent inside the envelope.

Methods

Determination of the theoretical sorbent absorption of feathers

Absorption of oil is permanently associated with the absorbent material relative to the unit weight. Durable bond with the

Table 1. *Parameters and tensile strength properties of materials used for the manufacture of the external layers.*

Parameter	Nonwovens of			Woven fabric PES
	PP fibre	PP fibre	PES Novitex fibre	
Mass per unit area, g/m^2	100	50	50	30
Thickness mm	0.56	0.38	0.39	0.32
Tensile strength (breaking force) in the longitudinal (machine)direction , N	41.6	59.0	-	198
Tensile strength (breaking force) in the cross direction , N	37.4	35.2	38.3	48.7

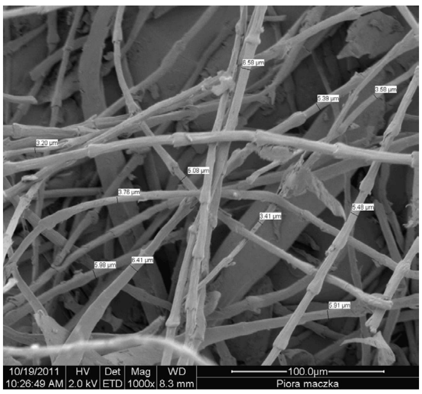

Figure 1. *SEM photo of chicken feathers after pre-treatment used for the preparation of mats.*

Figure 2. *Mat to remove technical oil contamination.*

sorbent is defined as the amount of oil which is removed from the water with the absorbent material. Oil, which has been around the sorbent agglomeration on water, and has not been removed from the water along with it, does not influence absorption of a sorbent due to the lack of binding interactions [31].

$$c = c_1/c_2 \text{ in g/g}$$

c_1 - amount of absorbed oil
c_2 - amount of sorbed.

Preparation of the mats
To determine the sorption capacity of the sorbent, in the form of feathers, mat samples were prepared with dimensions 10 × 10 cm, composed of different sorption layers of 5, 10 and 15 grams. Mat thickness was equal to, respectively: 1.1, 1.7, and 2.1 cm

Conducting the absorbency test
The samples prepared were placed in crystallizers of 500 ml containing 400 ml of demineralised water with the addition of 100 grams of oil. Test time was 30 min, after which the samples were removed from the bath, dried on filter paper at ambient conditions and the amount of the oil in g absorbed and retained by the active layer was assessed. For sorption studies, waste oil from vacuum pumps, with a density of d = 0.8725 g/cm^3, and a dynamic viscosity of 114 cP was used.

Bioremediation process of mats polluted with technical oil compounds
The tested material consisted of complex structures in the form of sorptive pillows containing chicken feathers contaminated with a mixture of hydrocarbons derived from: 1) waste oil from vacuum pumps, with a density of d = 0.8725 g/cm^3 and 2) a pure gas oil having a density d = 0.820 d/cm^3.

The bioremediation of the mat was carried out in a liquid medium, using selected strains.

Microorganisms for bioremediation
For the purpose of the investigation aerobic bacterial strains: *Gordonia alkanivorans S7, Pseudomonas sp. A34* (from the collection of the Institute of Technical Biochemistry, Lodz University of Technology) isolated from petroleum hydrocarbon-contaminated wastes in Poland was used. The bacterium was stored at 4 °C on LB agar plates coated with a film of sterile diesel oil hydrocarbons.

Conditions of biodegradation of contaminations absorbed on feathers
Biodegradation experiments of contaminated mats were performed in liquid medium “A” (yeast extract 2 g/l, glucose 2 g/l, NH_4Cl 2.5 g/l, $Na_2HPO_4^-$ 1.5 g/l per litre of tap water. pH of the culture was adjusted to 6.7 with HCl prior to autoclaving (121 ºC, 20 min). The biodegradation process was carried out in 500 ml flasks containing 60 ml of medium on a rotary shaker, at 30 °C, for 10 days. After autoclaving, one contaminated composite material (sorptive pillow engorged with chicken feathers) was added to each flask.

Analytical methods

Emulsification

Emulsifying activity of the culture liquid samples was determined by the method of Pearce and Kinsella (1978). To prepare emulsions, 1.0 ml of diesel oil and 3.0 ml of cell-free culture medium in 1 ml of 0.1 M phosphate buffer pH 7.0 were homogenised at 12000 R.C.F. for 1 min at 25 °C. Then 50 µl sample of the emulsion was taken from the bottom of the container and diluted in 5 ml of a 0.1% sodium dodecylsulphate solution. The absorbance of diluted emulsion was determined at 500 nm. The emulsifying activity was determined on the basis of the absorbance measured immediately after preparing the emulsion.

Assessment of hydrocarbons degradation

To determine the total degree of utilisation of hydrocarbons, complex sheets were subjected to two-step treatment, with the purpose of the extraction of the hydrocarbons from the feathers.

In the first stage samples were sonicated for 2 hours, after biodegradation, and then subjected to 2 h extraction with dichloromethane which was carried out in Soxhlet apparatus, using dichloromethane as the extracting factor. Resulting extracts were left until the solvent evaporated and the mass of extracted hydrocarbons was determined gravimetrically. The results were used to calculate the decrease in total hydrocarbons content caused by the microorganisms.

Gas chromatography (GC) analysis of hydrocarbons extracted from chicken feathers after bioremediation

Hydrocarbons extracted as described previously, were dissolved in 1 ml of hexane. Gas chromatography of hydrocarbons was performed using a Hewlett-Packard gas chromatograph (model 5980) equipped with a DB1 capillary column (30 m × 0.53 mm × 0.25 µm) and a flame-ionization detector (FID). Analysis conditions: the solvent - hexane, helium as a carrier gas, injector temperature of 300 °C, 1 µl injection volume, temperature program: 60 °C (increase by 4 °C in 1 min) 260 °C, FID temperature of 260 °C.

Analysis

All mathematical and statistical calculations were performed using the Statistica 10.0 program. One-way ANOVA was performed to compare the mean values of results of different treatments. When significant F values were obtained, differences between individual means and control mean were tested using Tukey’s test. The relationships between the intensity of carbohydrate degradation in the bioremediation processes (residual hydrocarbons content) and emulsification activity parameters were determined by *Pearson* correlation analysis with Statistica 10.0. Significance was set at $p = 0.05$.

Results and discussion

The aim of the present work was to develop and test a new kind of sorptive material for removing technical oil contaminations from the environment. The mat consists of an active layer made of disintegrated chicken feathers after pretreatment, placed between two layers of hydrophobic polypropylene or polyester nonwovens or woven fabrics. Results obtained in the present work allowed selecting the most suitable material to prepare mats in which chicken feathers were placed. The material should be a layer which separates two phases - water and oil.

One parameter that is crucial for the selection of sorbent is its absorbing capacity. A high value of this parameter allows using a small amount of the materials for the absorption of a given oil amount. This results in lower cost of spill liquidation and the technology itself becomes easier to implement.

Absorbing capacity of the sorbent (***Table 2,*** see page 104) is given as an average of 5 measurements for each mat variant.

*Table 2. Properties of sorptive mats; *- average value with significance.*

Type of material of the external layers	Feathers amount, g	Mass of the mat, g	Amount of absorbed oil, g	Amount of assembled oil, g	Absorption efficiency of the mat, dm^3/m^2	Absorptivity of the sorbent (chcicken feathers), g/g*
Novitex/Novitex	5	6.10	89	78	7	16 ± 0.2
Novitex/Novitex	10	11.15	87	83	7	8 ± 0.2
Novitex/Novitex	15	16.20	97	95	8	6 ± 0.2
PES/PES	5	5.70	85	66	6	13 ± 0.1
PES/PES	10	10.70	100	98	9	10 ± 0.2
PES/PES	15	15.80	100	99	9	7 ±0 .2
PES/Novitex	5	5.90	85	72	6	14 ± 0.1
PES/Novitex	10	10.80	93	92	8	9 ± 0.1
PES/Novitex	15	15.80	96	96	8	7 ± 0.2
PP/PP 50 g/m^2	5	5.95	78	70	6	14 ± 0.2
PP/PP 50 g/m^2	10	11.00	81	80	7	8 ± 0.2
PP/PP 50 g/m^2	15	16.00	71	71	6	5 ± 0.1
PES/PP 50 g/m^2	5	5.95	79	69	6	14 ± 0.1
PES/PP 50 g/m^2	10	10.95	92	90	8	9 ± 0.2
PES/PP 50 g/m^2	15	15.85	100	100	9	7 ± 0.2
PP/PP 100 g/m^2	5	7.15	67	57	5	11 ± 0.2
PP/PP 100 g/m^2	10	12.00	75	75	7	8 ± 0.1
PP/PP 100 g/m^2	15	17.00	74	74	6	5 ± 0.2
PES/PP 100 g/m^2	5	6.45	75	65	6	13 ± 0.1
PES/PP 100 g/m^2	10	11.35	84	83	7	8 ± 0.2
PES/PP 100 g/m^2	15	16.50	91	91	8	7 ± 0.2

Table 3. Emulsifying activity of bacterial strains during bioremediation of mats containing chicken feathers contaminated with oils; average value with significance at p = 0.05, and n = 3.

Sample	Emulsifying activity (OD_{500})				
	Day of bioremediation				Average activity, -
	3	5	7	10	
Pure chicken feathers	0.1 ± 0.033	0.12 ± 0.033	0.2 ± 0.088	0.22 ± 0.029	0.16 ± 0.029
Chicken feathers contaminated with waste oil from vacuum pomp	0.6 ± 0.150	1.2 ± 0.050	1.9 ± 0.010	1.8 ± 0.010	1.62 ± 0.230
Chicken feathers contaminated with diesel oil	0.5 ± 0.050	0.96 ± 0.030	1.4 ± 0.020	1.35 ± 0.075	1.23 ± 0.140

Table 4. Degree of total hydrocarbons uptake by Gordonia alkanivorans S7 and Pseudomonas sp A34 strains after 10-days of bioremediation of sorptive mat's insert; average value with significance at p = 0.05, and n = 3.

Sample	Mass of contaminated sorptive mat, g	Mass of contaminated chicken feathers, g	Mass of extracted contamination, g	Total hydrocarbons uptake, %
Chicken feathers contaminated with waste oil from vacuum pomp	64.8 64.5 65.0	59.8 59.4 59.9	32.8 33.0 32.5	48 ± 0.331 48 ± 0.320 49 ± 0.333
Chicken feathers contaminated with diesel oil	37.4 37.7 37.6	31.4 31.4 31.3	21.1 21.0 21.2	41 ± 0.305 41 ± 0.200 42 ± 0.088

All of the used mat's variants with the active impletion of chicken feathers has a high absorbing capacity in the range of 6 up to 9 dm^3/m^2. Commercially available products show an absorbing capacity in the range of 4 to 10 dcm^3/m^2. Absorbing capacity of chicken feathers, shown as the ratio of the amount of assembled oil to the amount of sorbent oscillate between 5 to 14 g/g. It was noticed that thickness of the active layer has a significant influence on absorption effectiveness. The mats of the smallest thickness demonstrated the highest absorbing capacity for oil substances (11- 16 g/g).

The mat with the active impletion of chicken feathers featured great sorption of a wide range of hydrophobic, petroleum compounds, selectivity (sorption of water immiscible liquid) and was unsinkable even in a state of the highest satiation of the mat (which was checked in laboratory conditions).

It must be remembered that petroleum substances removed from the environment, absorbed in the mat, preserve their dangerous properties, and therefore they must be utilized.

One of the cheapest, ecological and generally publicly acceptable methods of utilizing technical oil contaminations is bioremediation.

The effectiveness of the process depends on different parameters. One of them is bioavailability of the contamination for microorganisms which carry out the process of degradation. Bioremediation processes abilities are completed naturally by microbial systems, accelerated through suitable enhancers, such as biosurfactants and represented as bioavailability [26] Biosurfactants produced by microorganisms may enhance the contact between the hydrophobic substrate enclosed in the sorbent and bacteria during the biodegradation process. In this context, a very important parameter, which characterizes the ability of microorganisms for hydrocarbon degradation, is emulsifying activity. It defines the ability of bacteria for emulsifying hydrophobic substrate in the presence of biosurfactants produced by microbial cells.

Changes in emulsifying activity of bacterial strains during the bioremediation of the chicken feathers contaminated with petroleum hydrocarbons are shown in ***Table 3***. Values of the parameter were relatively high during all the processes. The highest emulsifying activity – OD_{500} 1.9 ± 0.010 was obtained on the 7^{th} day of biodegradation for samples where bioremediation of chicken feathers contaminated with petroleum hydrocarbons from vacuum pump was performed. Also, relatively high values of the parameter were obtained for sheets contaminated with pure diesel oil. Confirmation of these results in a strong positive Pearson correlation on the 10^{th} day of bioremediation – 0.989. Such a high value of the coefficient suggests that there is a correlation between the rate of petroleum hydrocarbons consumption by microorganisms and the increased amount of biosurfactants produced by bacterial strains.

During remediation the most important parameter is the evaluation of the process efficiency measured by the decrease in the amount of contamination from the

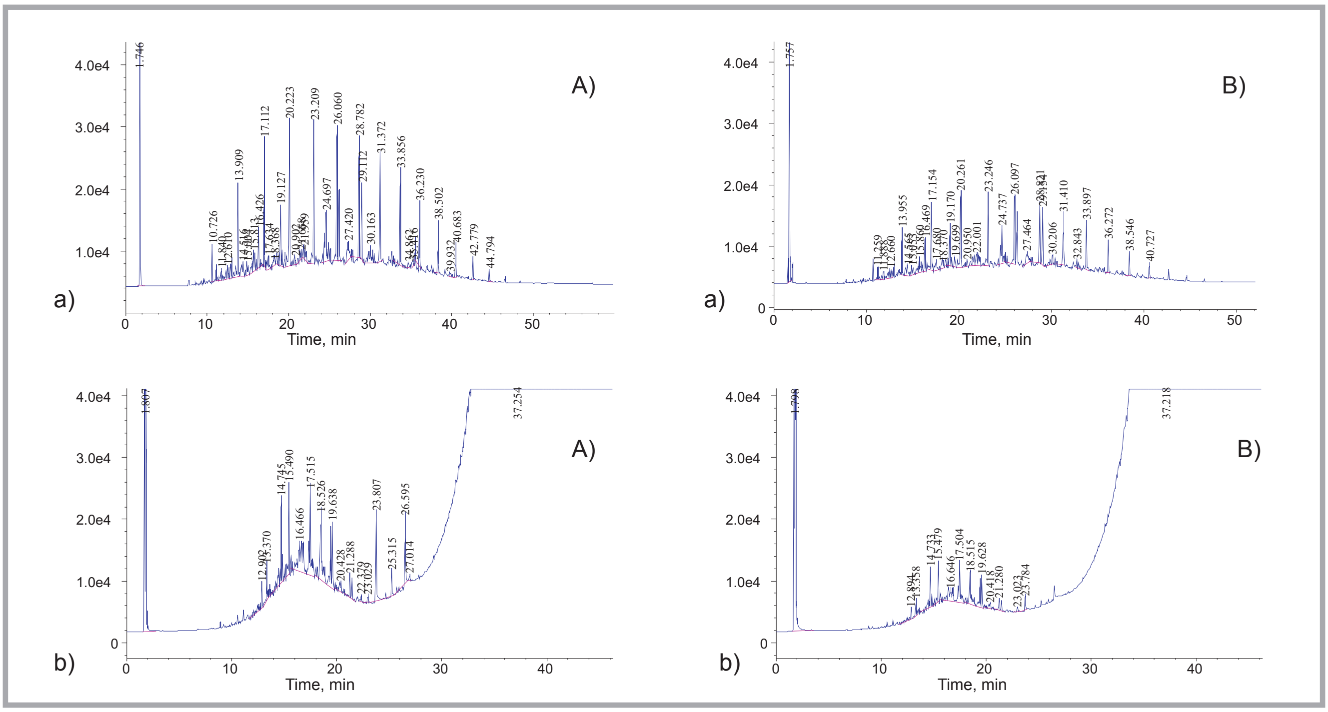

***Figure 3.** GC profile of hydrocarbons and their intermediate metabolites extracted from chicken feathers contaminated with: a) pure diesel oil and b) waste oil from a vacuum pump (A) before and (B) after 10 days of bioremediation with G. alkanivorans S7 and Pseudomonas sp. A34 bacterial strains.*

environment. The treatment of contaminated chicken feathers with *Gordonia alkanivorans S7* and *Pseudomonas sp A34* strain caused a relatively fast (within 10 days) decrease in the content of hydrocarbons (***Table 4***). High efficiency of the degradation – 40 - 50% obtained only after 10 days of biodegradation indicates that biological purification of the composite sheets containing chicken feathers contaminated with petroleum hydrocarbons is possible.

The results obtained after GC analysis (***Figures 3.aA*** and ***3.bB***) showed that the number and retention time of peaks, that are equivalent for each hydrocarbon and their intermediate metabolites in samples where the bioremediation process with bacterial strains was performed were about 40 up to 50% lower than for non-treated samples. Furthermore, relatively high efficiency of the oil contaminations utilization obtained only after 10 days of biodegradation process and high emulsification of the contamination suggest that the higher efficiency of the mat's remediation may be obtained by extending the time of bioremediation. The results obtained in the present work confirmed that using biological methods for the treatment of composite sheets containing chicken feathers contaminated with petroleum hydrocarbons or pure diesel oil with bacterial consortia consisting of *Gordonia alkanivorans S7 and Pseudomonas sp. A34* enables their relatively quick and cheap regeneration, although an economic calculation has not been made. Taking into consideration the availability and difficulty in the waste management of chicken feathers and the possibility of their reuse, the costs of producing the mat should be competitive for sorbents that already exist.

Conclusions

1. Sorptive mats with active impletion in the form of feathers feature excellent sorption of hydrophobic petroleum substances, selectivity (sorption of liquid immiscible with water) and subdivision even at the highest saturation of the mat. The absorbing capacity of feathers is defined as the amount of collected oil in relation to the amount of the sorbent and ranges from 5 to 16 g/g.
2. The thickness of the active layer is crucial for absorption efficiency. Sorptive mats with the lowest thickness feature the highest absorbing capacity of 11-16 g/g.
3. The use of bacterial consortia (Gordonia alkanivorans S7 and Pseudomonas sp. A34 strains) caused relatively fast (in 10 days of biodegradation) 40 – 50% utilisation of hydrocarbons from contaminated material.
4. High values of emulsifying activity (the highest value of the parameter OD_{500} 1.9 ± 0.010 was obtained for the sample with feathers contaminated with technical oil, on the 7th day of the process), indicate that bacterial species used for the biodegradation processes have the ability for biosurfactant production. This causes an increase in bioavailability of the contamination and in consequence leads to high utilisation efficiency (Pearson's factor 0.989).
5. The results obtained in the present work show that biological methods can be useful in the future for utilizing mats contaminated with petroleum substances.

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