

USING SUNFLOWER HUSKS FOR THE REMEDIATION OF OIL-CONTAMINATED SOILS

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Oil contamination is a global environmental problem due to its serious consequences for the environment and humans. Crude oil and petroleum products negatively affect the morphological, physicochemical, and biological properties of the soil, leading to the withdrawal of large areas of land from agricultural use as a result of reduced or complete loss of their productivity.

To improve the properties of oil-contaminated soils, localize the contaminants, and stimulate natural remediation processes, it is advisable to use various sorbents ameliorants – materials capable of absorbing large amounts of petroleum products, thereby preventing their further migration. At the same time, these materials improve soil properties by loosening it, optimizing gas exchange, acting as a source of important microelements, a matrix for the growth of oil-degrading microorganisms, etc.

The article investigates the sorption capacity of sunflower husks – an oil production waste – for oil and water and justifies the possibility of their use for the remediation of oil-contaminated soils. The physicochemical properties of sunflower husks were determined, and its positive effect on the water and air regimes of the soil is shown. The water absorption capacity of sunflower husks was 0.97 g/g, oil absorption was 0.78 g/g, and the sorption capacity for oil emulsion ranged from 1.19 to 1.26 g/g, depending on the oil content in emulsion. Due to the ability of sunflower husks to retain moisture and air within their structure, its use improves the water permeability and aeration of oil-contaminated soil, preventing it from drying out or waterlogging, reducing soil bulk density, and increasing soil porosity. The influence of sunflower husks on enhancing the efficiency of phytoremediation of oil-contaminated soils (5 wt.% oil in soil) using plants: *Secale cereal*, *Avena sativa*, *Sorghum saccharatum*, *Panicum virgatum*, *Zea mays*, *Linum usitatissimum* and *Melilotus officinalis* was investigated. It is shown that the addition of sunflower husks creates favorable conditions for plant growth and development, as evidenced by increased morphometric parameters and biomass accumulation. The obtained results show that addition of sunflower husks into oil-contaminated soil localizes oil pollution, loosens the soil, increases moisture retention capacity, reduces toxicity, thereby creating optimal conditions for the life of plants and microorganisms. Being an inexpensive, easily accessible, and environmentally friendly organic material, sunflower husks can be used in technologies for the remediation of oil-contaminated soils. *Key words*: sunflower husks, oil-contaminated soils, oil sorption, phytoremediation.

Використання лушпиння соняшника для відновлення нафтозабруднених ґрунтів. Шевчик-Костюк Л.З., Романюк О.І., Романюк Г.В., Борецька І.Ю.

Забруднення нафтою є глобальною екологічною проблемою через серйозні наслідки для довкілля та людини. Нафта та нафтопродукти негативно впливають на морфологічні, фізико-хімічні і біологічні властивості ґрунту, призводячи до виведення великої кількості земель з сільськогосподарського вжитку через зниження або повну втрату їх продуктивності.

Для покращення властивостей нафтозабрудненого ґрунту, локалізації забруднювача, активізації процесів природного відновлення доцільно використовувати різноманітні сорбенти-меліоранти – матеріали, які здатні вбирати у великих кількостях нафтопродукти, тим самим запобігаючи їх подальшій міграції, та одночасно покращувати властивості ґрунту, розпушуючи його, оптимізуючи газообмін, виступаючи джерелом важливих мікроелементів, матрицею для зростання мікроорганізмів-деструкторів нафти та ін.

У статті досліджено сорбційну здатність лушпиння соняшника – відходів олійного виробництва – щодо нафти та води і обґрунтовано можливість його використання для відновлення нафтозабруднених ґрунтів. Визначено фізико-хімічні властивості лушпиння соняшника і показано його позитивний вплив на водний та повітряний режими ґрунту. Водопоглинання лушпиння становить 0,97 г/г, нафтопоглинання – 0,78 г/г, сорбційна ємність по нафтовій емульсії становить 1,19-1,26 г/г, в залежності від вмісту нафти в емульсії. Завдяки здатності лушпиння соняшника утримувати вологу та повітря (в об'ємі), його використання покращує водопроникність та аерацію нафтозабрудненого ґрунту, запобігаючи його пересушуванню чи перезволоженню, зменшуючи щільність та збільшуючи пористість ґрунту. Досліджено вплив лушпиння соняшника на підвищення ефективності фітореMediaції ґрунтів, забруднених нафтою (5% нафти у ґрунті) за участі рослин: жита посівного (*Secale cereale*), вівса посівного (*Avena sativa*), сорго цукрового (*Sorghum saccharatum*), проса лозоподібного (*Panicum virgatum*), кукурудзи звичайної (*Zea mays*), льону звичайного (*Linum usitatissimum*), буркуну лікарського (*Melilotus officinalis*). Показано, що додавання лушпиння соняшника у нафтозабруднений ґрунт створює сприятливі умови для росту та розвитку рослин, що проявляється у зростанні їхніх морфо-фізіологічних параметрів та накопиченню біомаси. Отримані результати свідчать, що внесення лушпиння соняшника у нафтозабруднений ґрунт локалізує нафтове забруднення, розпушує ґрунт, підвищує вологоутримувальну здатність, зменшує токсичність, чим створює оптимальні умови для росту рослин та біодеградації нафти у ґрунті. Будучи недорогим, легкодоступним і екологічно безпечним органічним матеріалом, лушпиння соняшника може використовуватись у технологіях відновлення нафтозабруднених ґрунтів. *Ключові слова*: лушпиння соняшника, нафтозабруднений ґрунт, сорбція нафти, фітореMediaція.



Problem statement. The main environmental problem in regions with a developed oil and gas industry is the contamination of environmental components with oil and petroleum products. Accidental spills of oil and petroleum products occur during the extraction, transportation, processing, storage, consumption and disposal of products, and cause significant damage to ecosystems, up to environmental disasters, and lead to negative economic and social consequences. Oil is a complex mixture of organic substances consisting of a large amount of hydrocarbons (n-alkanes, polycyclic aromatic hydrocarbons, etc.), a small amount of non-hydrocarbon compounds (sulfides, nitrides, alkanolic acids, etc.), organic compounds of metals (vanadium, nickel, etc.), and microelements [1, 2]. When entering the soil, oil causes significant negative and sometimes irreversible changes: viscosity and density of the soil mass increase, content of heavy metals and carcinogenic polycyclic aromatic hydrocarbons, resins, and asphaltene increases, porosity, water permeability and moisture capacity of the soil decrease, nitrification capacity and activity of soil enzymes decrease, number and diversity of soil microorganisms decrease, the redox potential, carbon-nitrogen balance change [3]. These changes inhibit the growth and development of plants, lead to soil degradation and the withdrawal of such territories from agricultural use. Oil and petroleum products bind to soil components and decompose slowly and with difficulty, creating conditions for secondary contamination of atmospheric air, surface water and groundwater.

Relevance of the research. Today, many methods for the remediation of oil-contaminated soils have been developed. Phytoremediation is considered the most environmentally friendly approach. Research is being conducted to increase the efficiency of oil components biodegradation of in the process of phytoremediation and additional physicochemical extraction with the participation of remediation agents, in particular sorbents – substances capable of absorbing large quantities of oil products, thereby preventing their further migration. In addition, most sorbents function as soil amendments – they improve soil properties, loosen the soil structure, optimize gas exchange, act as a source of essential microelements, a matrix for the growth of hydrocarbon-degrading microorganisms, etc. Porous materials are mainly used as sorbents, including peat, ash, coke, silica gels, aluminogels, as well as various industrial and agricultural wastes: oilseed meal, sawdust, husks, bran, straw [4, 5].

Natural sorbents attract special attention due to their wide distribution in nature, low cost and ease of use. The combination of these advantages with rather high sorption properties makes them promising raw materials for soil remediation from pollution [5-8].

Analysis of research and publications. Sunflower is an important agricultural crop, the cultivated area of which has been increasing annually. During sunflower oil production, significant amounts of a by-product are

formed – sunflower husk – a hard, woody, multilayered formation, homogeneous in physical structure, with a stable chemical composition and physical and mechanical properties. Sunflower husk consists mainly of cellulose, lignin, hemicellulose and contains small amounts of proteins, fats and minerals [9]. It has high porosity, organic composition, and the ability to retain moisture, which makes it promising in terms of soil purification. However, information on the use of sunflower husk for the remediation of contaminated soils is poorly researched. Only the effect of sunflower husks combined with bird droppings for the decomposition of petroleum hydrocarbons in soil [10], the use of husks with immobilized microorganisms [11], and the application of modified sunflower husk-derived biochar as a sorbent for petroleum product removal [12] are reported. However, there is no information on the use of untreated sunflower husks (sunflower oil production waste) in phytoremediation technologies for the remediation of oil-contaminated soils.

The aim of the study was to establish the sorption capacity of sunflower husks for oil and water and its suitability for the remediation of oil-contaminated soils, in particular for enhancing the efficiency of phytoremediation.

Materials and Methods. Sunflower husks, a by-product of oil production, were used for the study. Bulk density was determined by weighing the husks in a measuring cup. The moisture content of the husk was determined by the gravimetric method – by drying the samples to constant mass in a drying oven at a temperature of 105 °C.

To determine water absorption capacity, 10 g of sunflower husks were soaked in water for 20 days at a temperature of 20 °C, until completely saturated with water. After saturation, the husks were removed from the water, shaken on a sieve, repeatedly blotted with filter paper, and weighed. Water absorption capacity was determined as the ratio of the mass of absorbed water to the mass of sorbent.

To determine the sorption capacity of sunflower husks for oil / oil emulsion (oil absorption capacity), similar actions were performed with the participation of husks and oil / oil emulsion. The oil emulsion was prepared by mixing water and crude oil to obtain oil contents of 2.5 wt.%, 5 wt.%, 10 wt.%. The husks were soaked in oil / oil emulsion for 20 days at a temperature of 20 °C. The experiments were conducted using oil from the Borislav oil field, with a density of 0.826 g/ml.

To investigate the effect of sunflower husks on enhancing the efficiency of phytoremediation of oil-contaminated soils, soil was artificially contaminated with oil at a concentration of 5%, sunflower husks were added, and the soil was planted with seeds of rye (*Secale cereale*), oat (*Avena sativa*), sugar sorghum (*Sorghum saccharatum*), switchgrass (*Panicum virgatum*), corn (*Zea mays*), flax (*Linum usitatissimum*), and sweet clover (*Melilotus officinalis*).

Morphometric parameters of plants were measured on the 45th day of the experiment using generally

accepted methods [13]. The content of photosynthetic pigments in plant leaves was determined spectrophotometrically [14]. Soil phytotoxicity was assessed using plant test objects: *Linum usitatissimum* L., *Helianthus annuus* L., *Fagopyrum vulgare* St. [15]. The bulk density of undisturbed soil was determined using the cutting ring method [16]. The particle density of the soil was determined by the pycnometric method [17]. Based on the data obtained, the soil porosity was calculated.

All experiments were performed at least three times. The results of the studies were statistically processed using Microsoft Excel.

Results and Discussion. The extent of oil degradation in soil depends on many factors. The efficiency of biodegradation of contaminants and the rate of soil remediation are influenced by oxygen availability, moisture content, temperature, nutrient content, soil structure and aeration. Unprocessed sunflower husks, when applied to the soil, can act as a natural structure-forming agent and improver of physical properties.

Some physicochemical properties of sunflower husks were determined. It was found that sunflower husks have a size of 9×5 mm, moisture content ranges from 9 to 10 %, and bulk density is 0.12 g/cm³ (Table 1). The geometric shape of a sunflower husk is an elongated hemisphere, the volume of which contains air (Fig. 1). Due to its geometric shape and fairly dense structure, the husk creates additional pore space in oil-contaminated soil, increasing

the porosity of the contaminated soil and preventing soil particles from sticking together. This creates favorable conditions for improving the water and air regimes of the soil, which are important for plants, aerobic oil-degrading microorganisms, and other living organisms.

Under natural conditions, when oil enters the soil environment, it mixes with water, forming oil emulsions that penetrate the pores of the soil covering its particles with a hydrophobic film. The hydrophobicity of oil-contaminated soils makes them overmoistened or overdried, and the sticking together of soil particles leads to its re-compaction. Sunflower husk is able to retain a certain amount of sorbed moisture and air (by volume), thereby regulating the water-air regime of the soil, preventing overdrying or overmoistening, and improving water permeability and aeration (Fig. 1). It was established that the water absorption capacity of the husk is 0.97 g/g, oil absorption capacity is 0.78 g/g, and the sorption capacity of sunflower husks for oil emulsion is 1.19-1.26 g/g, depending on the oil content in the emulsion. It is worth noting that the sorption capacity of sunflower husks for oil emulsion is higher than the sorption capacity for oil and water (Table 1). This can be explained by the fact that oil emulsion has a lower viscosity than pure oil. When the husk comes into contact with the emulsion, water penetrates the pores, causing the husk to swell, thereby increasing the sorption surface area and facilitating the access of oil droplets to the internal pores. That is, water

Table 1

Physicochemical properties of sunflower husk

Parameter	Value
Husk length, mm	9±1
Width, mm	5±1
Humidity, %	9,81±0,04
Bulk density, g/cm ³	0,12±0,02
Water capacity, g/g	0,97 ±0,03
Oil capacity, g/g	0,78±0,02
Sorption capacity for oil emulsion, g/g	
2.5 % oil in emulsion	1,26 ±0,02
5 % oil in emulsion	1,24±0,02
10 % oil in emulsion	1,19±0,01

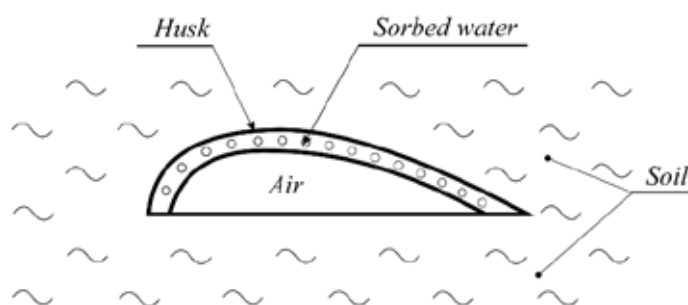


Fig. 1. Sunflower husk in moist soil

acts as a transporter that promotes deeper penetration of oil particles into the structure of the sorbent. In addition, the sunflower husk has both hydrophobic (in lignin) and hydrophilic areas (in cellulose and hemicellulose), and therefore is able to simultaneously retain both phases of the emulsion: the aqueous phase through hydrogen bonds, the oil phase through hydrophobic interactions, which increases the overall sorption capacity.

The data obtained show that sunflower husks are able to localize oil pollution and improve the properties of contaminated soil, thereby intensifying the processes of oil biodegradation by creating optimal conditions for the life of plants and microorganisms. Adding sunflower husks to oil-contaminated soil reduces its bulk density and increases total porosity (Table 2).

To confirm the above, we investigated the effect of sunflower husks on enhancing the efficiency of phytoremediation of oil-contaminated soils (5 wt.% oil) using plants: *S. cereale*, *A. sativa*, *S. saccharatum*, *P. virgatum*, *Z. mays*, *L. usitatissimum*, *M. officinalis*.

The different level of resistance of the studied plants to oil exposure were observed, as well as a variable effect of sunflower husks on plant growth in oil-contaminated soil (Fig. 2).

Corn is relatively resistant to oil contamination, and the addition of sunflower husks to contaminated

soil does not stimulate its growth parameters. For corn and flax, a decrease in root length of 19.7 and 3.6 %, respectively, was observed compared to oil-contaminated soil without husks. In the variant with sowing oats, a positive effect of sunflower husks on root length and shoot height was observed. The best results were obtained for switchgrass, sorghum, rye and sweet clover. For switchgrass and sorghum, relative shoot height was 28.11 and 35.95 % higher, respectively, compared to the shoot height of plants grown in oil-contaminated soil without husks, and also 19.83 and 16.07 % higher than the shoot height of plants grown in soil two years after oil contamination. Adding sunflower husks to oil-contaminated soil increased the resistance of sweet clover to oil contamination and enhanced its growth parameters. The relative root length and relative shoot height of sweet clover plants grown in oil-contaminated soil with the addition of sunflower husks were 5.26 and 10.02 % higher, respectively, than those of plants grown in oil-contaminated soil two years after contamination (Fig. 2, Fig. 4).

Adding sunflower husks to oil-contaminated soil resulted in increased biomass of phytoremediation plants, as indicated by higher fresh and dry plant mass compared to plants grown in oil-contaminated soil without sunflower husks (Fig. 3).

Table 2

Bulk density and total porosity of oil-contaminated soil

Options	Bulk density, g/cm ³	Total porosity, %	Moisture content, %
Control	1,23	46,2	24,1
Oil-contaminated soil (5% oil in soil)	1,34	38,5	26,3
Oil-contaminated soil (5% oil in soil)+sunflower husks	1,11	49,28	26,4

Note: Control – uncontaminated soil without oil and without sunflower husks

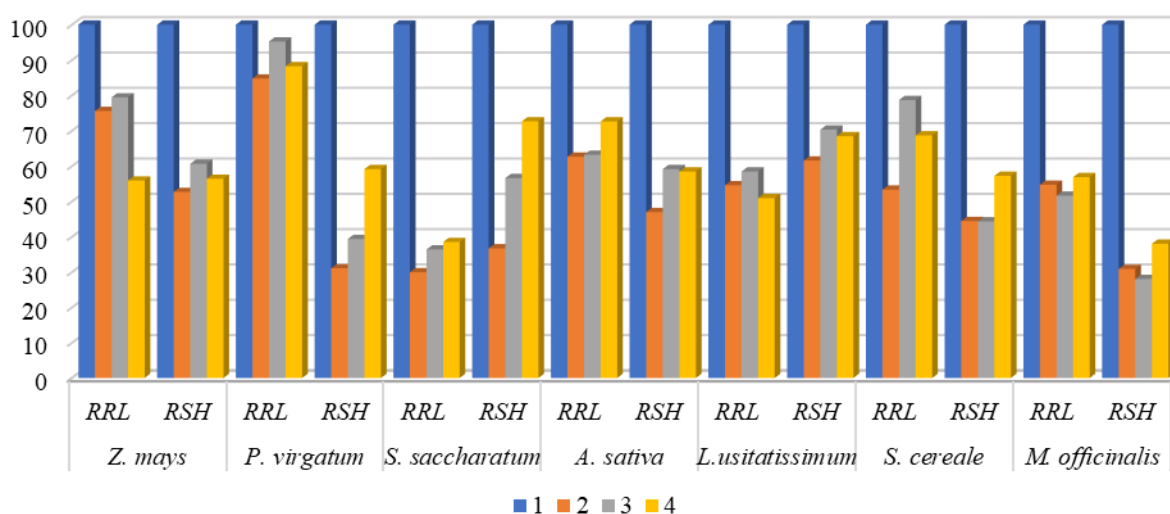


Fig. 2. Relative root length (RRL) and relative shoot height (RSH) of phytoremediation plants when grown in oil-contaminated soil: 1 – control; 2 – oil-contaminated soil (5 %, fresh contamination); 3 – oil-contaminated soil (5 %, two years after contamination), 4 – oil-contaminated soil (5 %) + sunflower husks

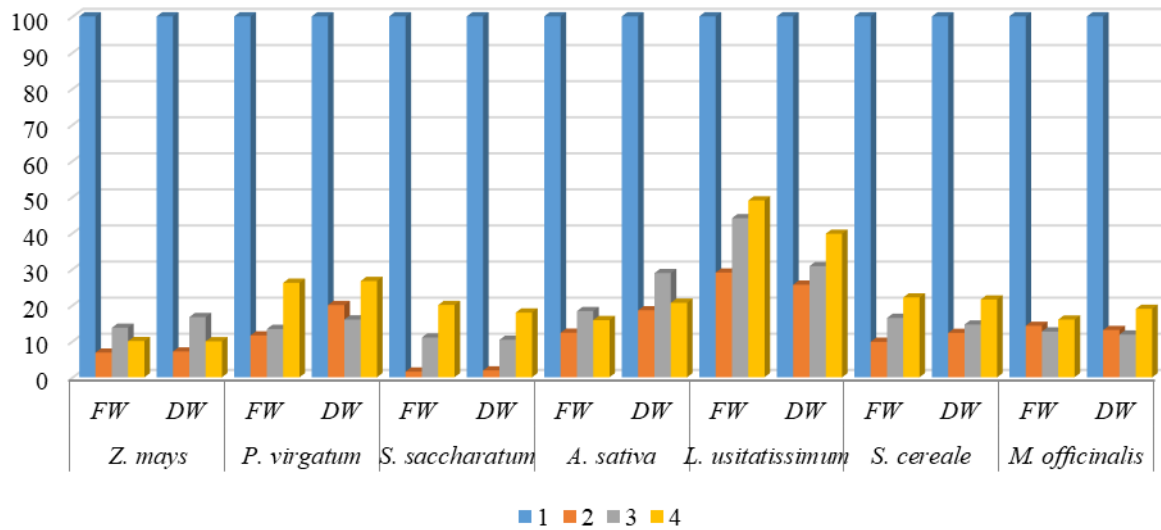


Fig. 3. Relative fresh weight (FW) and relative dry weight (DW) of phyto remediation plants grown in oil-contaminated soil: 1 – control; 2 – oil-contaminated soil (5 %, fresh contamination); 3 – oil-contaminated soil (5 %, two years after contamination), 4 – oil-contaminated soil (5 %) + sunflower husks



Fig. 4. Plants of *M. officinalis* (A) and *P. virgatum* (B) grown in oil-contaminated soil: 1 – control; 2 – oil-contaminated soil (5 %, fresh contamination); 3 – oil-contaminated soil (5 %, two years after contamination), 4 – oil-contaminated soil (5 %) + sunflower husks

The indirect effect of oil contamination on plants, caused by changes in the physicochemical properties of the soil, leads to disturbances in root nutrition and, consequently, to the inhibition of photosynthetic activity. This inhibition is associated with the alteration in the content of photosynthetic pigment, primarily chlorophylls and carotenoids. Under conditions of oil contamination, the content of chlorophylls *a* and *b* and carotenoids decreases compared to the control. Adding sunflower husks to oil-contaminated soil increases plant resistance to stress. The content of chlorophyll *a* and *b* and carotenoids in the leaves of sweet clover and switchgrass grown in oil-contaminated soil with the addition of sunflower husks were higher than those in plants grown in oil-contaminated soil without sunflower husks (Table 3).

The phytotoxicity of soils after phytoremediation with and without the addition of sunflower husks was determined using value of effective toxicity. Sweet clover plants were found to have a positive effect on oil-contaminated soil, resulting in a reduction of soil phytotoxicity when applied alone. However, an even greater decrease in phytotoxicity of oil-contaminated

soil was observed when sunflower husks were used in the phytoremediation (Table 4).

Therefore, sunflower husks enhance the phytoremediation of oil-contaminated soils by improving soil properties, including soil structure, moisture retention, reducing soil toxicity, and by creating optimal conditions for oil rhizodegradation. Being inexpensive, readily available, and environmentally friendly, sunflower husks can be used to remediation of oil-contaminated soils.

Conclusions. The sorption capacity of oil production waste, sunflower husks, was determined, and their potential application for the remediation of oil-contaminated soils was demonstrated. The water absorption capacity of sunflower husks was 0.97 g/g, oil absorption was 0.78 g/g, and the sorption capacity for oil emulsion ranged from 1.19 to 1.26 g/g, depending on the oil content in emulsion. Due to their ability to retain moisture and air within their structure, sunflower husks contribute to improving the physico-chemical properties of contaminated soil, thereby intensifying oil biodegradation processes. Sunflower husks are a natural, environmentally safe and economically viable sorption

Table 3

Content of photosynthetic pigments in the leaves of *M. officinalis* and *P. virgatum* grown in oil-contaminated soil

Options	Pigment content, µg/g			
	Chlorophyll <i>a</i>	Chlorophyll <i>b</i>	Chlorophyll <i>a+b</i>	Carotenoids
<i>M. officinalis</i>				
Control	2,331	1,375	3,705	0,540
Oil-contaminated soil (5 %, fresh contamination)	1,32	1,06	2,38	0,429
Oil-contaminated soil (5 %, two years after contamination)	1,43	0,975	2,405	0,484
Oil-contaminated soil (5 %) + sunflower husks	1,762	0,942	2,662	0,458
<i>P. virgatum</i>				
Control	0,549	0,236	0,785	0,107
Oil-contaminated soil (5 %, fresh contamination)	0,248	0,156	0,404	0,057
Oil-contaminated soil (5 %, two years after contamination)	0,226	0,100	0,325	0,051
Oil-contaminated soil (5 %) + sunflower husks	0,344	0,188	0,533	0,062

Table 4

Effect of *M. officinalis* on phytotoxicity of oil-contaminated soil

Test objects	Phytotoxicity		
	Oil-contaminated soil (5 %)	Oil-contaminated soil (5 %, two years after contamination)	Oil-contaminated soil (5 %) + sunflower husks
<i>L. usitatissimum</i>	0,523	0,210	0,270
<i>H. annuus</i>	1,403	0,480	0,610
<i>F. vulgare</i>	0,918	0,914	0,666

material and soil-structuring agent. Their inclusion in phytoremediation schemes improves the condition of plants and, consequently, the microbiological activity of the soil, and thus they can be used to remediation of oil-contaminated soils and for enhancing the efficiency of phytoremediation.

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