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THE ANALYSIS OF SOILS POLLUTION WITH PETROCHEMICALS BY THE MEANS OF ELECTROMETRIC METHOD

Introduction

Pollution of the biosphere components with crude oil and its derivatives are among the major environmental problems of the modern world. Annual global demand and production of petrochemicals grows by 3.5 % a year [1, p. 2]. But during extraction, transportation, processing and use of oil and oil products, their losses amount to about 10–20 million tons per year [2, p. 66]. The main causes of the losses are oilfield accidents, oil pipeline breaks during transportation, overloading and storage losses. Despite the reason, all the petrochemicals lost in operations become pollutants, causing degradation of soil, atmosphere and water quality. In this problem soil is often an intermediate between the source of pollution and its further advancement to adjoining media – air and water. For this reason, measuring soil pollution with petrochemicals is important for prevention of both soil destruction and pollution of atmosphere and hydrosphere. However, despite the fact that petroleum products are one of the main soil pollutants, approaches to detecting and analyzing their content in the respective environments are diverse but imperfect. Therefore, the purpose of the work is to comparatively analyze methods for studying petroleum contamination of soils and to improve the principles and to formulate a method of conductometric determination of the concentration of petroleum products in the soil.

Problem statement

Soil, as a biosystem, is important ecologically, both for the functioning of individual terrestrial ecosystems and for the existence of the biosphere as a whole, due to the diversity of its ecological functions. Although the most valuable function of soils is to provide the growth and development of plants, they also play another, less well known, but equally important global role. In the soil of this Earth's cover and its humus, the major part of the living substance of land and its biogenic energy is concentrated in [3]. Therefore, the soil-organisms ecological system is one of the most important elements for ensuring stability and productivity of the entire biosphere, together with the maintenance of a substances circulation.

Analysis of the latest research and publications

The modern trends of urban development state the need to modernization of municipal areas in the sustainable mode. This includes relocation the industrial facilities to the zones outside the areas of compact human residence. At the same time the major source of negative impacts on the urban environment are transport and its service infrastructure. Since the population is growing the number of private and public transport vehicles increases correspondingly contributing to environment pollution with fuels and lubricants.

While air pollution due to emissions of cars is widely covered in research works, the urban soils pollution at the areas adjoined to fuel retailing facilities and storage sites still lacks detailed studies.

Being the most common environment pollution associated with transport, petrochemicals represent a puzzling problem, as the chemical composition and thus the hazard levels imposed by each fuel spilled is different, depending on origin, method of production and storage period [4].

Petroleum is a mixture of more than 450 different substances, mainly hydrocarbons with different molecular weights, different properties and environment consequences. The main elements in its composition are C — 83–87 %, N — 12–14 %, N, S, O — 1–2 %, rarely 3–6 % at the cost of sulfur. The tenth and hundredth percentages of a percent of oil are numerous trace elements [5]. Oils of various origins also contain about 30 types of heavy metals, including V — 10^{-3}–10^{-2} %; Ni — 10^{-3}–10^{-2} %; Fe — 10^{-4}–10^{-3} %; Zn — 10^{-5}–10^{-3} %; Hg — about 10^{-5} %; Na, K, Ca, Mg — 10^{-3}–10^{-4} %. The total content of the metals in oil is on average 0.04 % [6]. Combined effect of diverse organic components of petrochemicals, some of which belong to POPs and highly toxic agents, and heavy metals depress soil community, reduce the overall fertility and contribute to salination of soils [6–8].

Thus, the soil and inhabited by billions of organisms, the processes of exchange are so diverse and complex that we have yet to come to their understanding. Therefore, any soil contamination is a threat to the relevant environmental functions of the soil and requires detailed description and analysis. In this regard, it is very important to develop new methods of efficient and in-time testing petroleum products in soils.

Electric properties of the system “soil-petrochemicals”

Given the variability of the chemical composition of oil, at present there are no standardized methodological approaches to the assessment of soil pollution by petroleum products, which is due to the lack of unified diagnostic criteria. The most common methods now are gravimetric method, fluorimetric method, UV spectrophotometric method, IR spectroscopy, GC gas chromatography or channel thin layer chromatography. All of them possess certain advantages, limited by a range of disadvantages: highly sensitive methods are normally instrumentally complicated, while simpler techniques are of low accuracy.

Another important issue is the problem of environmental regulation of petrochemicals content in soils, in fact, there is no legal framework for acceptable levels in the form of MPC or background concentration of petrochemicals in soil. Under such conditions there is a need to develop new reliable and express methods based on specific properties of both soils and petrochemicals. Here it is offered to use physical properties of the system “soil-petrochemicals”. Petroleum and petroleum products are dielectrics and are characterized by extremely high electrical resistance. For example, for paraffin it ranges from 2 to $0.3 \cdot 10^{8}$ Ohm·m [9]. Some of them are used in electrical engineering and radio engineering as insulating material (paraffin) or insulating medium (transformer oils) in transformers, oil rheostats and switches.

In contrast, soils are electric current conductors, ranging from clay soils, with lower resistance due to finer particles and greater points of contact between them, to sandy soils, with coarser particles and therefore with smaller points of contact between them. Thus, soil texture affects the soil conductivity, as well as content of soil organic matter, moisture and salt. Therefore, soils electrical conductivity, measured using electrical resistivity and electromagnetic induction, is among the most useful and easily obtained spatial properties of soil that has been widely used in agriculture since the end of XX century [10]. However, the idea of using conductivity/resistance to study soil pollution levels is not that widely accepted, and can be found in limited number of works, namely Seifi & Alimardani, Liu Z. et al., Medeiros et al. and Volkov S. But the results obtained by mentioned researches show the validity of this method [11–14]. Ukrainian researches Onyschuk et al., Bondar K., Bagriy & Kuzmenko, Chorny S., Lykhovyd P. have also worked in this field, but mostly used conductivity of soils as the indicator of agricultural quality or level of humidity and irrigation efficiency. However, Vyzhva et al. have developed the principles of electrometric testing of urban soils quality [15], while Hamkalo et al. offer to use soils conductivity for the evaluation of anthropogenic pressure intensity [16]. The most widely accepted application of conductivity testing is for salinization assessment [17]. We believe that the application of the given method is appropriate for local continuous control by the enterprise personnel, first of all fuel retailing objects.

Research methodology

The initial step in determining electrical soil resistance as a function of petroleum pollution is the development of model for the correlation between the resistance and the content of petroleum products. For this purpose, the samples of unpolluted soil are intentionally polluted with emulsions of oil product in water of a certain concentration. The soil samples should be taken from typical sites of the studied territory. For all the samples taken, the soil texture must be determined and it is advisable to use the sieve method. As a result, the aggregate percentages of the sand, dust, and clay fractions are estimated and the typical soil mechanical composition is determined by the USDA soil texture triangle.

Measurement of electrical conductivity/ resistance of the test system can be performed with a universal multitester equipped with contact electrodes. According to conventional approaches to the determination of the electrophysical properties of the soil, they must be wetted to full saturation with water or other solvent [14; 15; 18, p. 19] after the emulsion is added, if necessary. A control sample is also formed in which the oil emulsion is replaced with the same volume of pure water. Resistance measurement results are the average of three consecutive measurements.

In order to maximize the approximation of the experimental conditions to the real ones, the samples are stored in a ventilated room, which doesn't prevent the evaporation of petroleum products.

The measurement of the oil content must be carried out over a long period of time, since under the real conditions it is extremely rare that the measurement of the oil content in the soil are performed immediately after the pollutants are introduced into the soil.

The measurement results, together with the values of the concentration of petroleum products, are recorded in the Advanced Grapher spreadsheet to construct trend lines and to determine confidence intervals that will be the analytical boundaries for interpreting the results of actual soil samples testing.

Checking the dependencies obtained involves determining the relative error of measurements; information quality of the model dependencies is evaluated using the mean deviation of the actual values of the mean, with F-test and coefficient of determination. The predictive properties of the model are characterized by the Taylor index and the confidence interval width.

Electrometric Soil Surveys in Kyiv

To identify patterns of change in soil electrical conductivity/resistance, it is necessary to take samples of Kyiv soils to account their properties having influence on the results of testing. Therefore, soils of the following main types were selected: soddy-podzolic, dark-gray podzolic, gray forest and alluvial soils.

In accordance with the developed scheme of research, at the first stage, samples of appropriate soil types were taken from the background areas (suburban area of Kiev at a distance of at least 2 km from potential sources of pollution). The morphological properties, texture and pH of soils were defined (Table 1).

Table 1

Morphological properties of soil samples

Type of soil	pH	Soil texture	Morphological description
soddy-podzolic	6,9	light loam	grey wet tight
grey forest	7.0	Sandy	gray moist friable
alluvial	7.3	Loamy	dark gray moist dense
dark grey podzolic	6,7	Sandy	dark gray dry loose

From the obtained soil material, a series of samples were formed to simulate pollution with different types of petroleum products by introducing appropriate portions of the test substance. As the model the following market products were chosen: traditional gasoline A-95 and diesel fuel L (summer) and Semi-synthetic oil grade SAE 15W-40. After the introduction of the calculated portions of petroleum products, the samples were kept for 3 days to ensure a uniform distribution of heavy fractions along the soil profile and evaporation of the lightest fractions. Also each series of samples of each soil type included a control sample that did not contain petroleum products.

The electrical resistance of the samples (total number of samples — 54) was measured three times with 10-day intervals. The obtained data were averaged and the dependencies were plotted, showing the relationship between the concentration of different petroleum products in different soils and the level of its electrical resistance (see Figure).

At this stage, several important findings have been formulated:

1. Differences between the resistance of polluted and non-polluted samples, even with the lowest concentration, are significantly different by a value that exceeds the possible statistical error. The smallest difference is in soils polluted with gasoline, which can be explained by its rapid evaporation over time.

2. The highest values of electrical resistance are typical for samples containing semi-synthetic oil. It

is these petroleum products that are most commonly found in urban spills and resistant in the environment due to the low content of easily evaporating light fractions. At the same time, fuels — gasoline and diesel - will be more typical pollution near the storage facilities and petroleum products retailings.

3. Over time (total of 43 days), the resistance values changed, both increasing and decreasing values were detected, with the general tendency for a certain reduction of the resistance, which may indicate gradual change in the fractional composition of petroleum products due to evaporation, mineralization and reaction with humic components of soil.

4. The relative stability of changes in the electrophysical properties of soils polluted with petroleum products enables the use of this method to identify relatively episodic or chronic contamination of the relevant soil types.

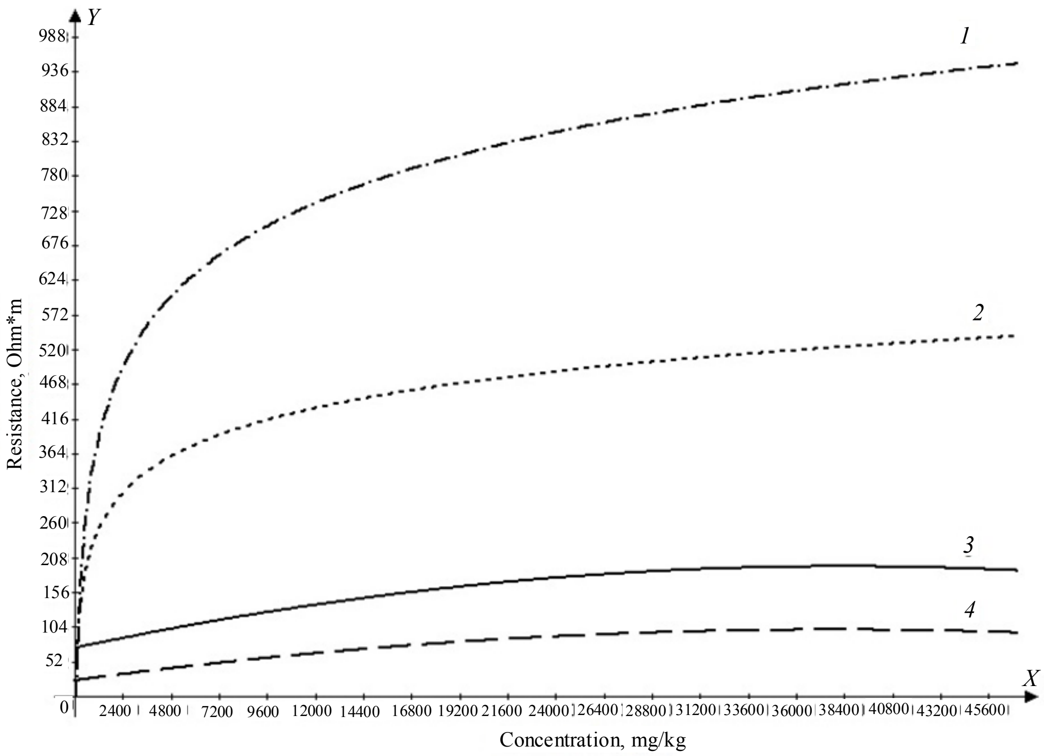
5. Differences in electrical properties of soils polluted with different types of petroleum products does not exceed 10–15 %, with the exception of extremely high pollution with lubricating oils, and therefore the averaged values may be used for the studied soil differences in real conditions provided uniform texture. Given the above, the model dependencies for each type of soil were developed without differentiation by types of petrochemicals (see Figure).

Approbation of model dependencies

In order to check the dependencies obtained, soil samples were taken in the area of influence of fueling facilities for public use. In the selection of objects, attention was paid to the types of soils at the facility, especially their texture. A total of 20 samples were taken — 5 samples for soils of each type.

Determination of oil content was carried out in parallel by two methods - the proposed electrometric method and gravimetric method according to the requirements of the standardized method of MBB № 081/12-0116-03 Method of measurement of petroleum products mass fraction by gravimetric method.

The measurement results (Table 2) show that the electrometric method can be used to determine petroleum soil, but certain limitations must be accounted.



Model dependencies of soil electrical resistance on petrochemicals content for major soil types:
1 — grey forest soil; *2* — alluvial soils; *3* — soddy-podzolic; *4* — dark grey podzolic

Table 2

Comparison of petrochemicals content determined by two methods

Sample number	Concentration by the gravimetric method, mg/m^3			Total relative error, Д	Concentration with absolute error, (m ± Д), mg/m^3	Concentration by electrometric method, (m ± Д)*, mg/m^3	Relative difference of actual and model values, %
	m_1	m_2	m				
1	5591	5543	5567,0	12,4	5567.0 ± 690.3	5650 ± 282.5	1.49
2	4958	5113	5035,5	12.7	5035.5 ± 639.5	5170 ± 258.5	2.67
3	6241	6290	6265,5	12,1	6265.5 ± 758.1	6350 ± 317.5	1.35
4	4381	4416	4398,5	13,0	4398.5 ± 571.8	4515 ± 225.8	2.65
5	3258	3297	3277,5	13.5	3277.5 ± 442.5	3398 ± 169.9	3.68
6	7048	7128	7088,0	11.7	7088.0 ± 829.3	7215 ± 360.8	1,79
7	6328	6387	6357,5	12,0	6357.5 ± 762.9	6480 ± 324.0	1.93
8	4453	4486	4469,5	13,0	4469.5 ± 581.0	4530 ± 226.5	1.35
9	5271	5332	5301,5	12.6	5301.5 ± 668.0	5465 ± 273.3	3.08
10	1713	1724	1718,5	14.3	1718.5 ± 245.7	1875 ± 93.8	9.11
12	3753	3777	3765,0	13.3	3765.0 ± 500.7	3946 ± 197.3	4.81
13	4240	4206	4223,0	13,1	4223.0 ± 553.2	4376 ± 218.8	3.62
14	2973	3061	3017,0	13.6	3017.0 ± 410.3	3188 ± 159.4	5.67
15	3103	3195	3149,0	13.6	3149.0 ± 428.3	3367 ± 168.4	6.92
16	1683	1649	1666,0	14.3	1666.0 ± 238.2	1790 ± 89.5	7.44
17	5328	5354	5341,0	12.5	5341.0 ± 667.6	5478 ± 273.9	2.57
18	4384	4329	4356,5	13,0	4356.5 ± 566.3	4475 ± 223.8	2.72
19	6443	6462	6452,5	12,0	6452.5 ± 774.3	6585 ± 329.3	2.05
20	4346	4325	4335,5	13,0	4335.5 ± 563.6	4465 ± 223.3	2.99

* **Note**: The error in the determination of the concentration of petroleum products by electrometric method only takes into account the value of the instrumental error of the digital ohmmeter, because the next work with the graphs of the concentration versus resistance used the built-in functions of the Advanced Grapher software, not visual estimates.

1. Low concentrations of petroleum products (within 25 % of sanitary standard) can be masked by measurement inaccuracy, since values up to 0.05 mg/m^3 are placed within the permissible error range by both gravimetric and electrometric methods.

2. The differences between the curves of the dependence of soil resistance on the hydrocarbons content for different petroleum products are partially imposed, which makes it impossible in some cases to make differentiated diagnostics: whether there is a high concentration of gasoline or the content of heavy oil fractions of lower concentration.

3. The level of soil resistance depends not only on the content of petroleum products, which leads to ambiguous interpretation of the results. In particular, the certain level of soils salinity, which is typical of soils located along the transport routes, leads to a decrease in soil resistance, which can mask oil pollution. However, other methods of determining the content of petroleum products in soil are also affected by impurities, as soil contains an extremely wide range of organic and inorganic components.

The Theil discrepancy coefficient calculated for the set of data obtained (actually determined by gravimetric concentrations and indirectly by the interpretation of electrometric data based on the developed models) is 0.024, which indicates a sufficiently high level of predictive reliability of the proposed dependencies. Therefore, the proposed method for determining petroleum pollution soil differs from the currently known methods by simplicity of implementation, minimal sample preparation sufficient accuracy.

Conclusions

1) Soil pollution with petroleum products have a wide range of environmental impacts and influences, among which two problems are of major concern: degradation of soil environmental functions and influence on soil biota and plants.

2) To determine the content of petroleum products in the soil, several basic methods are used, including gravimetric method, fluorimetric method, channel thin layer chromatography method, etc. However, the traditional methods are either instrumentally complicated or give not accurate results.

3) To analyze the petroleum products content it is possible to use electrometric analysis, which is

based on the change in electro-physical properties of soil, containing oil products.

4) To determine the electrical resistance of soil the samples of soils typical for Kyiv area were treated with emulsion of market petroleum products (gasoline, diesel fuel and lubricating oil) of different concentrations. The resistance values of the system under study were processed in order to reveal the mathematical regularity of the change in the value of electrical resistance depending on the concentration of petroleum products. This dependence has been formalized in curves that can be used to determine the gross content of oil product in soil, if the resistance of the wetted sample is known.

5) Soil sampling was conducted on the territory of the city of Kyiv in the area of influence of fueling facilities for public use to verify the dependencies obtained. The concentration of petroleum products in these samples was determined by the electrometric and traditional gravimetric method. The difference in results ranged from 1.35 to 9.11 %.

6) The proposed method can be used as an alternative to other conventional methods, although its application is limited by some considerations: inability to determine the exact composition of the pollution, relatively high error at low concentrations, and the effect of concomitant pollution on the results (primarily soil salination). Thus, the method is offered for application at fuel filling stations from the beginning of their exploitation when the initial resistance of soils at the adjoined areas is defined and controlled on a regular basis to detect any possible pollution by changes in typical resistance values.

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ДОСЛІДЖЕННЯ ЗАБРУДНЕННЯ ҐРУНТІВ НАФТОПРОДУКТАМИ ЗА ДОПОМОГОЮ ЕЛЕКТРОМЕТРИЧНОГО МЕТОДУ

Нафтопродукти — одне з найпоширеніших забруднень навколишнього середовища. Вони являють собою групу забруднювачів надзвичайно складного та різноманітного хімічного складу, що ускладнює їх виявлення та аналіз у компонентах навколишнього середовища. У статті розглянуті джерела та наслідки забруднення ґрунтів нафтопродуктами та відмічені найважливіші аспекти – погіршення якості ґрунтів та невиконання екологічних функцій. Порівняльний аналіз традиційних методів виявлення нафтопродуктів у ґрунті демонструє їх загальні недоліки — використання складного обладнання або низьку точність результатів. Зміна електричних властивостей ґрунту під впливом забруднення нафтою може бути використана як основа альтернативного методу дослідження. Для встановлення залежності між концентрацією нафтопродуктів та опором забруднених ґрунтів було проведено вимірювання електричного опору ґрунтів, характерних для території Києва, із штучно створеним забрудненням нафтопродуктами. Забруднення моделювалося за допомогою товарних продуктів — бензину, дизельного палива та напівсинтетичної оливи. Ґрунти, включені у дослідження — дерново-підзолисті, темно-сірі підзолисті, сірі лісові та алювіальні. Аналіз отриманих результатів показав, що основна різниця в опорі забруднених ґрунтів обумовлюється типом ґрунту, а не типом нафтопродуктів. Це дає можливість використовувати розроблені моделі для визначення присутності нафтопродуктів у конкретних ґрунтах, але перешкоджає точному визначенню складу забруднення. Запропонований спосіб був випробуваний на реальних зразках ґрунтів, взятих поблизу автозаправних станцій. Порівняння концентрацій, визначених електрометричним та гравіметричним методом, показує достатню надійність розробленої модельної залежності, але вони повинні бути пристосовані до конкретних властивостей ґрунту, зокрема текстури ґрунту, реакції та вологості.

Ключові слова: нафтопродукти; електрометрія; типи ґрунтів; забруднення.

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THE ANALYSIS OF SOILS POLLUTION WITH PETROCHEMICALS BY THE MEANS OF ELECTROMETRIC METHOD

Petroleum products are one of the most common components of chemical pollution of the environment. They represent a group of pollutants of extremely complex and diverse chemical composition, which complicates their detection and analysis in environmental components. Sources and consequences of soils pollution with petroleum products are considered in the paper, stating that it causes degradation of soil quality and failing to perform environmental functions. The comparative analysis of conventional testing methods of petroleum products in the soil demonstrates their common disadvantages – complicated equipment used or low accuracy of results. The change of electric soil properties under the influence of petroleum pollution is offered to be used as the basis of alternative testing method. The measurement of electrical resistance of soils, typical for the territory of Kyiv, with artificially created petroleum pollution was used to develop model dependencies between concentration of petrochemicals and resistance of pollutes soils. The pollution was modeled with market products – gasoline, diesel fuel and semi-synthetic oil. The soils involved in the research are soddy-podzolic, dark-gray podzolic, gray forest and alluvial. The analysis of the obtained results has demonstrated that major difference in polluted soils resistance is conditioned by the type of soil, but not the type of petrochemical in question. This enables the use of developed models to define the presence of petrochemicals in specific soils, but prevents the exact determination of the pollution composition. The proposed method was tested on real samples of soils taken at fuel filling stations. The comparison of the concentrations determined by electrometric and gravimetric method shows the sufficient reliability of the model dependancies developed, but they must be adapted to the specific soil properties, in particular soil texture, reaction and humidity.

Keywords: petroleum products; electrometry; soil types; pollution.

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ИССЛЕДОВАНИЕ ЗАГРЯЗНЕНИЯ ПОЧВ НЕФТЕПРОДУКТАМИ С ПОМОЩЬЮ ЭЛЕКТРОМЕТРИЧЕСКОГО МЕТОДА

Нефтепродукты — одно из наиболее распространенных загрязнений окружающей среды. Они представляют собой группу загрязнителей чрезвычайно сложного и разнообразного химического состава, что затрудняет их выявление и анализ в компонентах окружающей среды. В статье рассмотрены источники и последствия загрязнения почв нефтепродуктами и отмеченные важнейшие аспекты — ухудшение качества почв и невыполнение экологических функций. Сравнительный анализ традиционных методов обнаружения нефтепродуктов в почве демонстрирует их общие недостатки — использование сложного оборудования или низкую точность результатов. Изменение электрических свойств почвы под влиянием загрязнения нефтью может быть использовано в качестве основы альтернативного метода исследования. Для установления зависимостей между концентрацией нефтепродуктов и сопротивлением загрязненных почв было проведено *измерение электрического сопротивления почв, характерных для территории Киева, с искусственно созданным загрязнением нефтепродуктами. Загрязнение моделировалось с помощью товарных продуктов — бензина, дизельного топлива и полусинтетического масла. Почвы, включенные в исследование - дерново-подзолистые, темно-серые подзолистые, серые лесные и аллювиальные. Анализ полученных результатов показал, что основная разница в сопротивлении загрязненных почв определяется типом почвы, а не типом нефтепродуктов. Это дает возможность использовать разработанные модели для определения присутствия нефтепродуктов в конкретных почвах, но препятствует точному определению состава загрязнения. Предложенный способ был опробован на реальных образцах почв, взятых около автозаправочных станций. Сравнение концентраций, определенных электрометрическим и гравиметрическим методом, показывает достаточную надежность разработанных модельных зависимостей, но они должны быть адаптированы к конкретным свойствам почв, в частности текстуре почвы, реакции и влажности.*

Ключевые слова: нефтепродукты; электрометрия; типы почв; загрязнения.

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