EX-SITU REMEDIATION OF A PETROLEUM HYDROCARBONS CONTAMINATED SOIL

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Abstract. The remediation of polluted soils is a key concern of modern society due to importance of this natural resource for present and its conservation for future. Intensive anthropic activities facilitate sometimes the discharge of wastes or hazardous pollutants (like petroleum hydrocarbons) that lead to the soil contamination. The aim of this research is to summarize the experimental data resulted in the assessment of the quality indicators for a hydrocarbon contaminated soil (at depth of 5 cm and 30 cm) as well as results from an ex-situ remediation technology at laboratory scale performed using extraction with organic solvents (carbon tetrachloride, glacial acetic acid and hexane) and repeated water washes. The wastewaters containing traces of pollutant were collected and also treated by adsorption (with pine sawdust as adsorbent) followed by sedimentation.

The proposed ex-situ remediation was found efficient (>79% petroleum hydrocarbons removal) if the amount of excavated contaminated soil is limited and the proposed working conditions could be applied.

Keywords: ex-situ remediation, chemical extraction, organic solvent, hydrocarbons, adsorption.

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1. Introduction

Due to the intensification of economic activities as well as to the expansion of urban and rural agglomerations, the soil has modified its characteristics. Through the persistent action of some degradation processes unfavourable change/variation of some quantitative and qualitative quality indicators occur and lead to certain serious nutrients' imbalance in soil and mechanical-physical, chemical and biological pollution episodes. The main path for soil contamination consists in some accidental or incidental actions while in other cases are due to certain deliberate actions, i.e., waste disposal, storage, leakage, spills from containers or/and pipes, traffic accidents (Cresser *et al.*, 1993; Hester, 1986; Musy and Soutter, 1991; Zotica *et al.*, 2019; Zotica *et al.*, 2020).

Numerous changes of land use and predominant organic pollution episodes of soils were reported in the last thirty years. Consequently, investigations were designed to reveal the residual contamination of soils in order to estimate the potential risks and the measures necessary to bring these risks to an acceptable level and to redevelop the land to a 'sensitive' use (technical crops), or for a 'second' site rehabilitation (Keith,1991; Zaharia *et al.*, 2007; Zaharia and Suteu, 2011).

One of the highly resistant to biodegradation soil pollutants are the petroleum hydrocarbons (Hawrot-Paw and Nowak, 2012; Cresser *et al.*, 1993), capable to be accumulated in plants, or in living bodies (Hunt *et al.*, 2019; Waheed *et al.*, 2018) which exhibit neurotoxic or/and carcinogenic actions (Hunt *et al.*, 2019; Das and Chandran, 2011). The soil affected by this contamination is positioned mainly in the fuel storage areas, or fleets with fuel tanks (petrol/gasoline and diesel).

The selection of a viable solution for such soil remediation and further usage could be recommended only after the study of available techniques. The scientific literature has reported a lot of soil remediation methods such as physical, chemical or biological ones. Thus, for small and highly polluted area, the *ex-situ* soil remediation technologies with traditional mechanical-physical-chemical methods were proposed. The options could be: technologies based on the *physical extraction* of pollutants such as excavation, washing, flotation, electrokinetic extraction, or electro-flotation; technologies based on *chemical processes* such as extraction with organic solvents, acid or alkaline extraction, oxidation, precipitation and *thermal remediation processes* such as incineration, thermal desorption, vitrification (Ediene, 2020; Rada *et al.*, 2019; Simion, 2021; Sruti *et al.*, 2014). For a superficial or medium contamination, *phytoremediation*, or other *biological processes* such as biodegradation, bioaccumulation is *in-situ* alternative. The traditional technologies have some drawbacks associated with the relatively high costs, a possible impact on the environment if the *ex-situ*

treatment efficiency is not properly achieved and a lower acceptance from civil society.

However, the benefits of *ex-situ* soil depollution can compensate in a relatively rapid manner, the costs of excavation, transportation and physical-chemical treatment.

In this regard, the main objectives of this research work are:

(i) the assessment of an *ex-situ* remediation technique of a soil polluted with petroleum hydrocarbons (diesel and petrol/gasoline) by extraction with organic solvents: carbon tetrachloride (CT), or acetic acid 1:5 solution (AA), or hexane (Hx), followed by multiple washings with cold/hot water and

(ii) the selection of the available option which can support the practical use and allow the reuse of depolluted soil as support for technical crops.

2. Materials and methods

2.1. Reference soil and its sampling location

The reference soil samples were collected from the area adjoining the former controlled landfill Valea Seaca in Paşcani city), from each cardinal point of the site, at two depths (5 cm and 30 cm). The controlled landfill of municipal waste Valea Seacă (currently closed) is located on the DJ 208 road, at a short distance from the city (6 km). Groundwater was encountered on site at about 10.5 m. The freezing depth according to STAS 6054 is of 0.931 m from the landscape elevation. The site is arranged on a relief almost flat, with a slight slope to the south. All soil samples were mixed to prepare two representative soil samples for the depth of 5 cm and for 30 cm.

2.2. Fuel-contaminated and reference soil preparation

The two representative soil samples were treated in the laboratory with fuel (petrol/gasoline and diesel), becoming representative models of fuel-contaminated soils for the simulated area of the fuel tanks on this site: sample 1- with petrol/gasoline (d=0.71 g/cm^3; lower calorific power value of 30.77 MJ/L and upper calorific power value of 46.02 MJ/kg) and sample 2- with diesel (d=0.88 g/cm^3; lower calorific power value of 37.82 MJ/L and upper calorific power value of 45.61 MJ/kg (Zaharia, 2019). These two-representative fuel-contaminated soil samples were as follows: (a) two soil samples contaminated with petrol/gasoline: 1-5 cm and 1-30 cm, together with (b) two soil samples polluted with diesel: 2-5 cm and 2-30 cm. The principal quality indicator analysed for fuel-contaminated soil was the total petroleum hydrocarbons which was gravimetrically determined and expressed as the total extractible substances in petroleum ether (SETEP quality indicator).

2.3. Remediation technology used for soil decontamination

In experimental study of contaminated soil remediation, there were applied two operations: soil excavation and a chemical remediation by extraction with organic solvent followed by repeated washings (cycle of 2-3 washings) with water (cold/hot).

The reduction of total petroleum hydrocarbons (TPHs) contamination is expressed by the THPs removal (R, %) referring to the total extractibles in petroleum ether gravimetrically determined, Eq. (1):

$$R(\%) = \frac{c_i - c_f}{c_i} * 100 \qquad (1)$$

where, C_i is the initial SETEP content in the fuel-contaminated soil (mg/kg of dry soil) and C_f is the final SETEP content in the decontaminated soil (mg/kg of dry soil).

The washing with water is used to remove residual pollutants fixed in the soil matrix after extraction of petroleum hydrocarbons with organic solvents. The principle consists in the separation of pollutants from the soil and their transfer to the liquid or gaseous phase, under the action of water and mechanical shake. The wastewater treatment degree (%) related to the SETEP quality indicator is calculated with the well-known relation (Zaharia and Gradinaru, 2020) similar with Eq. (1).

In addition, the collected washing waters were subjected to a mechanical-chemical treatment process based on adsorption onto a residual sawdust pine biomass as adsorptive material, in order to attain the acceptable limits of direct discharge in an aquatic receptor nearby, or inside reuse.

Thus, the wastewater (WW) samples (washing waters used for fuel-contaminated soil after extraction with organic solvent) were contacted with the adsorbent (0.5 - 1.5 g of sawdust), in Berzelius flasks, volumes of 25 - 50 mL) and subjected to a slow initial shake (30 rpm) of 1-2 min, and then intermittently after each hour (for 1-2 min) of the adsorption period (no more than 10-24 h).

The pine sawdust was previously characterized (Zaharia and Gradinaru, 2020) being used only the particles with size < 1 - 2 mm.

2.4. Analytical analysis methods used for soil characterization

The main soil characteristics were analysed by using internationally standard analysis methods (Cresser *et al.*, 1993; Keith, 1991; LaMotte, 2008; Zaharia *et al.*, 2007) and pure analytical chemicals (p.a.) purchased from abroad (Merck Co.), or Romania (Chemical Co., Romania). The values of certain reference soil characteristics were reported as experimental data in a finished

research contract (Zaharia, 2003; Zaharia *et al*., 2007) and considered as reference soil characteristics in the investigated area.

Some quality indicators of the soil samples consists in (Zaharia *et al*., 2007; Zaharia and Suteu, 2011): (i) gases and vapours content (%), (ii) heat losses(%), (iii) actual pH (by direct reading of pH value using a HANNA pH-meter), (iv) total organic carbon (TOC) content (by oxidation method with concentrated H_2SO_4 and 0.25 N $K_2Cr_2O_7$ and further titration with Mohr salt solution in the presence of Feroin indicator), and (v) total petroleum hydrocarbons (TPHs) expressed by the total extractibles in petroleum ether (SETEP) (by their extraction from 20 g of soil with 30 mL of petroleum ether after mechanically 10 - 15 min-mixing and filtration over a funnel with 1g anhydrous Na_2SO_4 for water retention, and further collected filtrate evaporation in a weighted porcelain crucible).

The content of the total extractibles in petroleum ether (SETEP) was also analysed in the washing waters collected after repetitive soil washings with water. The SETEP determination consists in a gravimetric method application working on acidulated wastewater (pH $\leq$ 2, with 1 : 3 HCl) energetically mixed with 30 mL of petroleum ether in a separation funnel followed by separation of aqueous and organic phases; further, the organic phase is filtrated through a funnel with 1 g anhydrous Na_2SO_4 and the filtrate was subjected in porcelain crucible to evaporation (Mathieu and Pieltain, 2003). Difference between the weight of porcelain crucible before and after evaporation divided by the wastewater volume are the measures considered for determination of the SETEP value (mg/L).

3. Results and discussion

3.1. Characteristics of the reference soils and fuel-contaminated soils

The sampled soils from Valea Seaca site (Pascani) had a weakly acid to neutral reaction (6.63 - 7.85). The total organic content (TOC) varied between 1.89 – 3.92%, the total content of gases and vapours between 4.44 – 6.82%, heating losses were between 0.136 - 0.190 % and the total petroleum hydrocarbons expressed by SETEP indicator had values between 705 - 2500 mg/kg of dry soil for a depth of 5 cm, and 300 - 1750 mg/kg dry soil for a depth of 30 cm. The admissible value agreed by the environmental standards for SETEP is < 100 mg / kg of dry soil for agricultural land (sensible land) and < 200 mg/kg of dry soil for less sensitive land.

The results obtained for the non-contaminated reference soils for a depth of 5 cm and 30 cm are presented in Table 1. Carbonates occur only on the basis of the reference soil profile, and humus is present in quantities that fit well with the supply of organic matter.

The number of changeable cations, as well as those of hydrogen or aluminium, gives degrees of saturation greater than 80% which frames the soils

to eutrophic and certifies a good fertility. Also, the supply of nitrogen and potassium salts is good, and of mobile phosphorus is medium. As evidenced by the pedological, morphological, textural study and of the physic-chemical properties of the Siret terrace soils (Pascani county area), they have a natural production capacity between good and very good, the quality and quantity of the harvests depending on the agrotechnical and ameliorative works performed, the climatic characteristics of the year and the quality of the climate components.

Table 1

Certain quality indicators of prepared representative reference soils
(Valea Seaca, Pascani)

Soil quality indicator	Meas. units	Non-contaminated reference soil sample – **5 cm**	Non-contaminated reference soil sample – **30 cm**
pH	-	7.24 ± 0.61	6.98 ± 0.86
Gases and vapours	%	5.68 ± 1.24	5.52 ± 1.31
Heating loses	%	0.167 ± 0.027	0.144 ± 0.025
TOC	%	2.905 ± 1.015	2.41 ± 1.047
SETEP	mg/kg	705 ± 85	780 ± 98

The four prepared fuel-contaminated reference soils (2 reference soils for depth of 5 cm and 30 cm mixed separately with the two fuels: 1- petrol / gasoline and 2-diesel) were analysed, especially related to total petroleum hydrocarbons (TPHs) content (Table 2).

Table 2

SETEP values (mg/kg of dry soil) of the initial fuel-contaminated soil samples

No.	Fuel type	Soil sample	TPH (mg/kg of dry soil)	M.A.C. [mg/kg of dry soil]
1	1 – petrol/ gasoline	1 – 5 cm	11000	Normal value:
2		1 – 30 cm	12050	-Sensible land < 100
3	2 - diesel	2 – 5 cm	29500	-Less sensible land < 200
4		2 – 30 cm	15600	-Alert limit: < 1000
				-Intervention limit:< 2000

According to the values presented in Table 2, the fuel-contaminated reference soil samples are heavily polluted, far exceeding the values of the legislative norms (less than 200 mg/kg for less sensitive land, or less than 100 mg/kg soil for agricultural land), the exceeding's being 120 times and even 295 times higher than the legal norm.

The lowest THPs content expressed by SETEP value was of 11000 mg/kg of dry soil contaminated with petrol/gasoline (1-5 cm) and highest of 29.5 g/kg of dry soil contaminated with diesel (2-5 cm) for the lowest depth of soil sampling related to the surface, *i.e*. 5 cm.

3.2. *Ex-situ* remediation performance by extraction with organic solvents

Since preliminary laboratory tests have not led to high removals of TPHs using only water (< 21%, non-satisfactory results even under the conditions of using hot water, (70°C) (table 3), the extraction with organic solvents have been used followed by repeated washing/rinsing of treated soil with water (and further drying and relocation onto the original site).

Carbon tetrachloride (CT) was considered as a control solvent (all comparisons of TPHs extraction efficiencies with different organic solvents are related to this. Other two organic solvents had been tested for this *ex-situ* soil remediation technology, acetic acid 1:5 solution (AAc 1:5) and hexane (Hx).

In all series of laboratory tests performed, the same working methodology was applied and the gravimetric method was used for determination of THPs content (mg/kg dry soil).

The obtained results are summarized in Table 3.

Table 3

TPHs extraction efficiency from the studied fuel-contaminated soils

No.	Extraction with organic solvent + washings with water	Soil sample	Final TPHs, [mg/kg of dry soil]	(C.M.A.), [mg/kg of dry soil] Alert / Intervention	(No/Yes) exceeding C.M.A. after 1st soil extraction
1	CT + water (2-3 washings)	1 – 5 cm	2250	1000 / 2000	Yes
2		1 – 30 cm	3350	1000 / 2000	Yes
3		2 – 5 cm	2350	1000 / 2000	Yes
4		2 – 30 cm	1650	1000 / 2000	Yes / No
5	AAc 1:5 + water (2-3 washings)	1 – 5 cm	7168.5	1000 / 2000	Yes
6		1 – 30 cm	5660.4	1000 / 2000	Yes
7		2 – 5 cm	4047.9	1000 / 2000	Yes
8		2 – 30 cm	3120.3	1000 / 2000	Yes
9	Hx + water (2-3 washings)	1 – 5 cm	2750	1000 / 2000	Yes
10		1 – 30 cm	2250	1000 / 2000	Yes
11		2 – 5 cm	2570	1000 / 2000	Yes
12		2 – 30 cm	950	1000 / 2000	No
13	Water (3-4 washings)	1 – 5 cm	9103.3	1000 / 2000	Yes
14		1 – 30 cm	9737.1	1000 / 2000	Yes
15		2 – 5 cm	23310.3	1000 / 2000	Yes
16		2 – 30 cm	12450.2	1000 / 2000	Yes

The TPHs extraction efficiency with organic solvents were as follow:
(**a**) in the case of reference soil contaminated with gasoline supposed to extraction with (CT) (min 79.55% at 5 cm and 70.54% at 30 cm) closely followed by extraction with hexane (Hx) (min 75.00% at 5 cm and 81.33% at 30 cm) and

much smaller by extraction with acetic acid solution (AAc 1:5) which was of 34.83% at depth of 5 cm and 53.03% at depth of 30 cm, or

(**b**) in the case of reference soil contaminated with diesel fuel subjected to extraction with carbon tetrachloride (TC, control solvent of TPHs) (min 92.03% at 5 cm and 89.43% at 30 cm) closely followed by extraction with hexane (Hx) (min 91.29 % at 5 cm and 93.91% at 30 cm) and much smaller by extraction with acetic acid solution (AAc 1:5) (86.28% at 5 cm and 80.00% at 30 cm).

The results are clearly highlighted in Fig.1

The TPHs removal (Fig. 1) was very good for diesel-contaminated reference soils at both 5 cm and 30 cm, the values varying from min 86.28% to max 93.91% related to the much smaller ones obtained in the case of gasoline-contaminated reference soils (1.1 - 2.9 times lower).
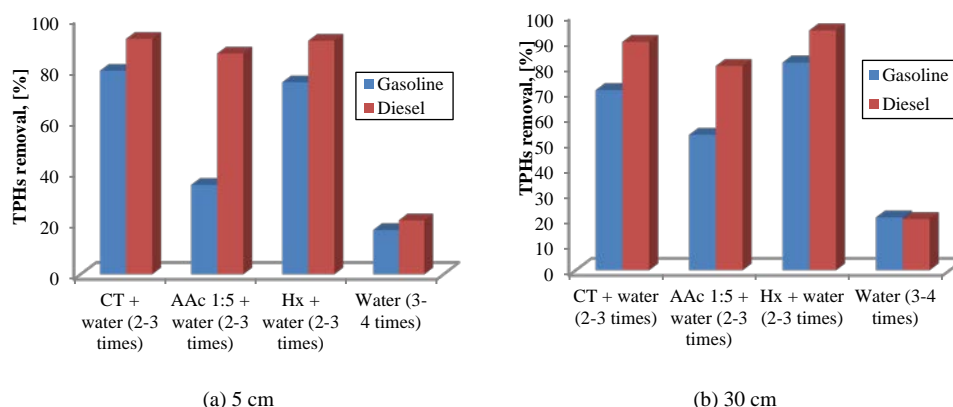

(a) 5 cm   (b) 30 cm

Fig.1 – TPHs extraction efficiencies from fuel-contaminated reference soils at depth of 5 cm (a) and 30 cm (b).

3.3. Wastewaters (washing waters) treatment performance based on a batch pine sawdust-based adsorption step

Following the repeated washing with water (3-4 times / washing cycle) of the decontaminated soils (after the extraction with organic solvent), there were collected wastewaters (WWs) which contained residual TPHs and other organics. These WWs have to be treated before direct discharge in an aquatic receptor, or inside reuse. A less expensive, simple, or ease to apply WW treatment step is a batch adsorption onto pine sawdust (fraction with no more than 1-2 mm size) for at least 10 h to 24 h for equilibrium establishment, followed by solid/water separation by centrifugation.

Currently, the adsorption treatment step using biomass waste as adsorbent is a technique considered in the context of circular economy. Therefore, the remaining WWs were treated with pine sawdust-based adsorbent

(0.5-1.5 g per 25-50 mL of remaining WW) in batch operating regime after an initial 1-2 min shake.

The results obtained on the removal of SETEP content were relatively good, and in some cases even ensured their complete removal (100%) (Table 4).

The best SETEP removal results were obtained in the case of WWs collected after the repetitive washings of diesel fuel-contaminated soil subjected to extraction with hexane (Hx). Much reduced SETEP removal values were found in the treatment of wastewaters subjected to the extraction with carbon tetrachloride (CT) and glacial acetic acid solution (AAc 1:5) applied on fuel-contaminated soil samples, mainly those at the earth's surface, *i.e.* depth of 5 cm.

Table 4

SETEP removal from remaining wastewaters collected after extraction with organic solvents of fuel-contaminated reference soils treatedby adsorption-settlement

Remaining WW after extraction and washing	Soil sample	Initial SETEP, [mg/L]	Final SETEP after adsorp-tion, [mg/L]	M.A.C. [mg/L] (NTPA 01)	Final pH
WW (**CT** extraction + 2-3 washings)	1 – 5 cm	66.67	35.00	20 / 30	6
	1 – 30 cm	50.00	20.00	20 / 30	6
	2 – 5 cm	116.67	27.50	20 / 30	6.5-6.8
	2 – 30 cm	66.67	35.00	20 / 30	6.5-6.8
WW (**AAc 1:5** extraction + 2-3 washings)	1 – 5 cm	66.67	40.00	20 / 30	3
	1 – 30 cm	66.67	17.50	20 / 30	4
	2 – 5 cm	50.00	32.50	20 / 30	3.5
	2 – 30 cm	50.00	0	20 / 30	3-3.5
WW (**Hx** extraction + 2-3 washings)	1 – 5 cm	80.00	0	20 / 30	5
	1 – 30 cm	14.00	66.80	20 / 30	5.5
	2 – 5 cm	150.00	0	20 / 30	6
	2 – 30 cm	133.33	25.00	20 / 30	5.5

In addition, it was found that, in the case of diesel fuel-contaminated soils, the remaining WW treatment degrees are of min 35% for extraction with AAc 1:5 solution (5 cm-depth) and max 100% in the case of extraction with hexane (Hx) (5 cm-depth), respectively, AAc 1:5 solution (30 cm-depth) (Fig. 2).

Also, after the pine sawdust-based adsorption step, the pH of the treated WWs (Table 4) must be taken into consideration. Thus, the WW resulted after extraction with carbon tetrachloride (CT) can be directly discharged into an aquatic receptor (natural water resource, or sewer system) because the final pHs fall within the admissible range of 6.5-6.8. In the rest of cases, the WW must be subjected to a neutralization step, since the pH is acidic, namely: between 3-4 for treated WWs resulted after extraction with AAc 1:5, and 5-6 for treated WWs resulted after the extraction with hexane (Hx). If the treatment process continues with other steps such as chemical oxidation and reduction, the neutralization is

no longer required, because the pH values are appropriate for the next treatment process step.

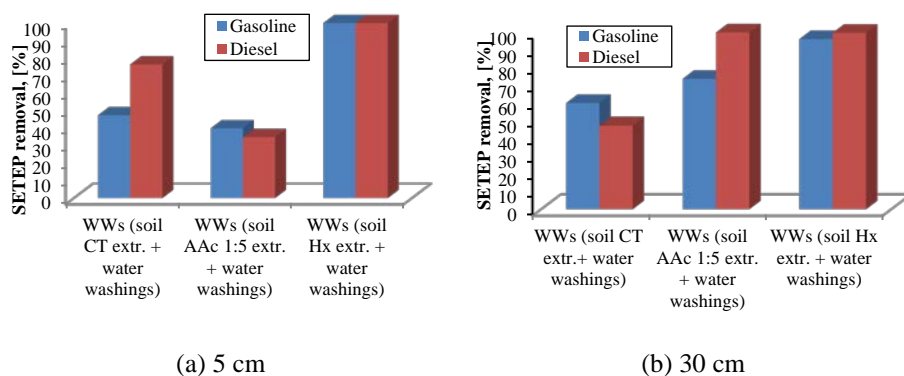(a) 5 cm   (b) 30 cm

Fig. 2 – Remaining WWs (reference soil washing waters) treatment efficiency (SETEP removal, %) by an adsorption-based step using pine sawdust as adsorbent

Fig. 2 clearly shows the efficiency of the WWs treatment process in the case of soils contaminated with diesel fuel compared to those contaminated with petrol/gasoline, as well as the total removal (100%) of SETEP content in the case of the washing waters resulted after the soil extraction with hexane (Hx) and repeated washings with water. As a result, the use of hexane solvent (Hx) in the extraction of petroleum hydrocarbons (THPs) from fuel-contaminated soils can be achieved with high efficiency (over 80%), and the treatment of remaining WWs can be complete in terms of SETEP content (*i.e.* extractables in petroleum ether, SETEP).

4. Conclusions

1. The *ex-situ* remediation technology of the studied soils contaminated with petroleum hydrocarbons by extraction with organic solvent (TC, AAc 1:5 and Hx) and repeated water washings is viable, ensuring the soil quality improvement.

2. The efficiency of *ex-situ* remediation by extraction with organic solvents (soil depth of 5 cm) ranged from a minimum value of 34.83% in the case of acetic acid 1:5 use till a maximum value of 79.55% in the case of carbon tetrachloride (CT) use; for soil sampled from the depth of 30 cm, the remediation efficiency varied in the range of 86.28% (with AAc1:5 solution) to 92.03% (with carbon tetrachloride-Tx).

3. In the case of diesel-contaminated soils, the remediation efficiency ranged from 86.28% (2-5 cm) in the case of AAc 1:5 up to 93.91% (30 cm) in the case of Hx use.

4. The remaining WWs treatment efficiency by an adsorption step using pine sawdust with regard to SETEP content removal is very good, in some cases even 100 % within the permissible limits of direct discharge in aquatic receptor.

5. *Ex-situ* remediation of fuel-contaminated soils by extraction with the three studied organic solvents is possible with good results, in particular if the amount of excavated soil is not too large.

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REMEDIEREA EX-SITU A UNUI SOL CONTAMINAT
CU PRODUSE PETROLIERE

(Rezumat)

Remedierea solurilor poluate este o preocupare cheie a societăţii moderne pentru a asigura calitatea solurilor şi conservarea acestora în viitor. Utilizarea intensivă a terenurilor, tratamentele chimice aplicate pentru maximizarea randamentului culturilor, deversarea unor deşeuri sau produse chimice periculoase accidentale sau alte activităţi antropice pot conduce la poluarea solului. Intre poluanţii periculoşi sunt considerate şi hidrocarburile petroliere provenite din combustibil (benzină şi motorină). Scopul acestei lucrări este de a rezuma rezultatele experimentale obţinute în analiza anumitor indicatori de calitate pentru două probe de sol de referinţă (la adâncimea de 5 cm şi 30 cm) şi de a evalua, la nivel de laborator, o tehnologie de remediere ex-situ a solului contaminat cu produse petroliere pe baza extracţiei cu solvenţi organici (tetraclorură de carbon, acid acetic 1:5 şi hexan) urmată de spălări repetate cu apă. Apele uzate colectate din procesul de spălare a solului au fost, de asemenea, epurate prin utilizarea unei trepte de adsorbţie (utilizând rumeguşul de pin ca adsorbant) urmată de sedimentare şi separarea fazelor.

Tehnica ex-situ propusă s-a dovedit eficientă (cu rezultate foarte bune în îndepărtarea hidrocarburilor petroliere >79 %), în special în cazul în care cantitatea de sol excavat nu este prea mare şi se aplică condiţiile adecvate de lucru.