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Surfactant-Enhanced Washing of Soils Contaminated with Wasted-Automotive Oils and the Quality of the Produced Wastewater

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ABSTRACT

An old automotive industrial site located at Mexico City with many years of operation and contaminated with heavy oil hydrocarbons, particularly spent oils, was assessed for restoration using the surfactant enhanced soil washing (SESW) process. The main goal of this study was to characterize the contaminated soil in terms of TPHs, BTEX, PAHs, and metals contents as well as microbiologically (total heterotrophs and specific degrading microorganisms). We also aimed to determine the surfactant type and concentration to be used in the SESW process for the automotive waste oil contaminated soil. At the end, sixteen kg of contaminated soil were washed and the produced wastewater (approximately 40 L) was characterized in terms of COD, BOD; solids, and other physico-chemical parameters. The soil contained about 14,000 mg of TPH/kg soil (heavy fraction), 0.13 mg/kg of benzo (k) fluoranthene and 0.07 mg/kg of benzo (a) pyrene as well as traces of some metals. Metals concentrations were always under the maximum concentration levels suggested by Mexican regulations. 15 different surfactants were used to identify the one with the capability to achieve the highest TPH removal. Surfactants included 5 anionics, 2 zwitterionic, 5 nonionics and 3 natural gums. Sulfopon 30 at a concentration of 0.5% offered the best surfactant performance. The TPH removals employing the different surfactants were in the range from 38% to 68%, in comparison to the soil washing with water (10% of TPH removal). Once the surfactant was selected, 70 kg of soil were washed and the resulting water contained approximately 1300 mg/L of COD, 385 mg/L of BOD (BOD/COD = 0.29), 122 mg/L of MBAS, and 212 mg/L of oil and greases, among other contaminants.

Keywords: Wasted Automotive Oils; Surfactants; Soil Washing; Waste Water

1. Introduction

The restoration of soil contaminated with hydrocarbons is often difficult and complex due, among other, to the adsorption on the soil matrix and the low solubility of these contaminants. It has been demonstrated that the more insoluble in water is the contaminant, the longer it remains in the soil matrix [1].

Many different techniques have been reported to restore soils contaminated with hydrocarbons; among of them the surfactant-enhanced soil washing (SESW) have recently emerged as highly cost-effective [2-4]. Surfactants reduce surface tension and form aggregates (*i.e.* micelles in aqueous solution), changing surface tension as result of surfactant's concentration on the solution's surface. Contaminants present in soil are removed by means of two phenomena: 1) The solubilization of compounds due to the reduction of surface tension (bellow the surfactant's critical micelle concentration (CMC) and 2) The mobilization of hydrophobic compounds due to the presence of the surfactant, at concentrations higher

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than surfactant's CMC value [5].

SESW process has shown very good results, and also has been considered as an economic and easy technique, so its application has increased in interest [3,6-9] as shown the literature review. Iturbe *et al*. [6] reported TPH (Total Petroleum Hydrocarbons) removals over 92% for contaminated soil with an initial concentration up to 17,238 mg/kg, when washing contaminated soils using the surfactant Canarcel TW80 in concentrations of about 0.5%. In other studies with SDS (sodium dodecyl sulphate), TPH removals above 90% were reported when treating oil-hydrocarbons contaminated soils [10].

Chin-Chi *et al*. [11], when washing contaminated soil, showed high TPH removals, between 63% and 62%, respectively using biosurfactants (*i.e.* rhamnolopids and surfactin) for the SESW process in a soil contaminated with a 9000 mg/kg of hydrocarbons. These authors also used two synthetic surfactants (Triton X-100 and Tween 80) for the same washing process, finding that synthetic surfactants were clearly less efficient (40% and 35%, removal, respectively). Another experiment using a non-ionic surfactant Brij 35 showed removals of crude oil from soils of 93.54% in a surfactant-enhanced washing of soil contaminated with 50,000 mg/kg of crude oil [12].

In this work, an old automotive industrial site located at Mexico City was assessed for restoration using SESW. The site maintains operations for many years and produced contamination with heavy oil hydrocarbons, in particular spent oils. Car service activities were carried out in the place, such as automotive oil change service, and wasted oils were stored in a submerged cement tank for many years. The company suspended its service more than 10 years ago and closed. The place was dismantled to become a residential zone.

During the process of characterization the site, which was carried out by the UAM-Azcapotzalco (Mexico), it was noticed that the oil cement tank suffered spills causing infiltration of the automotive oil waste in a large area of the old industry, contaminating the subsoil in an important extent. The soil contained about 14,000 mg of TPH/kg soil referring to heavy fraction (The analysis of the TPHs present in the soil was made by the suggested methodology in Mexican standard using dry soil). The maximum permissible limit established by Mexican regulations concerning contaminated soil with heavy fraction petroleum hydrocarbons suggests reducing the concentration of the site up to 6000 mg/kg.

The main goal of this study was to characterize the sub-soil of the old automotive industry, in terms of TPHs, BTEX (benzene, toluene, ethylbenzene and xylene), PAHs (polycyclic aromatic hydrocarbons), and a set of metals as well as microbiologically. To show the suitability of SESW process to remediate the site, including surfactant type selection and concentration to be used in the processes. Finally, the generated wastewater was characterized in terms of COD (chemical oxygen demand), BOD (biochemical oxygen demand), solids, and other parameters in order to determine the kind of process most suitable to treat the effluent to recycle the water into the soil washing process or to be disposed at the end of the soil remediation process.

2. Materials and Methods

2.1. Soil Sampling

The soil samples were taken from the subsoil of the site using a 25 SCRS Giddings model hydraulic punch (**Figure 1**), taking samples from 1.5 to 3.0 m deep, extracting about 70 kilograms of wet soil, which were stored in polypropylene black bags to avoid photo-degradation of pollutants and placed into pet boxes were they were maintained at room temperature during a period of 2 weeks before the experiments. Soil was thoroughly mixed to assure that concentrations of TPH as well as other organic and inorganic components were uniform for the whole batch of soil.

2.2. Soil Characterization

The composed soil sample was dried at room temperature for 3 days. The final moisture content in the soil was determined 10%, measured by mass difference. The physico-chemical characterization of the soil was carried out including texture, bulk and particle density, pore space, total and bioavailable nitrogen and phosphorus and conductivity measurements. TPH's, the 16 PAH (Polycyclic aromatic hydrocarbons) normed by USEPA and BTEX were also determined. Finally, some metals and metalloids concentrations (Na, K, Ca, Mg, As, Cd, Zn, Cu, Cr, Pb, Ni, Hg and Fe), were determined in the soil sample. The analyses were performed based on EPA

Figure 1. (a) Hydraulic punch employed at the sampling process; (b) Helicoidal drilling device.

standard methodology suggested in Mexican regulations, for TPH content the methods were EPA 9071B and EPA1664A.

The microbiological assessments were applied to the contaminated soil in order to determine the amount of present microorganisms. Plate counts were carried out using nutritive agar Petri dishes for heterotrophic bacteria count and for hydrocarbon degrading bacteria, diesel, gasoline or automotive-oil waste was employed as carbon source. The mineral medium used was as follows (in mg/L): KH_2PO_4, 8.5; K_2HPO_4, 21.75; $Na_2HPO_4 * 7H_2O$, 33.4; NH_4Cl, 1.7; $MgSO_4 * 7H_2O$, 22.5 $CaCl_2$, 27.5; $FeCl_3 * 6H_2O$, 0.25. These mineral media was adapted from the one suggested in a Mexican standard employed to determine BOD (NMX-AA-028 in NOM-001-SEMARNAT-1996). The bacteria were incubated at 30˚C over a period of 48 to 96 hours for heterotrophic and hydrocarbon degrading bacteria. The quantities of bacteria were expressed as a CFU/g of dry soil.

2.3. Washing Solutions

Five anionic surfactants were used: sodium dodecyl sulfate (SDS), sodium bencen-dodecyl sulfonate (SDBS), Texapon N40 (TN40), Sulfopon30 (S30) and Surfacpol A14104. The five nonionic surfactants employed along this work were Tween 80 (TW80), Tween 20 (TW20), Span 80 (SP80), Brij 35 (B35) and Emulgin W600 (EW). Two zwitterionic products were employed: Polafix CAPB and Polafix LO. Finally, three natural gums (capable to act as a surfactants/emulsifiers) were employed, *i.e.*, locust bean gum (LGB), guar gum (G) and mezquite seed gum (MZ). Distillated water was used as a blank for soil washing assessments. Some characteristics of the employed surfactants are shown in **Table 1**.

Two sets of soil washing experiments were performed; the first set was carried out using synthetic surfactants (anionic, nonionic and zwitterionic) concentrations of 0.5% and 0.1% for natural gums. In the second set of assessments, different concentrations of surfactants were tested of 0.25%, 0.5% and 1% for the synthetics surfactants and 0.05%, 0.1% and 0.2% for natural gums.

2.4. Soil Washing

The SESW experiments were carried out in 40 mL glass vials, where 6 g of contaminated soil dried at room temperature were added together with 20 mL of the washing solution or water. The vials were shaken at 200 rpm for a period of 23 hours and, then, they allowed to settle for an hour.

2.5. Measurement of TPH's in Soil Washing

After washing the soils, supernatant was separated and stored in the freezer until further analysis. The washed soil samples were placed in aluminum trays to be dried at room temperature (25˚C) for 3 days. Then TPH's concentration was determined by a gravimetric method after Soxhlet extraction using hexane as a solvent, described in Section 2.2. Soil humidity was determined and taken into account to report mg of heavy fraction/g of dry soil using the methodology suggested in Mexican standards.

Table 1. Some characteristics of the employed syntetic surfactants.

Surfactant	Ionic nature	Chemical name	Mol weight (g/gmol)	HLB	CMC (mg/L)	Reference
SDS	Anionic	Sodium dodecyl sulfate	288.4	40	400	[13]
SDBS	Anionic	Sodium dodecyl-bencenesulfonate	322.37	NR	1.5	[14]
Texapon 40	Anionic	Sodium lauryl ether sulphonate	442	NR	1458	[13]
Sulfopon 30	Anionic	Sodium lauryl sulphate	272	NR	150	This work
Surfacpol A14104	Anionic	NR	NR	NR	NR	[14]
Tween 80	Non-ionic	Sorbitan monoleate (Poe 20)	1308	15	65.4	[13]
Tween 20	Non-ionic	Sorbitan monolaurate	1226	16.7	60.74	[13]
Span 80	Non-ionic	Sorbitan monooleate	428	NR	NR	
Brij 35	Non-ionic	Lauric alcohol ether	1206	16.7	NR	[14]
Emulgin W600	Non-ionic	Nonyl phenol	483	11	45.06	[13]
Polafix CAPB	Zwitterionic	Cocoamide-propyl Betaine	NR	NR	80	[14]
Polafix LO	Zwitterionic	NR	NR	NR	NR	-

HLB: Hydrophilic Lipophilic Balance; CMC: Critical Micelle Concentration; NR: Not Reported.

3. Results and Discussion

3.1. Soil Characterization

Table 2 shows some of the physical and chemical characteristics of the soil employed in this study. As shown the content of organic matter, nitrogen and phosphorus; are interesting because suggest the possibility of applying biological treatment to the resulting waste water. The moisture content of the soil was 40%. The conductivity of the soil was normal in the case of a clayish soil (0.3058 µS/cm), as well as the pore space percentage (66%). The report suggests that pH is mildly basic. The organic matter content indicates a moderately rich soil. However, the nitrogen content in the soil is poor, contrasting with the very high contents of phosphorus. Metals and metalloids concentrations in soil (**Table 2**), were reported were below the limits set in Mexican regulation.

Two bacteria count processes were applied to the contaminated soil, heterotrophs and hydrocarbon degraders (diesel, petroleum and automotive oil waste). The results showed an heterotrophic microorganisms count of 4.1×10^7 CFU/g. Regarding the specific degraders, values of 1×10^8 CFU/g, 1.5×10^8 CFU/g and 1×10^8 CFU/g were found for diesel, petroleum and automotive waste oil degraders, respectively. The microbial count was similar to that reported by Iturbe *et al.* [6] for a soil contaminated with PAHs where bacteria counts of 1.8×10^8 CFU/g for heterotrophic and 5.4×10^8 CFU/g, 1×10^8 CFU/g and 5.6×10^8 CFU/g for diesel, petroleum and spent oil bacteria were reported. These values are higher than those reported by Bogardt and Hemmingsen [15] for soils contaminated with diesel and petroleum oil (1.9×10^7 CFU/g and 3.3×10^7 CFU/g, respectively). On the other hand, Hernández-Espriu *et al.* [16] reported a bacterial count of 2×10^{11} FCU/g for an agricultural soil contaminated with diesel. The observed amounts of microorganisms are higher than the minimum necessary for biodegradation process (10^4) stated by Fahnestock [6,16].

Regarding the analysis of PAHs, BTEX and TPHs, it was found that only the heavy fraction hydrocarbons exceeded the permissible limits established by Mexican legislation (**Table 3**). No BTEX were determined over the method detection limit and only two PAHs were found from the 16 PAH's regulated by USEPA. These PAHs, however, were found with concentrations below the maximum permissible limits established in the Mexican standards (**Table 4**). No light fraction oil content in the media was found indicating that the contamination was due solely to automotive waste-oil spills.

Despite the good performance and applications of surfactants for the transference of hydrocarbons into water, the removal efficiency depends also on several factors including nature, amount of surfactants, age of contaminated soil, soil properties and surfactant/oil/soil system behavior [10]. The highest removals were observed for

Table 2. Physicochemical characteristics of the contaminated soils.

Parameter	Result	Parameter	Result
Conductivity	0.3058 µS/cm	Pb	19.32 mg/kg
pH	8.5	Fe	4432.65 mg/kg
Apparent density	1.02 g/cm^3	Zn	13.60 mg/kg
Bulk density	3.0 g/cm^3	Na	652.22 mg/kg
Void space	66%	K	949.65 mg/kg
Soil percentual composition	60% clay 30% silt 10% sand	Ca Mg As	23855.82 mg/kg 14401.59 mg/kg 1.78 mg/kg
Texture	Clayey	Cd	2.80 mg/kg
Organic matter	2.12 %	Cu	6.73 mg/kg
Total phosphorus	605.60 mg/kg	Cr	<1.0 mg/kg
Available phosphorus	18.80 mg/kg	Ni	9.87 mg/kg
Total nitrogen	0.058%	Hg	0.11 mg/kg
Avail. nitrogen	9.93 mg/kg		

Table 3. Mexican standards for PAHs, BETEX and TPHs.

Parameter	Maximum concentrations (mg/kg dry soil)		
	Agricultural soil	*Residential soil*	*Industrial soil*
	TPHs		
Light fraction	200	200	500
Median fraction	1200	1200	5000
Heavy fraction	3000	3000	6000
	BTEX		
Benzene	6	6	15
Toluene	40	40	100
Ethylbenzene	10	10	25
Xylene	40	40	100
	PAHs		
Benzo (a) pyrene	2	2	10
Dibenzo (a,h) antracene	2	2	10
Benzo (a) antracene	2	2	10
Benzo (b) fluorantrene	2	2	10
Benzo (k) fluorantrene	8	8	80
Inden (1, 2, 3, cd) pyrene	2	2	10

Table 4. TPH's and some PAH's present in the contaminated soils.

Parameter	Results (mg/kg)
TPH's (heavy fraction)	14,705
Benzo (k) fluoranthene	0.1280
Benzo (a) pyrene	0.0682

Sulfopon 30, Tween 20, CAPB Polafix and mezquite seed gum with 59%, 54%, 52% and 55% of TPHs removal respectively. Zamudio-Pérez *et al.* [17] reported TPHs removals of 57.7% when washing an oil-contaminated soil, employing Brij 35 (**Figure 2**). They also tested natural gum as washing solutions. The best TPHs removal for natural gums was obtained with locust bean gum (31%).

The second set of washing assessments different surfactant concentrations was carried out with the best of each type except mezquite seed gum. The last was replaced by guar gum because mezquite seed gum is not a commercial surfactant and its production method is complex. Three concentrations for every surfactant were evaluated in the second washing procedure. It was observed that Sulfopon 30 (0.5%) achieved the highest percentage of removal 60%.

Tween 20 (TW20) rendered removal efficiencies of 20%, 54% and 55% with solutions of 0.25%, 0.5% and 1% respectively. CAPB showed similar behavior, in this case, removals of 53% and 54% were achieved using concentration of 0.5% and 1% (**Figure 3**).

Regarding the TPHs removal efficiency using guar gum, it was observed that the concentration of 0.1% produced the best result, reaching 54%. Same result as in the first wash. It is noteworthy that although it was not the highest removal percentage the experimental set showed the greatest amount of TPHs removal per gram of product (**Figure 4**), as the washing solutions used has lower concentration than synthetic ones.

In experiments made before, the standard deviations were not over the 5%, in this experiment the analysis showed the same behavior.

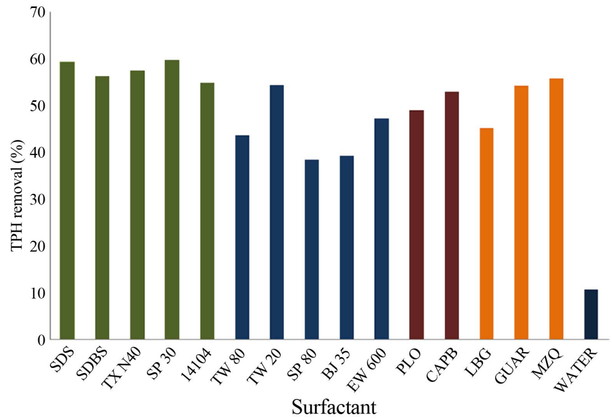

Figure 2. TPHs removals for the 15 washing solutions and water.

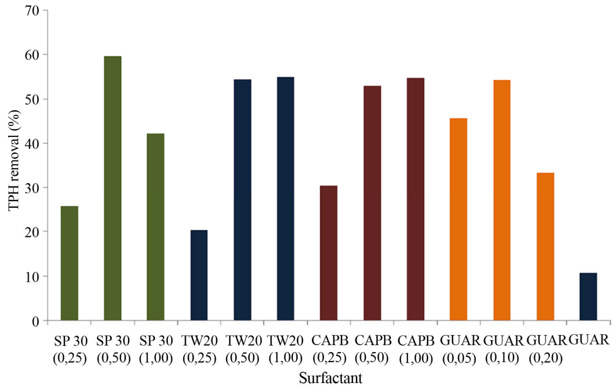

Figure 3. TPH's removals when using different surfactants concentrations for SP30, TW20 and guar gum.

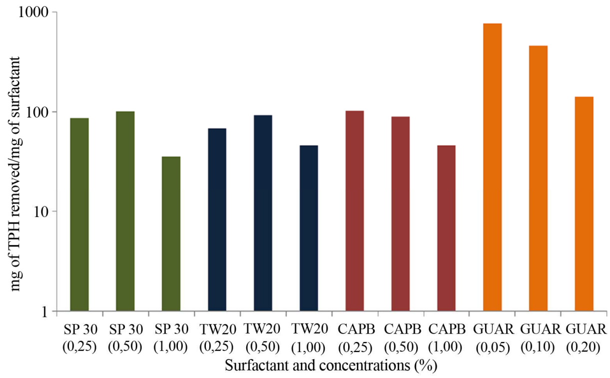

Figure 4. TPH's removals per mg of surfactant employed.

3.2. Characterization of the Generated Wastewaters

The scaling-up of the washing process were carried out at the best conditions. In order to do that 16 k of soil were washed with Sulfopon 30 at 0.5%. Approximately, 40 L of wastewater were produced and characterized in terms of COD, BOD_5, turbidity, electrical conductivity, color, hardness, MABS (methylene blue active substances), oils and greases, as well as 4 selected metals. Results are depicted in **Table 5**. As shown COD value was above 1300 mg/L which is below the reported by Bandala *et al.* [18] for refinery wastewater. Measured of BOD concentration was 380 mg /L generating a BOD_5/COD ratio of 0.29 rating the wastewater as poorly biodegradable. Torres *et al.* [19] reported a rate of biodegradation on 41.7% (moderately biodegradable) of soil washing waste water from a site contaminated with hydrocarbons. The higher COD and BOD values obtained are due to the oil content

Table 5. Characteristics of the produced wastewater.

Parameter	Result	Zamudio Perez *et al.* [17]*	Torres *et al.* [3]
COD	1329 mg/L	1468.0 mg/L	20,153 mg/L
BOD_5	385 mg/L	289.6 mg/L	8410 mg/L
BOD_5/COD	0.29	0.197	0.41
Turbidity	1540 FAU	525 UNT	NR
Conductivity	1107 µS	2580.0 µS	1,353 µS
Color	92 Pt/Co	3625 Pt/Co	NR
Hardness as $CaCO_3$	489 mg/L	22.50 mg/L	337.31 mg/L
MBAS	122 mg/L	0.015 mg/L	3368.0 mg/L
Oil and greases	212 mg/L	6.0 mg/L	94.5 mg/L
Pb	0.401 mg/L	20.13 mg/L	1.11 mg/L
Fe	19.05 mg/L	11.25 mg/L	289.64 mg/L
Cr	0.07 mg/L	0.023 mg/L	1.25 mg/L
Al	24.21 mg/L	23.62 mg/L	429 mg/L

*When using TW80.

in the soil and the surfactant used in the washing procedure.

Differences in organic content for the wastewaters produced in this work, and those reported by Torres *et al*. [19] are significant. Nevertheless, it is important to remark that the produced wastewater characteristics will derive from several factors, *i.e.*, 1) the soil type (sandy, loamy, clayey); 2) the contaminant type (light, medium, or heavy fractions in the case of oil derivatives); 3) the history of the contaminated soil, subjected to aging processes (young *versus* old spills); 4) the efficiency of the washing system (surfactant type and concentration, soil/ water ratio, energy input).

As observed, similar values for conductivity, hardness, the four selected metals and even for oil and greases were found. Important differences can be observed between the wastewaters generated in this work, and those reported by Torres *et al*. [19], when comparing COD (15 fold), BOD (21.8 fold) and the BOD/COD value (1.4 fold). It is interesting to note that MABS and oil and greases values were rather different (27.6 and 0.44 fold, respectively).

There is very little information on quality of wastewaters produced during the washing of contaminated soils; however, the characterization of these effluents is relevant regarding its treatment [17,19,20].

4. Conclusions

In the selection of a surfactant for a washing soil, it could be helpful to propose different type of surfactants to identify the higher removal of the contaminant. Also it is necessary to establish the concentration of the surfactant at the greater removal.

The removal of TPH's by soil washing vary for each type of surfactant, in this case, the greater removals were observed after using the SP30, however when looking at the milligrams of TPH removed for each gram of surfactant employed, the natural gum removed more TPH, because the concentrations required were five times lower. The characterization of the resulting water is relevant due to the treatment as suggested of the high rates of removal of TPH's also the containing of the surfactant used in the washing process.

Our research group is working on the treatment of the generated wastewaters in a low-cost packaging material submerged biofilter inoculated with hydrocarbon-degrader microorganisms isolated from the original contaminated soil. Besides, the changes in the biofilter micro-flora due to the system operation (*i.e.*, residence time, wastewater COD initial concentration and surfactant concentration) are being evaluated using DGGE technology.

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