



Remediation of aged hydrocarbon contaminated soil by washing in fluidized bed column

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Abstract: Soil contamination with hydrocarbons represents a worldwide problem, especially for oil-rich countries. Soil contamination becomes inevitable due to different accidents, aboveground spills, and leakage, threatening the fauna and flora. The purpose of this study is to remediate One-year aged contaminated soil with crude oil (23490 mg/kg) using the fluidization technique in a laboratory-scale column. Free water and surfactant solutions were used for washing at different operating conditions. The efficiency of the method was evaluated by the calculation of the total petroleum hydrocarbons (TPH) removal ratio. Without the addition of surfactant, the cleaning operation was not sufficiently efficient, especially at room temperature where the removal ratio was only about 18%. Raising the liquid temperature leads to some improvement where the TPH removal ratio reached 49% at 50°C. With the use of solutions containing Sodium Laureth Sulfate (SLES) as a surfactant, an important enhancement of removal ratio was noted, along with an important reduction in operating time, washing solution volume, and energy consumption. The use of alternatively working/stopping operation mode contributes to the improvement of efficiency. TPH removal ratios up to 99% were obtained under some favorable conditions. This research shows encouraging results for expanding towards the industrial level with clean and sustainable resources.

Introduction

Crude oil production is the driving force behind the economic development of many countries, particularly oil-rich ones. However, the release of crude oil due to their extensive use during extraction, refining, and distribution (aboveground oil spills, leakage, and other accidents) have caused a serious threat to the environment and human health, owing to their carcinogenicity and neurotoxicity (Han et al. 2009; Gao et al. 2014; Fanaei et al. 2020).

To maintain the sustainable development of the local ecosystem, the remediation of petroleum-contaminated soil is essential. Many technologies and strategies have been developed, including natural attenuation, bioremediation, phytoremediation, thermal treatment, soil washing, electrochemistry, and oxidation. These approaches can be used either independently or in combination. However, these techniques may have some drawbacks in terms of environmental, economic, and efficiency application (Huang et al. 2020; Liu et al. 2021).

Among all the aforementioned methods, washing processes have exhibited more interest in the last years, judging by the number of annual publications which has increased from 32 in 2012 to 91 in 2019 (Liu et al. 2021). This is mainly due to their low operating costs and high efficiencies (Kuppusamy et al. 2017). In the majority of previous studies dealing with

soil washing, the liquid effluent is directed downward and crosses a fixed bed of contaminated particles (Viglianti et al. 2006; Gitipour et al. 2015; Saeedi et al. 2019). Furthermore, laboratory-scale devices were used causing difficulties to scale up to industrial levels (Han et al. 2009; Olasanmi and Thring 2020).

In comparison with washing using a fixed bed of particles crossed by a descendant liquid flow, upward fluidization of the soil medium has the advantage of rupturing the soil skeleton, allowing the release of trapped hydrophobic contaminants. The use of fluidization for soil washing was first performed in 1998 by Niven and Khalili (Niven and Khalili 1998). In their study to remediate diesel contaminated soil, they proposed an in-situ treatment referred to as upflow washing. Despite their impressive results showing the efficiency of the treatment, an important parameter was not taken into account, namely the aging effect (the contamination was processed 2 minutes before washing). In fact, weathering results in greater binding strength of crude oil to the soil as well as higher contaminants' average molecular weight, viscosity, and density. Therefore, weathered contaminants are more difficult to remediate than recently contaminated soil (Urum et al. 2005). Several other studies had used fluidization as washing technique for soil remediation. Including but not limited to, thermal treatment using Nitrogen gas fluidization (Lee et al. 1999) or for enhancing the aeration for the biodegradation of diesel-oil contamination (Arrar et al. 2007).

It is known that the solubility of oil in water is the dominating factor for contaminated soil washing (Huang et al. 2020). Thus, surfactants were applied to ameliorate the washing efficiency. Surfactants can reduce the interfacial tension between the different polar phases because of their unique structure which consists of both hydrophilic and hydrophobic components. By reducing the interfacial tension, the solubility increases, thus reducing the desorption time (Huguenot et al. 2015; Rongsayamanont and Tongcumpou 2020).

The purpose of this work is to analyse the removal of total petroleum hydrocarbons (TPH) from contaminated soil in a liquid fluidized bed. The aim is to examine the efficiency of the washing operation when aged-contaminated soil is treated using both free water and Sodium Laureth Sulfate (SLES) solutions. For the knowledge of the authors, this is the first study that tests the efficiency of the fluidization technique on washing procedure in the presence of surfactant, especially SLES. Effects of several operation conditions on operation efficiency were tested (temperature, surfactant concentration, column operation mode: the working/stopping procedure).

Material and methods

Materials

The crude oil used for the contamination was supplied by a petroleum company (SONATRACH) working in an oil field situated in the south of Algeria (Haoud Berkaoui field, Ouargla). The crude oil was characterized as light oil with low sulfur content (density at 288.15K: 0.8061 g.cm⁻³, viscosity at 293.15 K: 3.87, salinity: 10.3 mg.l⁻¹).

Sodium Laureth Sulfate (SLES) (Texapon N70) was used as a surfactant. This surfactant (commercial grade) was purchased from BASF, Germany, and was used without any further purification. Its critical micelle concentration (CMC) was measured by the Du-Noüy Ring method. Its chemical structure is presented in Figure 1 and some of its key properties are listed in Table 1. Though it has not been widely explored in soil remediation studies, the selection of SLES as a surfactant in this study was based on its availability, its commercial low price

(2.07 USD/kg (Assawadithalerd and Phasukarratchai 2020)) and its low toxicity (1995 mg/kg, acute oral toxicity for rats (Walker et al. 1967)). In addition, good results were obtained when SLES solutions were used to the removal of pesticides (Ortiz et al. 2018), cadmium and zinc (Assawadithalerd and Phasukarratchai 2020), and diesel (Hernández-Espriú et al. 2013) from contaminated soils.

The soil used for the experiments was collected from a non-contaminated area, out of Ouargla City (31°54'09.2"N 5°08'12.7"E). It consists of dune sand, the same one found in most contaminated Saharan regions. The soil was initially filtered through a 2 mm sieve to remove coarse materials, then passed through six other sieves of different openings to determine its granulometric composition. The soil was characterized in terms of particle size distribution, density, and minimum fluidization velocity (see Table 2).

Sample preparation

The contamination was artificially made by pouring 50 g of crude oil into 1 kg of the soil and mixing vigorously to get homogeneous contamination of 50,000 mg/kg. A total mass of 20 kg of contaminated sand was prepared this way to have enough quantity for further experiments. The contaminated soil was left in the open to age for one year. At the end of this period, the contamination degree was measured again before starting the washing experiments.

Washing procedure

The experiments were carried out in a fluidized bed apparatus (GUNT, CE220), which consists of a removable Plexiglas column washing test 44 mm inside diameter and 550 mm height. A diaphragm pump provides the washing solution from a storage tank and delivers it at the bottom of the test column. The washing solution flows upwards through a porous sintered-metal plate. Another sintered-metal plate is placed at the top of the test column whose role is holding entrained particles. The effluent at the column exit passes through a funnel and the entrained oil is separated from the washing solution. The former is collected, and the latter is recycled. Figure 2 represents a schematic of the setup.

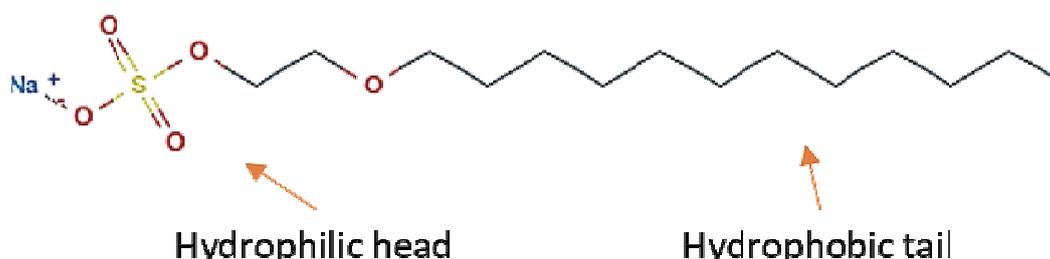


Fig. 1. The chemical structure of SLES (SLES)

Table 1. Some SLES chemical properties

Commercial name	Chemical name	Type	Molecular weight (g/mol)	Chemical formula	PH	CMC (mg/l)
TEXAPON® N 70	Sodium Laureth Sulfate (SLES)	anionic	382	C ₁₄ H ₂₉ NaO ₅ S	7.0 to 9.0	200

At the end of each experiment, any floating oil was collected. The washing solution was recycled or removed, and the soil was air-dried for at least 24 hours, then oven-dried at 105°C for 1–2 hours. The soil sample was homogenized afterward and prepared for the extraction process. The column and its component were washed between experiments with water and detergent.

Preliminary tests were performed to determine the minimum fluidization velocity of soil particles (U_{mf}) by means of pressure drop measurements across the bed. All experiments were performed beyond this minimum fluidization velocity to ensure good mixing of the soil particles. Two types of experiments were conducted: using ordinary water as washing media and using SLES surfactant solutions.

Extraction method

The Soxhlet extraction method (EPA 1996) was applied to measure the TPH concentration before and after fluidizations. The TPH removal ratio was estimated using a weighting procedure and the following equation:

$$\text{TPH removal ratio} = 1 - \frac{\text{mass of residual contamination}}{\text{mass of initial contamination}}$$

$$\text{TPH removal ratio} = \frac{\text{mass of washed sand before extraction} - \text{mass of washed sand after extraction}}{\text{mass of initial contamination}}$$

For the experiments performed in duplicate, the average, and standard deviation were calculated. One-way analysis of variance (ANOVA) was applied to determine the statistical significance of parameters. ANOVA was tested for $P < 0.05$ for significance.

Results and discussions

Soil and contamination characterization

By sieve analysis, the used soil was classified as fine sand with a mean diameter of 199.76 μm . Table 2 gathers some of its main characteristics.

TPH content of the contaminated soil after one year of aging was found to be 23.49 g/kg. This indicates evaporation of approximately 53% mass of the initial contamination (this value is consistent with the light nature of the used crude oil). In order to test if all volatiles were evaporated during the aging period, a sample of 100 g of contaminated sand at 23.49 g/kg was oven-dried for one hour at 105°C. The mass loss due to evaporation was found to be not more than 0.2% of the initial contamination mass. Therefore, it was considered for all further experiments that the contamination degree of the soil was at 23.49 g/kg. This value is within the range of real observed contaminations in a number of sites all over the world (24.26–1072.45 g/kg in the northwest of Algeria (Mebarka et al. 2012), about 32 g/kg at some sites in Mexico and real contaminated lands in Iran (Hernández-Espríu et al. 2013; Fanaei et al. 2020).

Table 2. Some soil characteristics. a: using Leva's (1959) equation

Particle size	Coarse sand (1.0–0.5 mm)	0.35%
	Medium sand (0.5–0.25 mm)	45.60%
	Fine sand (0.25–0.10 mm)	44.45%
	Very fine sand (0.10–0.05 mm)	8.86%
Texture	Fine sand	
Mean diameter (μm)	199,78	
PH	4.06	
Density (g/cm^3)	2.708	
Theoretical U_{mf} (m/s) ^a	$4.05 \cdot 10^{-4}$	
Experimental U_{mf} (m/s)	$3.3 \cdot 10^{-3}$	

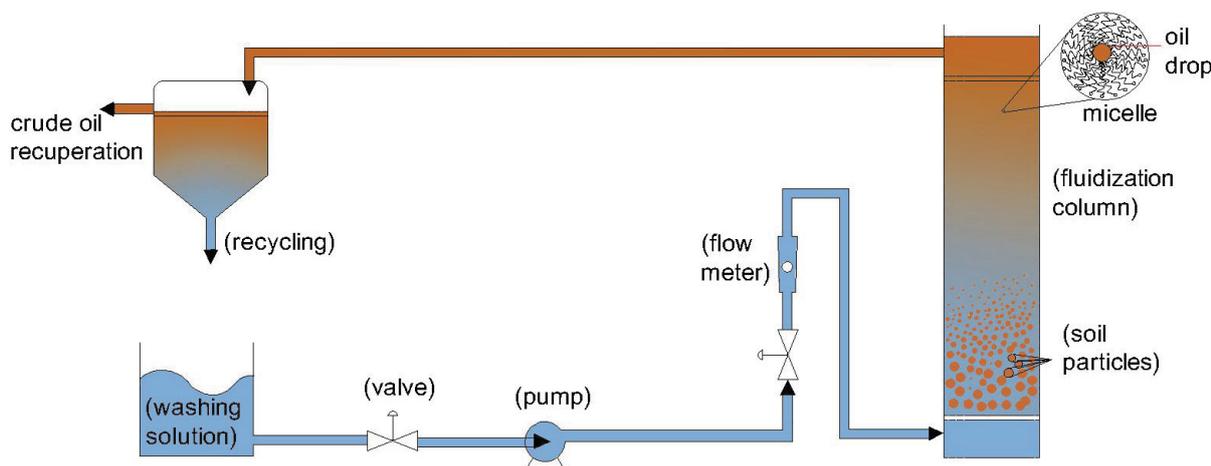


Fig. 2. Schematic representation of laboratory experimental setup

General observations during washing tests

During free water fluidization tests at 2.7 times the minimum fluidization velocity and room temperature, strong aggregation was observed causing caking, with the appearance of the channeling phenomenon leading to poor fluidization quality. The fragmentation of the soil block takes up to 20 minutes before the fluidization occurs. Oil removal occurs by releasing small droplets that stick to the column walls, producing – when coalescing – brown lines along the column wall. On the top of the fluidized bed, oil droplets coated with fine sand grains are observed. With an outside disturbance, some of those droplets were released and entrained to the freeboard of the bed. Similar observations were reported by Niven and Khalili (Niven and Khalili 1998).

With the presence of SLES in the washing solution, the column conditioning was fast and spontaneous. Neither channeling nor bed cohesion was observed. The fluidization occurs in a perfect way right after the first couple of minutes and the recovered oil forms an emulsion with the washing solution. However, after one week of settling the effluent, a layer of dark oil appears.

Free water washing tests

Two factors were considered when investigating washing efficiency: fluidization time and water temperature. In each experiment, a mass of 200 g of contaminated soil was fluidized at 0.88 cm/s (2.7 times U_{mf}) using approximately 7 liters of water recycled continuously. For the fluidization time effect, four experiments were performed along different operating times. Results of TPH removal are listed in figure 3. It can be seen that the separation efficiency was relatively low for all experiments. Indeed, by doubling the fluidization duration (from 60 to 120 min) the TPH removal ratio increases only by approximately 2%. The results of the ANOVA test at an important level beyond 0.05 ($P = 0.25$) indicate no significant effect of washing time on TPH removal ($P > 0.05$), yet a reasonable standard deviation was found (2.5% max). This is indicated by the error bars in figure 3. Therefore, the reproducibility of the results was considered acceptable.

These observations are in agreement with the report of Qi, B. et al (2021) on the effect of dry and wet soil on oil removal, where they found that the dry soil (even for sandy soil, which is relatively easy to wash) is selective and the adherence of the strong polarity compounds presented in the crude oil exhibits difficult mobility (Qi et al. 2021).

In fact, the observed low decontamination efficiency was due to the appearance of particle segregation phenomenon caused by a poor mixing inside the fluidized bed at the considered water velocity. These observations are in agreement with those reported in several studies in which it was shown that the removal of petroleum hydrocarbons using free water is not considerably affected by the mixing time (Lai et al. 2009; Chaprão et al. 2015). However, some other studies (Gautam et al. 2020) supported that TPH removal efficiency is positively correlated with mixing time.

During fluidization, it was observed that segregation takes place causing the bed to split into two layers: a layer at the bottom constituted of medium particles and a dark layer at the top constituted of fine ones. The oil removed from medium particles was caught by fine ones forming a stabilized oil/water/fine particle emulsion. Measures performed separately for both layers have revealed that in the fine fraction, the contamination has increased from 23.49 g/kg initial to 24.95 g/kg after washing, while in the medium fraction, the TPH removal ratio was 26.34%.

The effect of water temperature on TPH removal was investigated over four experiments. For each experiment, the operation time was fixed at 60 minutes while changing the temperature from ambient to 50°C. Results showed an increase in the TPH removal ratio from 18 to 49.54% (see Figure 4). This raise is due to a combination of two but not exclusively different effects: The increase of temperature reduces oil viscosity and interfacial tension which enhances the desorption process and facilitates its entrainment by water. Similar effects were reported by several authors (Fanaei et al. 2020). Olanmi and Thring, reported that the effect of temperature on the soil washing operation efficiency depends on the type and the nature of contamination (Olanmi and Thring 2020). The result at 50°C was in good

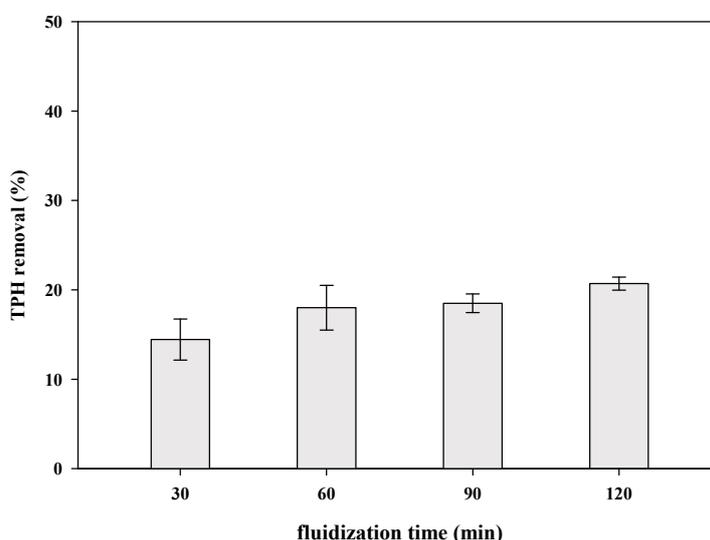


Fig. 3. Variation of TPH removal ratio with time (free water washing)

agreement with that found by Ould Saadi and Dounit (Ould Saadi and Dounit 2014). Although using free water is the best environmental and economical solution, the results are yet not sustaining.

What makes the situation worst the temperature exciding 50°C at summer in the studied regions, which according to (Vuruna et al. 2017) results in faster vertical migration of oil contaminants due to lowering its viscosity particularly in sandy soils because of their higher permeability. So the idea is to further study the effect of temperature using solar or sustainable energy making use of this high temperature.

SLES solutions washing tests

Kinetics of the washing operation

The kinetic aspect of the clean-up operation was investigated over a series of experiments performed with the use of SLES solution at 5 g/l concentration (25 CMC). In each experiment, a mass of 200 mg of contaminated soil was fluidized by the SLES solution at $2.7 U_{mf}$. The operation time of the experiments

ranges from 3.5 to 60 minutes. Results are reported in figure 5, where the TPH removal ratios obtained for SLES solutions are represented together with those obtained with the use of free water. With the presence of SLES, the efficiency of the clean-up was considerably enhanced, going from 20.69% to 93.45%. The washed sand appears clean on visual observation. Figure 5 shows also that the remediation proceeds with fast kinetics over the first 3.5 minutes and then slows down. Thus, when dealing with industrial decontamination, the operation time is fixed with respect to economic considerations. There is no need to proceed with the washing over long times because the gain on TPH removal is relatively low after say 10 minutes. This behavior can be explained by the presence of surfactant in the solution at a concentration much over the CMC. The excess of micelles contributes to the mobilization and solubilization of contaminants just as pointed out by Han et al. (Han et al. 2009). Many other studies found consistent results (Urum and Pekdemir 2004; Fanaei et al. 2020; Olanami and Thring 2020).

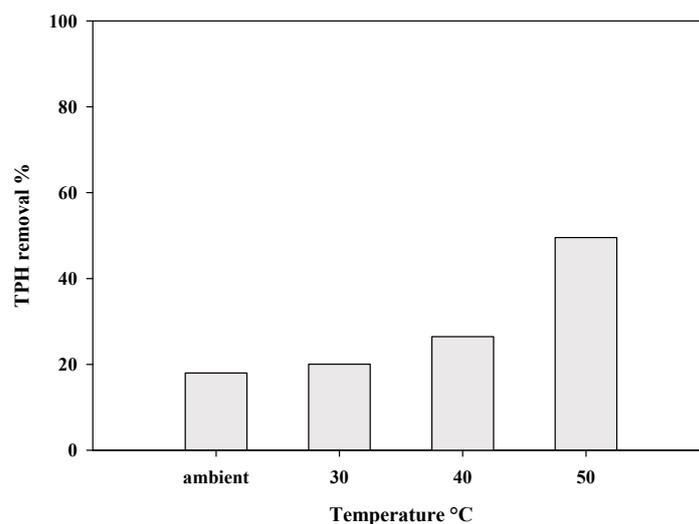


Fig. 4. Temperature effect on TPH removal in free water fluidization experiments

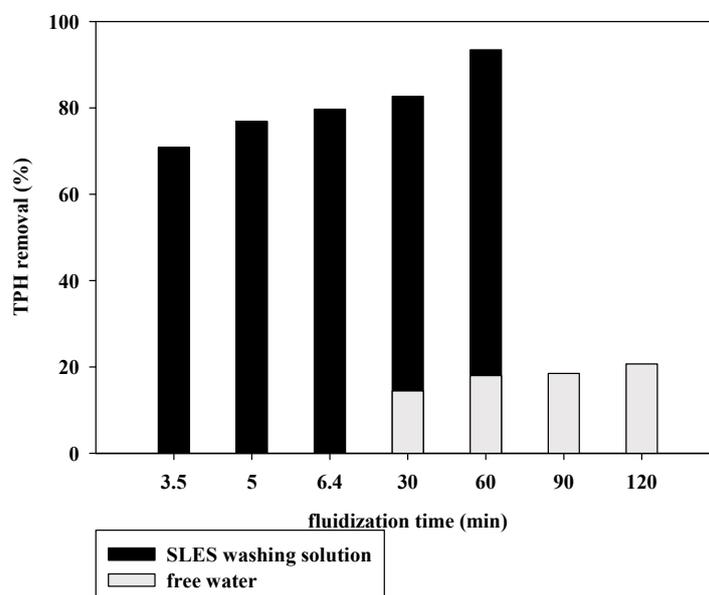


Fig. 5. The effect of fluidization time on TPH removal rate for free water and surfactant solutions

Effect of operation mode

During experiments, it was observed that when the fluidization was suddenly stopped, soil particles settle, and a thin layer of dark brown droplets appears on its top. By restarting the pump at its maximum flow rate (0.8 l/min), a good mixing was created leading to the release of the oil droplets. The high collision rate between particles contributes, with the surfactant action, to the enhancement of oil desorption from the surface of soil particles.

The quantitative analysis of the effect of operation mode on the depollution was investigated over a series of washing experiments using at each test 4 liters of SLES solution (25 CMC). The operation mode in each experiment was a succession of shutdowns and restarts of fluidization, at fluidizing/stopping time ratios of 3/1, 2/1, and 1/1 (minutes/minute). This leads to 1, 2, and 4 stops respectively. The results of the TPH removal ratio are reported in figure 6. By increasing the number of stops/restarts, the TPH removal increases from 70.85% to 84.63%. This is explained by the fact that by a sudden restart at the maximum velocity, the medium soil particles move to the top of the bed colliding with the fine particles creating a sort of good mixing before the segregation dominates again. This again shows the important effect of mixing on the TPH removal efficiency. Indeed, the ANOVA results ($P = 0.021$) indicate a significant effect of the working/stopping mode on the TPH removal ($P < 0.05$).

In order to verify this explanation, a non-stop fluidization experiment was conducted for 30 minutes, adding (from the top of the column) 50 g of clean medium sand (250 μm sieve diameter) every 5 minutes. In total, 300 g of clean sand was added to the bed. The added soil collides with all the contaminated soil particles before it settles to the bottom layer of the bed through segregation. The TPH removal ratio measured at the end of the test was 92.5% which is much greater than that observed at the same operating conditions (section 3.4.1) without adding fresh sand (70.85%).

Furthermore, the value of the TPH removal ratio obtained during 10 minutes at fluidizing/stopping times ratio of 1/1 (84.63%) is in the same order of magnitude as that obtained in the same operating conditions with continuous fluidization during 30 minutes (82.67%). This indicates that the use of

fluidizing/stopping operation mode significantly reduces the processing time and permits lowering of operation costs.

Effect of surfactant concentration

The effect of surfactant concentration on TPH removal was tested over a series of experiments performed in fluidizing/stopping mode with 5/1 min cycles. Each test was performed at ambient temperature for 30 minutes using solutions at different concentrations corresponding to 1, 2, 3, 5, 10, and 25 CMC. Results in Figure 7 show that initially the TPH removal ratio increases with surfactant concentration and then stabilizes around 96% at 10 CMC. Moreover, by visual observation, the washed sand seems almost clean. This behavior can be explained by the fact that in the presence of surfactant, the petroleum hydrocarbons released are trapped into the hydrophobic core of the micelles formed at concentrations higher than the CMC. The more the concentration of surfactant is, the more is the number of micelles in the solution. This leads to an increase in both solubility and desorption of hydrocarbons from the soil particles into the water. In other words, the flux of oil transferred from the solid phase to the liquid phase was enhanced. It attains its maximum at 10 CMC and no further desorption occurs at higher surfactant concentrations. The residual oil is strongly attached to the soil. However, it should be noticed that many factors must be taken into consideration when augmenting the surfactant concentration, for instance: surfactant adsorption into soil texture, toxicity, biodegradation, and economic aspects (Zhang et al. 2011; Fanaei et al. 2020)

The TPH removal rates recorded in this study are comparable to those reported by (Olasanmi and Thring 2020), where the remediation of a real contaminated soil with an initial TPH of 3276 mg/kg, with Rhamnolipid washing, gave a maximum of TPH removal of 58.5%. Gitipour et al. 2015b reported results of the same magnitude as those presented in this study, for an initial contamination of sand and coarse soil of 1.2×10^3 and 2.2×10^3 mg/kg contaminated by aliphatic and aromatic compounds respectively. The authors used SDS (an anionic surfactant) washing and reported maximum removal ratios of 92.34 and 92.39%. For the same surfactant used in this study, (Hernández-Espriu et al. 2013) studied its effect on TPH removal among other surfactants, 71.27% was achieved.

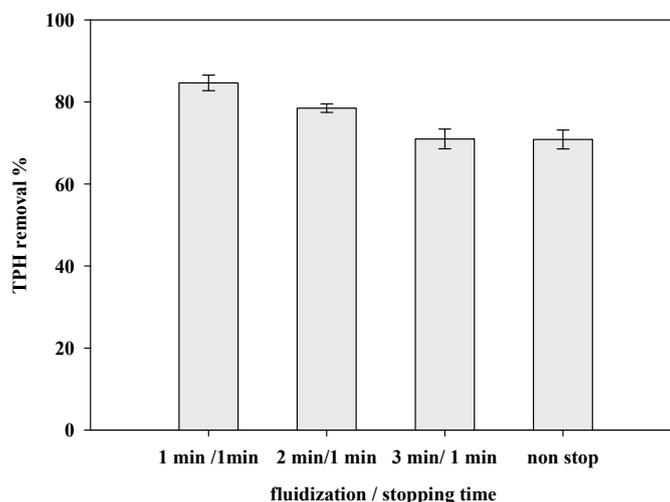


Fig. 6. Effect of fluidizing/stopping time ratio on TPH removal, using 5 g/l of SLES

Effect of temperature

The effect of temperature on the TPH removal was evaluated over four experiments, each performed during 30 minutes, in 5/1 min fluidizing/stopping mode using 1 CMC SLES solution. The four experiments were realized at 22, 30, 40, and 50°C respectively. Results are reported in figure 8, together with those performed using free water.

In contrast to the observations found using free water where temperature exerts an important effect on TPH removal, in the presence of SLES, the effect of temperature was minor. The TPH removal ratio increases from 70.2% at 22°C to 79.4% at 50°C. Indeed, the reduction of viscosity due to an increase of temperature leading to an increase of TPH removal was completely hidden by the effect of SLES which enhances the solubility of oil and reduces its surface tension.

Finally, the best operating conditions deduced from the whole study were selected to perform an ultimate washing test (temperature of 50°C, SLES concentration of 10 CMC, 30 min fluidization by 5/1 min fluidizing/stopping ratio). Obtained TPH removal ratio was 99.02%.

Washing solution contamination

Since the soil washing procedure consists of a displacement of contamination from soil to solution, it is important to examine the extent of residual contamination in solution after oil separation by decantation. This is done by measuring the Chemical Oxygen Demand (COD). A portion of the washing solution (at 4CMC) was collected and filtered, then the COD was spectrophotometrically measured using LCK 114 test kit (HACH, Germany). the results indicate high contamination of water estimated at 949 mg/l. This indicates that further treatment of the washing solution is necessary to achieve a sustainable process.

Concluding remarks

This study has shown that the remediation of aged hydrocarbon contaminated soil by washing in a fluidized bed is an efficient process under certain operating conditions. Results obtained using free water at ambient temperature has indicated low process efficiency, even after 120 minutes of operation, since

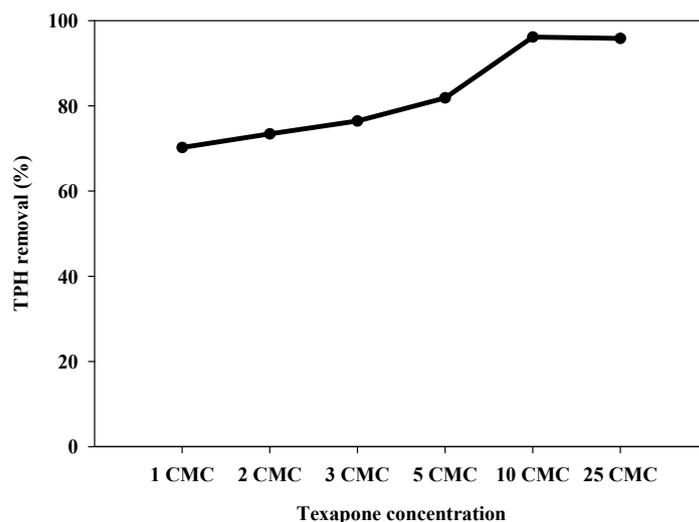


Fig. 7. Effect of SLES concentration on TPH removal at ambient temperature

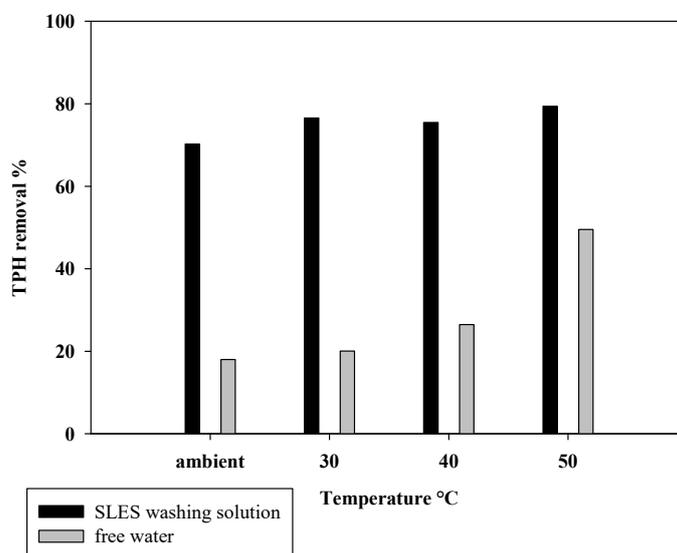


Fig. 8. Effect of temperature on TPH removal for free water washing, and SLES solution washing

the TPH removal ratio was only 20.69% (from 23.46 mg/kg initial contamination to 18.55 g/kg residual). Strong soil aggregation and channeling were observed leading to poor mixing. In such conditions, fluidization is not sufficient alone. The temperature of water exhibits an important influence since the TPH removal becomes approximately 50% at 50°C. This gives insight to perspective work based on solar energy testing further temperatures.

In the presence of SLES, high efficiency for TPH removal was observed. The use of the so-called fluidizing/stopping operation mode is recommended since it significantly reduces the operation time and solution volume. The temperature has little effect, and the optimum surfactant concentration was found to be at 10 CMC.

Because of the dependence of the soil washing efficiency on many soil properties (e.g. initial contamination degree, particle size distribution, pH, salinity, organic and inorganic matter ...) and washing conditions (e.g. liquid-solid contact type, temperature, type and concentration of surfactant, solution to soil ratio ...), the comparison with other studies performed without using fluidization is limited. Thus, further investigations needed to be done using different surfactants and biosurfactants testing the significance of each factor alone and the interactions between factors on TPH removal.

Despite the studied process and surfactant have given good results, it seems important to investigate the effects of other parameters such as the soil type, the fluidization velocity regarding the technique, the adsorption of SLES on the soil surface and the use of bio-surfactants. The promising results encourage the scale-up of the research to the industrial level with clean and sustainable resources.

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