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SURFACTANT ASSISTED REMOVAL OF ENGINE OIL FROM SYNTHETIC SOIL

Effectiveness of surfactants SDS (sodium dodecyl sulfate), AOT (sodium dioctyl sulfosuccinate) and Brij 35 (polyoxyethylene lauryl ether) in removing residual oil from soils have been determined. Experimental results indicated that oil desorption efficiencies with surfactants are 7 to 18 times higher than using water alone. 0.6% Brij 35 at was the most effective surfactant to remove oil from soil, and it did not display any significant change in oil desorption with pH changes. A comparison study also showed that pore volume was a more significant parameter than soil washing flow rate to improve oil desorption.

1. INTRODUCTION

Upward energy demands by society have increased the use of petroleum products such as engine oil. These lead to contamination of soils by oil spills and storage tank leaks. Only a fraction of free phase oil can be removed by pumping or drainage. A significant residual oil fraction gets trapped in soil pores or stays on soil particles. It may volatilize into the air or leak into groundwater and pose risks to human beings. Soil vapor extraction, soil flushing or soil washing, chemical treatment, bioremediation, thermal desorption and physical separation are several soil remediation procedures that reduce soil contamination caused by residual petroleum products. For stable petroleum mixtures such as engine oil, soil washing and soil flushing are effective management technologies. For the current study, commercial engine oil was chosen as the petroleum product due to its wide use and its greater stability in the soil.

In several studies, surfactants were shown to enhance remediation of contaminated soil and groundwater. They enhance the dissolution of residual non-aqueous phase liquids (NAPLs) into an aqueous phase by solubilization or by mobilization. Surfactants reduce interfacial tension between NAPLs and the aqueous phase and restore the

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mobility of trapped NAPLs in soils. At concentrations at or above the critical micelle concentration (CMC), surfactant molecules form micelles and allow NAPL to reside in them. Previous studies have shown that loss of surfactants to soil occurs in soil –water systems [1] and hence CMC is higher in these systems than in water alone.

Many earlier investigations, besides a few recent current studies [2–4] demonstrated the use of surfactants to enhance remediation of soil contaminated with organics. Zhu et al. [4] have made a detailed evaluation of surfactant concentrations during enhanced remediation of a site contaminated with diesel oil. Besides earlier studies, some current studies have also reported on the ability of surfactants to remove soil bound heavy metals [2, 6–9]. Shin et al. [2] and Khodadoust et al. [11, 12] have clearly demonstrated the use of soil washing fluids to remove mixed contaminants (organic and inorganic) present in the soil. Zhang and Lo [12] determined the optimum pH and surfactant concentration for the removal of organics (marine diesel fuel) from soils, using surfactant SDS and EDTA, in the presence and absence of lead. Surfactants are also shown to assist the uptake of heavy metals from soil bound mixed contaminants (heavy metals and engine oil) by Indian mustard plants [13]. Malefic et al. [14] presented a detailed study on to the impact of hydrocarbon (crude oil and diesel oil) concentration on the natural weathering process, when the pollutant is present in sandy soils. In their studies, evolved CO_2 served as a measure of oil biodegradation. In recent phytoremediation studies [13], the amount of CO_2 liberated was also used to assess surfactant enhanced degradation of engine oil in a sandy soil that contained mixed contaminants (engine oil and heavy metals). In a large scale test, Bianchi et al. [15] convincingly demonstrated the amelioration of very salty sediment that contained mixed contaminants (petroleum hydrocarbons and heavy metals) based on a novel three component phytobiodegradation system.

The desorption characteristics of engine oil in the surfactant–water–soil system was evaluated in the present study. Soil samples were a mixture of clean fine sand and montmorillonite. Anionic surfactants sodium dodecyl sulfate (SDS), sodium dioctyl sulfosuccinate (AOT) and non-ionic surfactant polyoxyethylene lauryl ether (Brij 35) were used as washing fluids. Effects of surfactant type, surfactant concentration, soil composition and pH on the removal of the petroleum product from the soil contaminated with engine oil were found based on batch tests. A few column tests were also conducted to examine the influence of flow rate on oil desorption from the soil. Effects of pH and the interaction of metal ions in the soil with respect to oil adsorption and desorption were also determined. Selected surfactants (SDS, AOT, Brij 35) are generally not toxic in terms of affecting public health and living part of soil (e.g., *Docusate sodium Material Safety Data Sheet* from ScienceLab.com, accessed in March 2013).

To avoid interference effects due to extraneous factors, tests were conducted under controlled conditions, using a contaminated soil whose contents were well defined. Ingredients in natural soils vary widely from site to site and may contain small quantities of other organic and inorganic pollutants. To get more accurate results about natu-

ral soils in field applications, it may be necessary to fine tune the tests and form soil samples that closely resemble site specific soils, by incorporating other soil characteristics in them. Lo and Yang [16] have used synthetic soil earlier to study adsorption desorption characteristics of heavy metals under controlled conditions.

2. MATERIALS AND METHODS

Batch tests were conducted to select an economical surfactant to remove engine oil from the contaminated soil. The sand, montmorillonite clay soil, as well as reagent grade surfactants SDS, AOT and Brij 35 were supplied by Fisher scientific. As most soil surfaces are negatively charged, cationic surfactants were not chosen for tests. The spectrometer Lambda-40-UV/VIS (Perkin Elmer Instruments) was used to find concentrations of engine oil. Photometric accuracy of the device was ±0.003 A[0]. The test soil was prepared by mixing clean sieved sand and montmorillonite clay soil. To form soil samples, 160 mg of engine oil (contaminant) was well mixed with 40 g of sandy soil. For gravimetric data, an electronic balance with a detection limit of 0.1 mg was used.

Soil samples. Soil samples were a mixture of uniformly mixed sand and montmorillonite clay with a ratio of 5:1 by weight. Both sand and montmorillonite soil were supplied by Fisher Scientific Inc. The size of sand used here corresponded to mesh 40–100 (420–149 μm of U.S. standard). The diameter of montmorillonite clay soil particles was around 63 μm. It contained nearly 70–90% fine sand, 0–30% silt, and 0–15% clay.

Table 1

Properties of sand and montmorillonite

Item	Sand	Montmorillonite
CEC, meq/g	0	1.05 [17]
pH in water	6.7	3.7
Particle size, μm	149–420	63
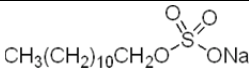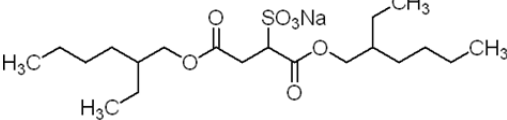
Table 2

Properties of engine oil

Property	Value
Density, kg/m^3	852
Kinematic viscosity at 40 °C, cSt	70.8
Molecular weight, g/mol	500
Specific gravity, d/cm^3	0.854
Flash point, °C	238

Table 3

Characteristics of surfactants

Product	Chemical structure	CMC [mg/dm^3]
SDS	$CH_3(CH_2)_{10}CH_2O-S(=O)(=O)-ONa$	2100[a]
AOT	H_3C...O...SO_3Na...CH_3 structure with O, CH_3, CH_3, H_3C	1066[b]
Brij 35	$CH_3(CH_2)_{10}CH_2(OCH_2CH_2)_{23}OH$	120[b]

[a]According to [5].
[b]According to [18].
[c]According to [19].

The soil was air dried for 24 h before usage. The characteristics of soil compo-nents are given in Table 1. The target contaminant was engine oil (Table 2). The en-gine oil was purchased from Canadian Tire (Table 2). Surfactant characteristics are given in Table 3.

Batch experiments. The sampling tubes containing artificially contaminated soil samples were thoroughly mixed in a shaker for 24 h. Contaminated soil samples were kept at room temperature for 24 h before usage to obtain adsorption equilibrium. Batch tests were conducted at 24±1 °C. A pH meter was used to measure pH. 1 M NaOH solution was used to adjust pH in the soil and surfactant system. The efficiency of surfactant enhanced oil desorption was determined for each test using the initial concentration and the final concentration of the oil in the sample after batch test. SDS, AOT and Brij 35 at various concentrations were used as washing solutions. Effects of soil composition, pH, surfactant concentration and surfactant type on desorption of oil from the soil were studied.

The ratio of sand to montmorillonite in clean soil samples was changed from 5:1 to 9:1 and 1:1 to 1:4 (by weight) in batch tests, to study the effects of soil composition on the desorption of engine oil from the contaminated soil. Only two surfactants (1.2% SDS and 0.6% Brij 35) were selected as washing solution to study the effect of soil composition on desorption of oil from the soil sample. In batch tests, contaminated soil samples (4.0 g) were weighed and placed in capped centrifuge tubes (50 cm^3). To each sampling tube containing the contaminated soil, 40 cm^3 aliquots of various sur-factant solutions ranging in concentration from 0.01% to 1.2% were added. The sam-ple tubes were placed on a wrist action shaker (60 oscillations/min) for 24 h to obtain desorption equilibrium. Following this, sample tubes were centrifuged at 1500 rpm for

30 min to separate solid and aqueous phases. The concentrations of oil in aqueous phase in the supernatant were measured using a Lambda 40 UV/VIS spectrometer at the wavelength of 275 nm. Batch tests were repeated three times to report average oil concentrations. Error bars in the figures denote the standard deviation for the test results. Distilled water was used as a washing solution in batch tests to compare oil removal efficiency with distilled water and with surfactants. As oil is almost insoluble in pure water, it is difficult to determine the oil concentration in water directly. A high performance liquid chromatography (HPLC) grade *n*-hexane was used as the organic solvent to extract the oil washed off by distilled water.

The mixture of contaminated soil sample and distilled water was placed in a centrifuge after desorption equilibrium was reached. After centrifugation, 20 cm^3 of supernatant was transferred into a clean centrifuge tube with a measuring pipette. 5 cm^3 of *n*-hexane was added to the supernatant (20 cm^3). The liquid mixture in the centrifuge tube was capped and shaken for 10 min to complete the oil removal with *n*-hexane. The oil concentration in the upper liquid layer (*n*-hexane) was measured after standardizing the UV device. Based on the volume of the upper liquid layer and the oil concentration in it, the oil volume was estimated.

Column tests. A few column tests were conducted to determine the effect of surfactant flow rate on enhanced removal of engine oil. The test soil was added to a Plexiglas column with the internal diameter of 4.0 cm and the height of 25.0 cm. The contaminated soil was added in small 2 cm increments to fill the column and tapped several times gently around the periphery with a wooden rod (1.27 cm in diameter) to ensure a homogeneous packed soil column. End disk filters set in the column ensured uniform entry and exit of the washing fluids. Only distilled water and Brij 35 (0.6%) were used in the column tests. A constant head reservoir ensured steady flow of the surfactant solution through the column. The flow rate was determined by collecting the effluent for a known period during which nearly 10 pore volumes flowed through the column at various flow rates. The oil in the column test effluent was extracted using *n*-hexane from the oil–water effluent during column tests. Based on the concentration of the oil in *n*-hexane, the amount of oil desorbed was estimated. The oil content in the water–hexane system was negligibly small. Blank tests were carried out in parallel to note any possible loss of the volatile hexane. Details of column test procedure are provided in related publications [3].

3. RESULTS AND DISCUSSION

Initial pH of surfactants before they were added to the soil samples and the plots of standardized curves developed for the UV analyzer are available in a related publication [6]. In batch tests, 160 mg of engine oil was mixed with 40 g of clean sandy

soil. 4g of the contaminated soil was mixed with 40 cm^3 of the washing solution (surfactant). The oil removal efficiency was calculated by dividing the amount of oil desorbed from solid phase into the aqueous phase by the amount of initial oil present in soil samples. The former is the product of the oil concentration in the aqueous phase and the volume of the washing solution. The oil concentration in the aqueous phase was measured 3 times for each sample.

3.1. EFFECTS OF SURFACTANT CONCENTRATION AND ITS TYPE ON OIL DESORPTION

Desorption efficiency is the ratio of oil desorbed from the soil sample to the initial amount of oil present in the sample. Results for oil desorption by the three selected surfactants tested at various concentrations are shown in Figs. 1–3. The error bars shown denote standard deviations which were generally lower than 5%. For samples with oil concentration higher than 150 mg/dm^3, the measurements were more accurate and hence the standard deviations were lower as one would expect. They ranged from 3% to 1%. Results of oil desorption from the soil enhanced by surfactant SDS are shown in Table 4 and Fig. 1.
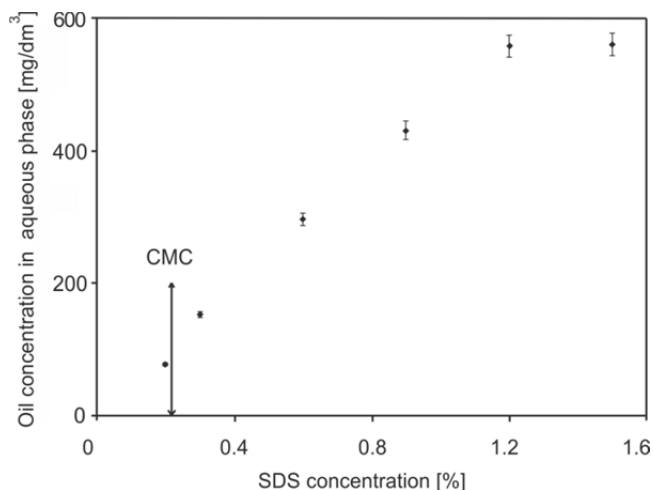

Fig. 1. Desorption of engine oil by SDS

Table 4

Oil desorption efficiency for surfactants

Surfactant	Optimal concentration [wt.%]	Desorption efficiency [%]
SDS	1.2	14.7%
AOT	0.6	7.7%
Brij 35	0.6	18.4%

They indicate that the oil concentration in the aqueous phase increased with the concentration of surfactant. Near SDS concentration of 1.2%, the oil desorption efficiency was close to the maximum oil extraction. The efficiency did not enhance further significantly with further increase in the concentration of SDS. From cost considerations, SDS concentration of 1.2% could possibly be considered as the optimal concentration for surfactant SDS to remove the oil from soil samples. The vertical line in Fig. 1 denotes SDS concentration at CMC in water.
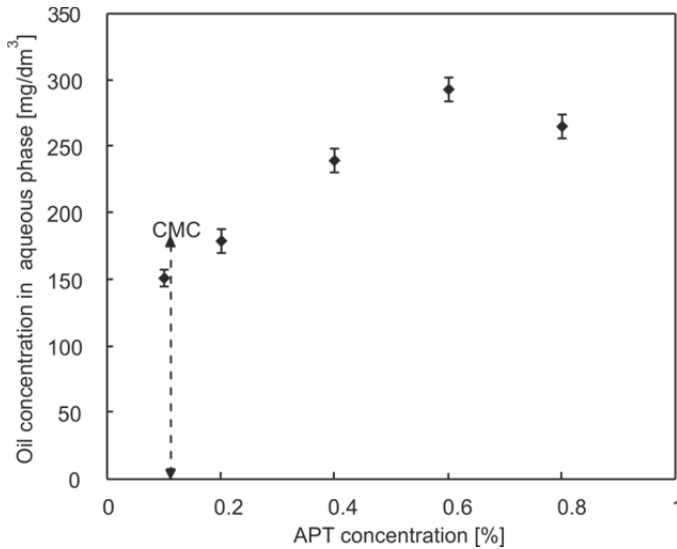

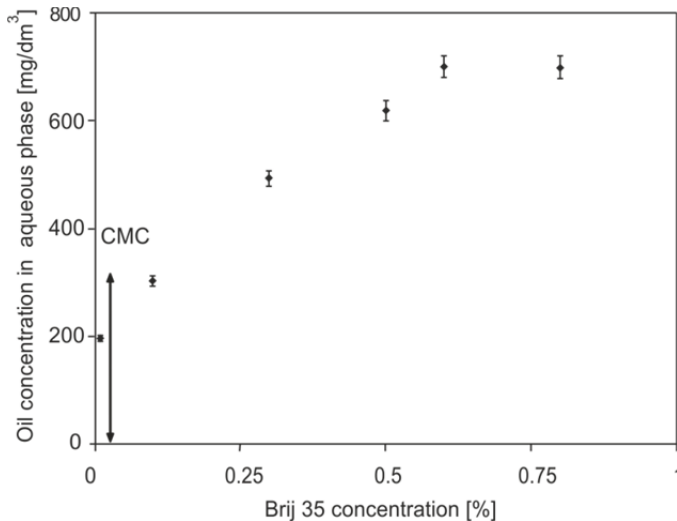Fig. 2. Desorption of engine oil by AOT

Fig. 3. Desorption of engine oil by Brij 35

Figure 2 shows that oil desorption efficiency does not increase with the concentration of AOT when its concentration exceeds 0.6%. The desired concentration of Brij 35 to desorb the maximum engine oil from the contaminated soil sample is noted to be 0.6% (Fig. 3). Figures 1–3 demonstrate that the concentration of oil removed into the aqueous phase tends to be higher with increased surfactant concentrations above their CMC. The existence of micelles noticeably promotes the solubility of oil in the aqueous phase. CMC values in water for SDS and AOT are 2100 and 1065 mg/dm^3. Generally, when cost factors are comparable, a surfactant with a lower CMC is preferred in the oil removal process, as less of the surfactant is needed for oil desorption. The batch results of the two ionic surfactants demonstrate that surfactant SDS achieved a better desorption efficiency of oil removal from contaminated soil than AOT. In the tests, pH range of the soil solution varied from 4.3 to 5.0. The acid environment of surfactant AOT may have decreased its effectiveness in desorbing oil compared to SDS. One notes that loss of surfactants occurs in the soil media, as part of the surfactant gets adsorbed onto the soil particles when it is added to the soil sample [22]. More pertinently, the difference in removal efficiencies of surfactants SDS and AOT may be attributed to their chemical configuration (Table 3). The hydrophilic functional group $(NaSO_3^-)$ of AOT is in the middle of its hydrophobic chain and the hydrophilic functional group of SDS is at the end of its hydrophobic chain (Table 3). Eeffectiveness of a hydrophilic functional group in promoting removal of oil from soil sample decreases as the functional group position is moved starting from the end towards the middle of its hydrophobic chain as in AOT [20]. Table 4 summarizes the batch test results of oil desorption from the contaminated soil by the 3 surfactants near their optimal concentrations. At equilibrium, the concentration of the aqueous phase oil which is desorbed by distilled water alone is 38 mg/dm^3 and the desorption efficiency is 1%. Figures 1–3 show that the highest aqueous concentrations of engine oil desorbed by SDS, AOT and Brij 35 were 558 mg/dm^3, 292 mg/dm^3 and 700 mg/dm^3 respectively. The corresponding desorption efficiencies of oil are hence 14.7, 7.7 and 18.4 times higher than that for distilled water. The optimal washing concentrations for the surfactants occurred at specific concentrations well above their CMC in water. One notes that the non-ionic surfactant Brij 35 shows a better oil desorption efficiency than the two anionic surfactants. That may possibly be traced in part to the fact that the non-ionic surfactant is not influenced by the ionic interactions of the soil solution.

3.2. EFFECT OF SOIL COMPOSITION

A part of the study was to examine the effect of soil composition on engine oil desorption from the contaminated soil. Four types of different soil compositions (sand plus montmorillonite mixtures) were prepared and contaminated with oil. The compositions expressed as ratios of sand to montmorillonite clay soil of the four prepared soil samples (Tables 5 and 6) were 9:1; 5:1; 1:1; and 1:4. The soils were washed using

only two surfactants SDS and Brij 35 at their optimal concentrations of 1.2 wt. % and 0.6 wt. %, respectively. Oil spiking of the soils was the same as described earlier for the other batch tests. The oil desorption results with SDS and Brij 35 are given in Tables 5 and 6, respectively.

Table 5

Oil desorption on 1.2% SDS from samples 1–4

Item	Sample No.			
	1	2	3	4
Sand:montmorillonite	9:1	5:1	1:1	1:4
Oil concentration, mg/dm^3	130	558	152	79
Oil desorption efficiency,%	36.6	14.7	4.0	2.1

Table 6

Oil desorption on 0.6% Brij 35 from samples1–4

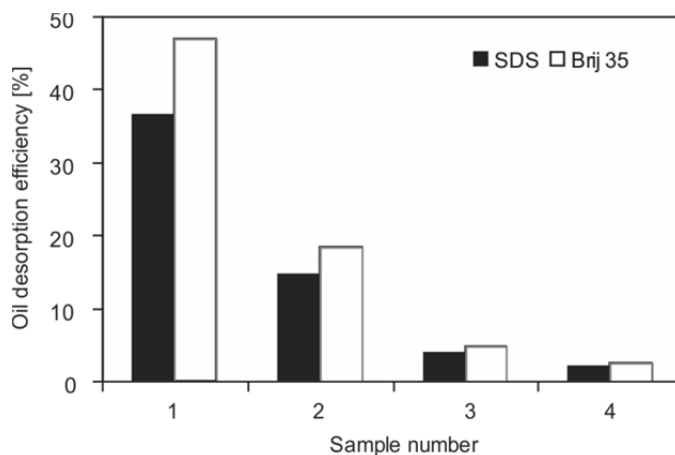| Item	Sample No.			
	1	2	3	4
Sand: montmorillonite	9:1	5:1	1:1	1:4
Oil concentration(mg/dm^3)	1780	700	182	95
Oil desorption efficiency (%)	46.8	18.4	4.8	2.5

Fig. 4. Effect of soil composition on oil desorption by SDS and Brij 35

Oil is adsorbed mainly by van der Waals forces and not through ionic interaction with the soil particles. Figure 4 shows that engine oil desorption efficiency is en-

hanced as the ratio of sand in the soil increases indicating that the non-polar oil mole-
cules interact mainly with the surface of montmorillonite and not that of sand parti-
cles. The results confirm the fact that sandy soils are more suitable for surfactant re-
mediation than clay soils, since clay sorption reduces surfactant's effectiveness in oil
desorption [21]. For the same soil composition, the oil desorption efficiency by the
non-ionic surfactant Brij 35 is better than the anionic surfactant SDS for all soil com-
positions tested.

3.3. EFFECT OF RELATED FACTORS ON OIL DESORPTION

The original range of pH of soil solution indicated that it was weakly acidic
(4.3– 5.0). 1 M NaOH was used to keep pH near the neutral range of 6.7–7.0. The
surfactants used here were 1.2% SDS, 0.6% AOT and 0.6% Brij 35 and the volume in
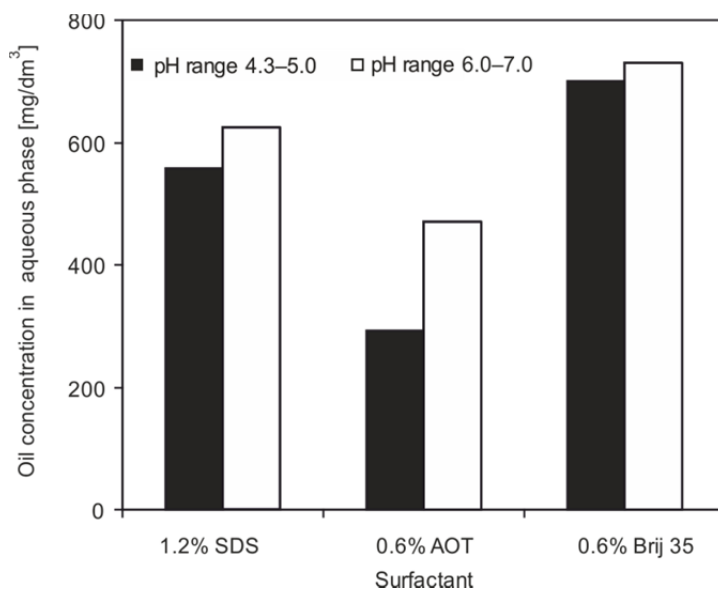each case was 40 cm^3, respectively.

Fig. 5. Effect of pH on oil desorption by surfactants

The results (Fig. 5 and Table 7) show that pH did not affect the non-ionic surfac-
tant Brij 35 treatment significantly in oil desorption. Lower pH appears to decrease oil
removal efficiency of anionic surfactants, especially in the case of AOT. Non-ionic
surfactant is not involved in the ionic interactions in the solution, while the anionic
surfactants could be affected by the level of H$^+$ concentration in the system.

T a b l e 7

Effect of pH on oil desorption

Parameter	pH	Oil concentration [mg/dm^3]	Oil desorption [%]
1.2% SDS	4.3–5.0	558	14.7
	6.7–7.0	625	16.4
0.6% AOT	4.3–5.0	292	7.7
	6.7–7.0	470	12.4
0.6% Brij 35	4.3–5.0	700	18.4
	6.7–7.0	729	19.2

The quantity of washing fluid used for all column tests was 10 pore volumes. At the flow rate of 10 cm^3/min, a very small amount of oil is desorbed with distilled water compared to the amount desorbed with Brij 35 at various flow rates (Fig. 6).
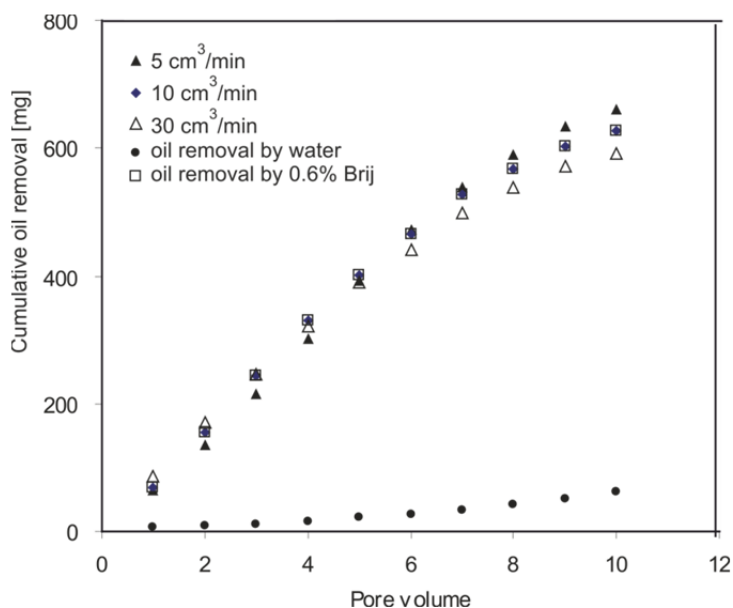

Fig. 6. Effect of flow rate on oil desorption in column tests
(oil removal versus the number of fixed pore volume)

The amount of oil desorbed at a higher flow rate of 30 cm^3/min appears to be only slightly smaller than the desorption rate at 10 cm^3/min. This indicates that the role of fluid flow shear in removing the oil from the surface of soil particles is not significant and that pore volume is more prominent parameter than the flow rate for oil desorption by Brij 35 (0.6%).

4. CONCLUSIONS

Batch tests indicated that all three surfactants used in this study enhance the removal of engine oil from sandy soils. The efficiency of desorption of engine oil from the soil was determined to be high and can be ranked as follows: Brij 35 (18.4%) > SDS (14.7%) > AOT (7.7%). Compared to distilled water alone (1%), desorption efficiencies with surfactants are almost 7–18 times higher. Batch tests also showed that the non-ionic surfactant Brij 35 at the lower concentration (0.6%) was the optimal surfactant for oil removal among the three surfactants tested. Batch test results also indicated that the best concentrations for SDS, AOT and Brij 35 to desorb oil from the same contaminated soil were 1.2%, 0.6% and 0.6%, respectively. If the unit prices of these three surfactants are somewhat similar based on a cost perspective, the surfactant that has the highest oil desorption efficiency at the lowest concentration could be considered the optimal one. Soil composition can affect the desorption of oil from contaminated soils. From the batch test results, it was readily observed that surfactant enhanced oil desorption form soil was significantly less effective with higher montmorillonite content in the soil. For the soil with the lowest clay content (sand: montmorillonite equal to 9:1), the oil desorption efficiency with 0.6% Brij 35 was 46.8%. For the soil with the highest clay content (sand: montmorillonite equal to 1:4), the oil desorption efficiency with 0.6% Brij 35 was 2.5%. The results confirm the fact that sandy soils are more suitable for surfactant remediation than clay soils, since clay sorption reduces surfactant's effectiveness in oil desorption.

Batch tests also demonstrated that pH of the soil environment affects surfactant enhanced oil desorption from the contaminated soil. Desorption of oil by anionic surfactants SDS and AOT was more affected by pH than desorption by the non-ionic surfactant Brij 35, because Brij 35 is not involved in ionic interactions with soil particles.

Column tests indicated that slightly more oil desorption from the soil occurs at lower flow rates linked to higher residence times that favour oil removal from the inner pores of soil particles. This indicates that the role of fluid flow shear in removing oil is not very significant and the pore volume is a prominent parameter than the flow rate for oil desorption from the soil by surfactant Brij 35 (0.6%).

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