

Sorption Characteristics of Nonionic Surfactant Triton X-100 in Soil Contaminated with Diesel

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1. INTRODUCTION

Petroleum and its derived products are considered to be increasingly menacing pollutants of our environment. These natural products are introduced to the environment due to various anthropogenic activities, such as accidental spills from transportation processes, leaking underground storage tanks, and poor waste disposal practices.

These compounds are commonly found in soil groundwater aquifers in industrialized areas. Some special classes of petroleum hydrocarbons are commonly referred to as non-aqueous phase liquid (NAPL), they are difficult to recover from the subsurface system and represent a long-term source of soil and aquifer contamination [Lee, 1999; Taylor, 1999].

Limited solubility of petroleum hydrocarbons is a major constraint over biodegradation of these compounds. The soluble portion will be degraded at faster rates while less soluble or insoluble compounds will have limited bioavailability thus reducing the degradation rate of these compounds.

Petroleum hydrocarbons are recalcitrant contaminants in the natural environment that make rehabilitation of contaminated sites difficult. In addition to their low aqueous solubility, petroleum hydrocarbons have high interfacial tension and a tendency to sorb onto soil particles. The high interfacial tension results in large capillary forces that resist washing by water. One of the most common types of groundwater contamination is from spills of petroleum based fuels and solvents from underground storage tanks.

When the problem of subsurface contamination begins, the initial remediation approach involves removing contaminated soil and pumping the contaminated water for treatment. Pump-and-treat remediation technology was initially prescribed for cleanup of subsurface contamination from both organic and inorganic contaminants. It is the most commonly used in situ remediation technology for contaminated aquifers [Liu and Roy, 1992].

Other conventional treatment technologies for soil contamination involve landfill disposal and incineration are applied. With landfill disposal only the location of pollutants is changed and the future liability is delayed. And so their complete destruction is not achieved. Incineration of hazardous wastes is both costly and difficult to implement.

It has been known that surfactants are able to improve the mass-transfer of hydrophobic pollutants from solid or non-aqueous liquid phase into aqueous phase by decreasing the interfacial tension and by accumulating the hydrophobic compounds in the micelles [Li and Chen, 2002; Tiehm, 1994; Volkering et. al, 1995]. Therefore, surfactants have been extensively studied in recent years for enhancing the subsurface contaminants remediation [Liu et. al, 1991; Zheng and Obbard, 2002; Meeren and Verstraete, 1996].

Surfactant enhanced subsurface remediation is one of several technologies being developed for remediation of subsurface non-aqueous phase liquid NAPL contamination. The removal of total petroleum hydrocarbons TPH was increased by 60% in the presence of surfactants compared to water only [Peters, et. al, 1992].

With surfactants, more hydrophobic contaminants, including the sorbed and entrapped ones, are mobilized in the aqueous phase. It is an emerging technology that has been demonstrated by a few pilot scale applications to date, but currently is the focus of wide-spread research and optimism. Economic analyses indicate that this technology can be competitive with conventional pump-and-

treat, landfill and incineration remediation if surfactant losses can be minimized, contaminant elution is maximized and surfactant-contaminant separation and surfactant reuse are implemented [Sabatini, 1995].

The aim of this research is to study the effect of nonionic surfactant in enhancing the remediation of insoluble hydrophobic organic compounds by solubilization and mobilization mechanisms, which in turn increase the bioavailability and hence the biodegradability of petroleum hydrocarbons. To study the effect of sorption characteristics of surfactant on soil at different amounts of diesel in term of adsorption isotherms, and the effect of these factors (i.e. soil and diesel) on the CMC value of the surfactant and interpretation of these effects in term of sorption behavior. To study different factors that may positively or negatively affect the sorption behavior of surfactant onto soil.

2. MATERIALS AND METHODS

2.1 Surfactant Analysis and CMC Determination

The surfactant used to prepare surfactant solutions was nonionic surfactant, Triton-X 100 purchased from Segma Aldrich Company. It was used as received at 97% purity. Surface tension measurements were used to determine the CMC of the surfactant. Surfactant solutions were prepared at ten different concentrations by diluting stock surfactant solution. The selected surfactant solutions as percent volume by volume were 2.0, 1.0, 0.5, 0.1, 0.05, 0.01, 0.005, 0.001, 0.0005, and 0.0001.

These concentrations range approximately from below CMC to above CMC. Surface tensions of surfactant solutions were measured by Fischer Surface Tensiometer Model 21 and the procedure of measuring surface tension by ASTM D 1331-89 (Reapproved 1995) "Standard Test Method for Surface and Interfacial Tension of Solutions of Surface-Active Agent". The room temperature while performing the surface tension measurement was recorded and it ranged between 19.5 and 21°C during the entire experimental phase. Each sample was tested at least four times to ensure that consistent values were obtained.

The averages from the measurements of each solution were plotted as a function of surface tension against the logarithm of surfactant concentration to identify the concentration at CMC. The surfactant concentration at which the surface tension no longer decreases significantly with increasing surfactant concentration is taken the representative surfactant CMC [Ku0, 1988].

2.2 Soil

Three replicates of agricultural soil were obtained from Al-Jeftlick, north of Nablus city, Palestine. Moisture content of each sample was determined by drying for 24 hours in Ari J. Levy oven at 500 °C. pH was determined by Jenway 3510 pH meter. Organic carbon and organic matter of each replicate was determined by Walkley-Black Titration Method. Nitrogen percent of each sample was determined by Kjeldahl determination method.

Each soil replicate was analyzed in order to evaluate moisture, the organic carbon percent, organic matter, and nitrogen percent.

Table 1 presents moisture content, pH, organic carbon percent, organic matter percent and Nitrogen percent of each sample.

After drying them each replicate was sieved and hydrometer test was performed in order to evaluate the soil texture of each replicate. Table 2 presents soil texture of each replicate.

2.3 Diesel Sorption onto Soil without Surfactant

The task of this experiment was first to study the sorption behavior of diesel in soil without surfactant. And second to determine the amount of diesel sorbed at different diesel concentration. Three mixtures were prepared by mixing 1 g of soil with 10 mL solution of diesel dissolved in methanol at three different concentrations; 0.25%, 0.50% and 1.00% (v/v).

The mixtures were shaken for two hours, and then left in hood for 24 hours for evaporation of diesel. 500 μL of liquid phase of each were taken and diluted with methanol in 10 mL volumetric flask, to test the amount of diesel remained after the completion of sorption process by UV-Visible spectrophotometer at 254 nm.

2.4 Surfactant Sorption onto Soil without Contaminant

The purpose of this task is to test the sorption behavior of surfactant onto uncontaminated soil. Seven mixtures, each one consists of 1 g of uncontaminated soil and 50 mL of surfactant solution with concentration 0.0005%, 0.001%, 0.005%, 0.1%, 0.5%, 1.0% and 2.0% (v/v) (i.e. each concentration for 1 mixture). The mixtures were shaken for 2 hours at 300 RPM then left for 36 hours for soil to settle. A 30 mL of the aqueous phase of each were taken for surface tension measurements.

2.5 Surfactant Sorption onto Contaminated soil at Different Diesel Concentrations

The purpose of this task is to determine the effect of diesel concentration on surfactant sorption onto soil. Sixty three samples were prepared in 250 mL Erlenmeyer flasks and separated into 3 groups for each soil replicate, 1, 2 and 3 (i.e. 21 samples for each soil replicate). The diesel concentrations were 0.25%, 0.50% and 1.0% (v/v) dissolved in methanol. Methanol was the preferred solvent because it showed no effect on surfactant solubilization; higher alcohols would affect CMC values significantly [Edward et. al, 1991].

Seven surfactant concentrations were used in this test, 0.0005%, 0.001%, 0.005%, 0.1%, 0.5%, 1.0% and 2.0% (v/v). The surfactant solutions used in this test were prepared and measured for surface tension.

In each group of soil, the samples were separated into three diesel concentrations, 0.25%, 0.50% and 1.0% (v/v).

The artificially contaminated soil was made by adding 10 mL of diesel dissolved in methanol to 1 g soil at different diesel concentrations. The artificially contaminated soils in the flasks were closed by Teflon screw caps and shaken for 2 hours to make sure that the contaminant was completely adsorbed on the soil. After shaking, the contaminated soil samples were opened and left in hood for 24 hours to evaporate the methanol. The amount of contaminant evaporated with methanol in each sample was assumed to be equal in every group samples. 50 mL of surfactant solution was added into the artificially contaminated soil after methanol was evaporated for 24 hours and 0.7 mL of mercuric chloride (corresponding to 18 mg) was added into each sample to inhibit the biodegradation process during the experiment. The samples were shaken for 2 hours and left to settle for 36 hours. The supernatants were taken from the samples and centrifuged for 15 minutes. 30 mL of the supernatant was placed in 100 mL beakers to measure the surface tension at room temperature, (it was between 20.5 and 22.5°C). The results from the surface tension readings were plotted versus the logarithm of surfactant concentration in mol/L.

3.0 RESULTS AND DISCUSSION

3.1 Surface Tension Measurements and CMC Determination

Surface tension measurement is a well-established method for determining the CMC of surfactants. The surface tension for each concentration of surfactant Triton X-100 was measured at least four times. The surface tension curve is composed of two linear segments; the intersection of the two linear portions represents the CMC. The surfactants concentration at which the two linear segments intersect represent the CMC [Zheng and Obbard, 2002].

From the result, the CMC point was found as the point (-3.47, 35.2) where the concentration of surfactant solution is 3.388×10^{-4} mol/L and the surface tension of the surfactant mixture 35.2 dynes/centimeter. The range of CMC determined by other researchers was from

6.89×10^{-5} to 3.3×10^{-4} mol/L [Liu et. al, 1991; Zheng and Obbard, 2002; Liu et. al, 1992; Zhao and Brown, 1996].

The variation in this number depends on the different techniques used to test CMC and the room temperature during measurements.

3.2 Diesel Sorption onto Soil without Surfactant

The experiment in this part of the research was to evaluate the amount of Diesel sorbed onto soil at the three concentrations of diesel, 0.25%, 0.50% and 1.00% (v/v). 500 μ L of solution of diesel dissolved in methanol was taken after the sorption process has equilibrated, and diluted with methanol in a 10 mL volumetric flask. The cell of UV-Visible Spectrophotometer was filled from the diluted solution and tested. The results were compared with calibration curve from UV-Visible Spectrophotometer. The comparison has shown that the amount of diesel sorbed on soil was ranging from 7% to 13% of the original amount. These amounts of sorbed diesel were slightly low; this probably was due to the low organic content of the soil itself which decreased the tendency of diesel to be sorbed onto it.

3.3 Surfactant Sorption onto Soil without Diesel

Results of this experiment have shown that the seven surfactant concentrations; 0.0005%, 0.001%, 0.005%, 0.1%, 0.5%, 1.0% and 2.0% (v/v) mixed with 1 g soil were slightly sorbed onto it. The surface tension of the supernatant of each sample mixture was measured and compared with surface tension of surfactant solution without soil to determine the amount of surfactant sorbed at each concentration; Q_g . Fig. 1 shows a plot of the surface tension reading versus surfactant concentration. Fig. 2 shows the comparison between surface tension measurement with and without soil. From fig. 2 we can calculate C_{sorb} and by the equations proposed Q_g can be determined [Liu et al, 1992]. It was observed that Q_g of surfactant was ranging 9-15% for all seven concentrations.

This small amount of sorbed surfactant may probably was due to the low organic content of soil itself. This conclusion may be attributed to the postulate that says "more organic content of the soil leads to more tendency of an organic substance to be sorbed on it".

3.4 Effect of Contaminant on Surfactant Sorption on Soil

The Experiments in this section were performed in batch mode. Diesel dissolved in methanol was used as a contaminant in soil at different concentration 0.25%, 0.50% and 1.00% (v/v) to study how the contaminant affect the amount of surfactant sorbed onto soil. The contaminant mixed with soil to produce an artificially contaminated soil and surfactant solutions were added and agitated for the sorption process to equilibrate. Then the aqueous phase was removed from the sample and the surface tension measurements were performed.

The average surface tension results for each sample of aqueous phase removed from contaminated soil at different diesel concentrations are shown in Table 3. The average surface tension readings from each sample were found for each soil at 0.25%, 0.50% and 1.00% (v/v) diesel concentration. The results have shown that, the presence of diesel with surfactant in solution together enhance the sorption for each onto soil; especially at surfactant concentration lower than CMC.

From the results of surface tension measurements, soil with 0.25% diesel reached CMC at (-3.315, 35.12) or the surfactant Triton X-100 dose 4.84×10^{-4} mol/L, soil with 0.50% diesel reached CMC at (-3.23, 36.31) or 5.89×10^{-4} mol/L and soil with 1.00% diesel reached CMC at (-3.11, 37.64) or 7.76×10^{-4} mol/L.

Fig. 3 shows the graph plotted to compare among the results of surface tensions in the presence of soil containing different diesel concentrations, 0.25%, 0.50% and 1.00% (v/v), and the surface tensions of surfactant solution without soil.

The data shows that the greater the diesel concentration in the soil, the greater the amount of surfactant needed to be added into the system in order to reduce the surface tension by a given amount. The amount of surfactant sorbed on soil at any aqueous phase surfactant concentration can be calculated by using the data from surface tension plots [Liu and Roy, 1992, Zheng and Obbard, 2002].

Referring to Fig. 3, the abscissa for a selected data point on the surface tension curve for the aqueous system without soil gives an aqueous-phase surfactant concentration, C_{surf} (-4.03 from Fig. 3 or 9.33×10^{-5} mol/L). The corresponding ordinate, a particular value of the surface tension, σ (43 dynes/cm), is then located on the surface tension plot for the soil/aqueous system.

The abscissa on this plot that corresponds with this value of σ yields a value for, $D_{s, \sigma}$ (-3.76 from fig. 3 or 1.74×10^{-4} mol/L) the bulk surfactant dose in soil/aqueous system that produces surface tension of σ in the supernatant. The difference between this value of $D_{s, \sigma}$ and the selected value of C_{surf} is equal to C_{sorb} (8.07×10^{-5}), the number of moles of surfactant sorbed per liter of solution, evaluated at the particular bulk solution surfactant concentration. The product of C_{sorb} and the ratio V_a to W_{soil} , the volume of the aqueous solution in liters divided by the weight of the soil in grams, yields a value for Q_{surf} (4.035×10^{-3} g/g of soil), the number of moles of surfactant sorbed per gram of soil or gram surfactant per gram of soil, the amount of surfactant adsorbed onto soil can be calculated [Dzomback and Luthy, 1984].

Surfactant sorption may also be expressed as Q_g , the number of grams of non-ionic surfactant sorbed per grams of the soil, using the Freundlich isotherm:

$$Q_g = K \cdot C^n \quad (\text{equ.4. 1})$$

Where K : a measure sorption capacity.
 $1/n$: an indicator of the curvature of the isotherm.

Table 4 shows the amount of surfactant sorbed per gram of soil contaminated with diesel at different concentrations calculated from the equations (4.1) and (4.2) by [Liu et. al, 1992]. This is the surfactant sorption at sub-CMC level and the surfactants sorbs onto the soil are in the form of surfactant monomers.

Fig. 4 shows number of grams surfactant sorbed per gram of soil at 0.25%, 0.50% and 1.00% (v/v) diesel. Using the Freundlich isotherm, the values of K and n were found.

At 0.25% diesel	$y = 2.3531x^{0.7775}$	$R^2 = 1$
At 0.50% diesel	$y = 13.138x^{0.8527}$	$R^2 = 1$
At 1.00% diesel	$y = 17.631x^{0.8448}$	$R^2 = 1$

However, in many environmental applications, the linear form of the Freundlich isotherm applies [Dzomback and Luthy, 1984]. For the linear adsorption isotherm, $1/n=1$. From the result, the values of n at 0.25%, 0.50% and 1.00% (v/v) diesel are close to 1. If the value of n assumed to be equal 1, the following linear equations are obtained:

At 0.25% diesel	$y = 14.972x + 0.0003$	$R^2 = 0.9579$
At 0.50% diesel	$y = 36.76x + 0.0014$	$R^2 = 0.8303$
At 1.00% diesel	$y = 50.488x + 0.0018$	$R^2 = 0.9066$

Table 5 shows CMC values at different levels of diesel, 0.25%, 0.50% and 1.00% (v/v). The CMC values increase as the diesel concentration increase. Since the higher diesel concentration tends to adsorb more surfactant monomers, the amount of surfactant monomer left to form micelle is reduced. Therefore, the CMC of the higher diesel concentration in soil is reached at the higher surfactant concentration.

According to other researchers [Liu et. al, 1991; Zheng and Obbard, 2002; Ku0, 1988], the smallest surfactant dose that corresponds to the minimum plateau values of surface tension for the soil/aqueous system gives, after subtracting the CMC and multiplying by the ration of V_a to W_{soil} , a specific value for Q_{surf} that is equal to Q_{max} , i.e. the maximum value of sorption for surfactant on that particular soil. From Q_{max} the effective CMC, CMC_{eff} can be estimated by surface tension technique [Zheng and Obbard, 2002] as the following:

$$CMC_{eff} = CMC + Q_{max} \left(\frac{W_{soil}}{V_{aq}} \right) \quad (\text{equ.4. 2})$$

Q_{max} is an important parameter in predicting surfactant solubilization of organic contaminant. From the data, the CMC_{eff} and CMC were determined. Therefore the amount of surfactant sorbed onto soil and Q_{max} can be calculated from equation (4.2). Table 5 shows the amount of surfactant sorbed onto soil in mol/L and by percent lost the amount Q_{max} at each diesel concentration. The amount of surfactant sorbed on soil are 19.6%, 33.9% and 49.9% and the Q_{max} are 2.96E-3, 2.24E-3 and 1.2E-2 g/g of soil, for the diesel concentration 0.25%, 0.50% and 1.00% (v/v) respectively.

The surfactant dose required for micelle formation in soil-water system (CMC_{eff}) is shown in Table 5, at different concentrations of diesel.

CONCLUSION AND RECOMMENDATION

The amount of surfactant Triton X-100 sorbed onto soil increased with increasing surfactant concentration. After the sorption process has been completed, the additional amount of surfactant added into soil/aqueous system was no longer sorbed by soil but formed micelles which play an important role in the solubilization of hydrophobic organic compounds. From the experiments performed in this study, it was observed that CMC_{eff} value increased with increasing diesel concentration in soil. In soil/aqueous systems with no contaminant present in the soil, the CMC_{eff} depends on the organic content of soil itself. The CMC of surfactant Triton X-100 in aqueous system without soil and contaminant was 3.88E-4 mol/L and CMC_{eff} values of soil/aqueous systems in the presence of diesel with concentration 0.25%, 0.50% and 1.00% (v/v) were 4.84E-4 mol/L, 5.89E-4 mol/L and 7.76E-4 mol/L respectively.

In this investigation, the amount of surfactant sorbed onto soil at the surfactant concentration below the CMC was calculated and the relationship between diesel concentrations the amount of surfactant sorbed on the soil was developed. In the soil/aqueous system, the Freundlich isotherm and linear equation could be applied to all three types of the soil with 0.25%, 0.50% and 1.00% (v/v) diesel.

Not only the CMC_{eff} value for each soil type is important but the amount of surfactant sorbed on each soil type is also crucial for utilizing surfactant to enhance soil remediation. The amount of surfactant sorbed on soil can be calculated as the percent surfactant lost due to sorption on soil and gram of surfactant sorbed per gram of soil at any surfactant concentration and also maximum gram of surfactant sorbed per gram of soil, Q_{max} . The amount of surfactant Triton X-100 sorbed onto soil with no contaminant present ranged from 9% to 15%. However, the amount of surfactant Triton X-100 tends to be sorbed more onto contaminated soil. Also, the amount of surfactant sorbed on the soils contaminated with Diesel ranged from

19.6% to 49.9%. These results indicate that the presence of organic contaminant may result in greater surfactant sorption onto soil, thereby further increasing chemical costs in possible surfactant-enhanced subsurface remediation applications.

Overall, the results of this study suggest that the addition of aqueous surfactant solutions to contaminated soils may facilitate the removal of organic contaminants from soil. However, surfactant losses due to sorption onto soil appear significant and may increase due to presence of organic contaminants.

Table. 2: Soil texture of the soil samples.

Soil texture	Replicate (1)	Replicate (2)	Replicate (3)
Sand fraction 2-0.06 mm (%)	12	10	11
Silt fraction 0.06-0.002 mm (%)	46	56	69
Clay fraction < 0.002 mm (%)	42	34	22

Table. 3: Surface Tension of Surfactant Solutions Mixed with Diesel Contaminated Soil at Different Concentrations.

% (v/v) diesel	Surfactant concentration (%) (v/v))	pH	Temp °C	Surface tension (dynes/cm)
0 % diesel	0.0005	6.08	21	63.2
	0.001	6.09	21	53.9
	0.005	6.06	21	44.3
	0.1	6.11	21	36.1
	0.5	6.32	21	35.2
	1	6.23	21	34.3
	2	6.42	21	35.9
0.25% diesel	0.0005	5.99	21.5	64.2
	0.001	6.02	21	62.3
	0.005	6.07	22	45.9
	0.1	5.93	20.5	37.1
	0.5	6.21	21	36.1
	1	6.13	21.5	34.2
	2	6.06	21.5	35.7
0.50% diesel	0.0005	6.08	22	65.8
	0.001	6.12	22	62.6
	0.005	6.01	21.5	54.1
	0.1	5.97	22	36.1
	0.5	5.86	20.5	37.5
	1	5.94	20.5	35.9
	2	6.11	21	38.3
1.00% diesel	0.0005	5.93	20.5	67.4
	0.001	5.87	20.5	64.2
	0.005	6.02	21	57.2
	0.1	6.07	20.5	38.1
	0.5	5.86	21.5	39.2
	1	5.91	21	37.8
	2	6.04	21	37.3

Table.4: Surfactant Sorbed onto soil at Different Diesel Concentrations in gm/gm soil.

Surface tension (dynes/cm)	Surfactant Concentration , (mol/L)				C _{sorb} (g surf/g soil)		
	Soil-Free Surfactant Solution	0.25% Diesel	0.50% Diesel	1.00% Diesel	0.25% Diesel	0.50% Diesel	1.00% Diesel
55	1.63E-5	3.76E-5	5.02E-5	6.18E-5	6.65E-04	1.06E-03	1.42E-03
54	1.99E-5	3.89E-5	5.62E-5	6.61E-5	5.93E-04	1.13E-03	1.44E-03
53	2.37E-5	4.07E-5	6.03E-5	7.59E-5	5.30E-04	1.14E-03	1.63E-03
52	2.63E-5	4.68E-5	6.76E-5	1.00E-4	6.40E-04	1.29E-03	2.30E-03
51	2.95E-5	5.13E-5	7.76E-5	1.23E-4	6.80E-04	1.50E-03	2.92E-03
50	3.19E-5	5.48E-5	8.32E-5	1.34E-4	7.14E-04	1.60E-03	3.19E-03
49	3.78E-5	6.03E-5	1.21E-4	1.48E-4	7.02E-04	2.60E-03	3.44E-03
48	4.17E-5	6.92E-5	1.45E-4	1.58E-4	8.58E-04	3.22E-03	3.63E-03
47	4.89E-5	7.76E-5	1.62E-4	1.91E-4	8.95E-04	3.53E-03	4.43E-03
46	5.81E-5	8.71E-5	1.78E-4	2.29E-4	9.05E-04	3.74E-03	5.33E-03
45	6.57E-5	1.00E-4	2.04E-4	2.88E-4	1.07E-03	4.31E-03	6.94E-03
44	7.64E-5	1.32E-4	2.40E-4	2.95E-4	1.73E-03	5.10E-03	6.82E-03
43	8.72E-5	1.45E-4	2.63E-4	3.16E-4	1.80E-03	5.48E-03	7.14E-03
42	9.96E-5	1.66E-4	3.09E-4	3.89E-4	2.07E-03	6.53E-03	9.03E-03
41	1.17E-4	1.91E-4	3.31E-4	4.07E-4	2.31E-03	6.68E-03	9.05E-03
40	1.43E-4	2.27E-4	3.73E-4	4.36E-4	2.62E-03	7.18E-03	9.14E-03
39	1.79E-4	2.63E-4	3.98E-4	4.89E-4	2.62E-03	6.83E-03	9.67E-03
38	2.01E-4	3.16E-4	4.37E-4	5.62E-4	3.59E-03	7.36E-03	1.13E-02
37	2.52E-4	3.72E-4	4.79E-4	6.31E-4	3.74E-03	7.08E-03	1.18E-02

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