

# EFFECTS OF CHEMICAL DISPERSANT ON OIL SEDIMENTATION DUE TO OIL-SPM FLOCCULATION: EXPERIMENTS WITH THE NIST STANDARD REFERENCE MATERIAL 1941B

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## ABSTRACT

As it is well established that application of chemical dispersant to oil slicks enhances the concentration of oil droplets and reduces their size, chemical dispersants are expected to enhance oil sedimentation if applied in coastal waters rich in suspended particulate matter (SPM) and if flocculation between chemically dispersed oil and SPM, which leads to formation of oil-SPM aggregates (OSAs), occurs readily. New laboratory experiments were conducted to establish a quantitative understanding of the process and to verify this hypothesis. This paper presents findings from experiments conducted using Standard Reference Material 1941b prepared by the National Institute of Standards and Technology, Arabian Medium, Alaska North Slope and South Louisiana crude oils, and Corexit 9500 and Corexit 9527 chemical dispersants. Results showed that OSAs do form with chemically dispersed oil. Oil sedimentation increases with sediment concentration and reach a maximum at a sediment-to-oil ratio of approximately 2:1 for most of the oils used. No obvious effect of chemical dispersant on oil sedimentation was measured for sediment concentration of 100 mg/L and higher. However, measured oil sedimentation was 3 to 5 times higher with chemical dispersant than with physically dispersed oil at low sediment concentration of 25 and 50 mg/L. UV epi-fluorescence microscopy showed that OSAs formed with chemically dispersed oil contain many oil droplets that are smaller than those trapped in OSAs formed with physically dispersed oil.

## 1. INTRODUCTION

Several studies have been conducted to understand the formation of oil-SPM aggregates (OSAs), which results from the aggregation between suspended oil droplets and suspended particulate matter (SPM) (Owens, 1999; Khelifa et al. 2002, 2005a; Muschenheim and Lee, 2002; Omotoso, 2002; Owens and Lee, 2003; Payne et al., 2003; Ajijolaiya, 2004; Ali, 2006, for a review). However, most of the studies were conducted with physically dispersed oils. Formation and fate of OSAs with chemically-dispersed oil are poorly understood. The few studies conducted on OSA formation with chemical dispersants showed conflicting results. Mackay and Hossain (1982) found that chemically-dispersed oil tends to associate less with mineral matter than naturally dispersed oil, while Guyomarch et al. (1999, 2002) measured high amounts of oil trapped in OSA when various oils and Inipol IP90 chemical

dispersant were mixed with clay minerals using WSL and IFP testing methods. Additional experiments performed in the Polludrome facility (the wave flume facility at CERDE) to validate the small scale (flask) tests showed that 30 minutes of wave action on a chemically-treated oil were sufficient to disperse the oil. Almost 80 % of this oil was trapped in OSAs and settled to the bottom of the flume (Guyomarch et al., 1999). Recent laboratory experiments on size and fractal-dimension measurements conducted by Sterling et al. (2004) showed that OSA form when artificially-weathered, Medium Arabian Crude oil is treated with Corexit 9500 and mixed with bentonite clay. The experiments were conducted in a shear reactor. In their recent study in a wave tank Li et al. (2007) reported that combined application of chemical dispersant and engineered sediment (kaolin) increased oil dispersion in the water column.

Theoretically, chemical dispersants are expected to affect formation and fate of OSA because: 1) application of chemical dispersant reduces the size of oil droplets and increases their concentration in the water column (Khelifa et al., 2007a); 2) chemical dispersants alter surface chemical properties of oil droplets and, hence, they may change the bonding forces making solid fines stick to oil droplets; and 3) smaller chemically-dispersed droplets require fewer solid fines in suspension to form negatively buoyant OSA than larger naturally-dispersed droplets. More importantly, in areas with high SPM, OSA formed with small and numerically-abundant sediment fines, chemically-dispersed droplets may be expected to sink to the seafloor because the small droplets get coated with large quantities of denser solid fines and thus increase their *in situ* density. Natural solid particles in marine waters commonly have specific gravities 2 to 3 times higher than the density of most crude oils. The conditions (concentration and type of sediment, oil type, dispersant-to-oil ratios, mixing energy, etc.) under which this process of oil transfer from water surface to the bottom occurs are not known. Recent laboratory and numerical simulations studies showed that a critical range of sediment concentration exists where formation of OSA with naturally-dispersed oil increases rapidly (Guyomarch et al., 1999; Khelifa et al. 2003, 2004, 2005b, 2007b; Ajijolaiya, 2004; Khelifa, 2005; Ajijolaiya et al., 2006; Ali, 2006). Within this range and at higher sediment concentration, sedimentation of dispersed oil is expected to increase rapidly also. No research has been conducted with chemically dispersed oil to this regard.

This paper aims to present a first part of a new laboratory data on OSA formation obtained with chemically dispersed oil with special emphasis on oil sedimentation. Specifically, following

the experimental procedure proposed recently in Khelifa et al. (2007b), new measurements on formation of sinking OSAs with the Standard Reference Material 1941b (SRM-1941b, prepared by the National Institute of Standards and Technology) and different types of crude and chemical dispersant are presented.

## 2. EXPERIMENTAL METHOD

The experimental method used in this study was described in details by Khelifa et al. (2007b). Briefly, the method includes several steps such as OSA preparation, isolation of sinking OSAs, oil extraction from sinking OSAs, and sediment filtration. Following established protocols (Khelifa et al., 2002, 2005a,b, 2007b; Stoffyn-Egli and Lee, 2002; Ajjolaiya, 2004; Khelifa, 2005; Ajjolaiya et al., 2006; Ali, 2006), OSAs were prepared using a reciprocating shaker at a constant shaking speed rate and stroke in a cold room at a controlled temperature of 15 °C. Erlenmeyer flasks (500 mL with silicone stoppers) containing 250 mL of sodium chloride solution at 33 ‰ salinity were used as reaction chambers. The solid phase (SRM-1941b sediment) was added dry to the seawater. The flasks were then shaken for 5 minutes before the addition of the oil phase. A pre-calculated volume of the test oil to deliver 50 mg of oil is added at the water surface using a syringe. For each test oils, the syringe was pre-calibrated to dispense 50 mg of oil. The flasks were then shaken (Figure 1) for a period of three hours and then left to settle overnight in the cold room at 15 °C.

After an overnight settling period, sinking OSAs (settled oil-sediment mixture) were carefully isolated from the reaction chambers (Figure 2). The mixture is then used for oil extraction using distilled-in-glass grade DCM and liquid/liquid extraction procedure. For each sample, the extraction was repeated three times. After the third extraction, the 0.45 µm filter (used in the sediment filtration step) was kept drying overnight in a fumehood. The filter was weighed to determine the mass of sediment trapped in sinking OSAs. The total oil content was measured by gas chromatograph using a flame ionization detector (GC/FID) method. Individual n-alkanes and the total GC-detectable petroleum hydrocarbons (TPH, defined as the sum of resolved plus unresolved aliphatic and aromatic hydrocarbons) were quantified by the internal standard (IS) method using the baseline-corrected total area of the chromatogram (Wang et al., 2004a,b,c). A 20 ppm o-terphenyl surrogate added to the samples prior to extraction verified extraction efficiency. The relative response factor used for determination of TPH was the average of the response factors of n-alkanes relative to the IS over the entire analytical range (see Khelifa et al., 2007b for detailed description of the experimental procedure).

Qualitative verification showed that the method captured the expected increase of oil sedimentation with sediment concentration (Figure 3). Results showed that the GC-FID measurement procedure captures both very small amounts of oil (few mg, Figure 3a) as well as the high oil concentrations obtained with high sediment concentration of 100 mg/L and higher (Figure 3c). For quantitative verification, a single combination of SRM-1941b and Alaska North Slope oil was repeated seven times (OSA preparation and all the extraction steps) using seven different Erlenmeyers. Very consistent results were obtained. The uncertainty is about 0.8 mg (2σ=9%) for TPH measurements and 2.4 mg (2σ=12%) for sediment mass measurement for 95 % confidence (Khelifa et al., 2007b).



FIGURE 1. REACTION VESSELS SET FOR SHAKING ON THE RECIPROCATING SHAKER



FIGURE 2 REMAINING OIL-SEDIMENT MIXTURE (SINKING OSAS ) AFTER EXTRACTION OF THE FLOATING MATERIALS AND WATER. SAMPLES READY FOR TRANSFER INTO SEPARATORY FUNNELS FOR EXTRACTION. NOTE SEDIMENT CONCENTRATION INCREASES FROM RIGHT TO LEFT.

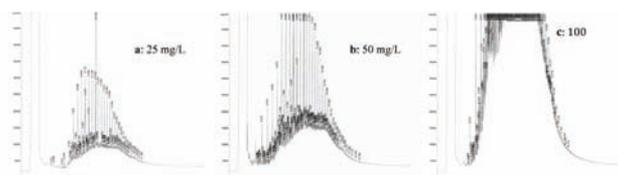
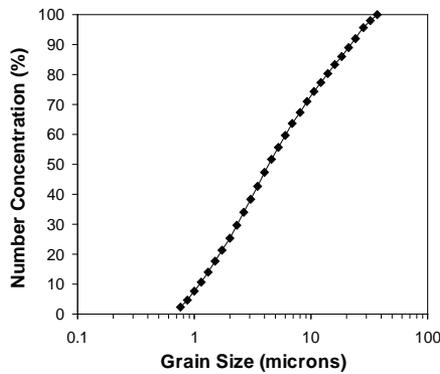


FIGURE 3 EXAMPLES OF GC-FID CHROMATOGRAPHS AT CONSTANT SCALE FOR OIL SAMPLES EXTRACTED FROM SETTLED OSAS PREPARED WITH ALASKA NORTH SLOPE AND SRM-1941 SEDIMENT AT VARIOUS CONCENTRATIONS, AS INDICATED ON THE FIGURE.

## 3. EXPERIMENTS

In this study, OSA formation was studied using SRM-1941b sediment and the preparation and extraction methods described briefly in section 2. The SRM-1941b sediment was prepared by the National Institute of Standards and Technology. Its preparation was based on sediment sampled from the Chesapeake Bay at the mouth of the Baltimore (MD) Harbor near the Francis Scott Key Bridge. The sediment was freeze-dried and sieved at 150µm. For this study, SRM-1941b sediment was dry sieved in our lab at 102µm. The sediment is rich in fine grains (Figure 4). About 56 % (number) of the grains are smaller than 5.4µm. The median grain size is about 5.2 µm. The sediment is also rich in organic matter, which represents about 11 % in weight.



**FIGURE 4.** MEASURED GRAIN SIZE DISTRIBUTION FOR THE SRM-1941B SEDIMENT.

Experimental conditions (Table 1) include three crude oils: Arabian Medium, Alaska North Slope and South Louisiana, and Corexit 9500 and Corexit 9527 for chemical dispersants. Sediment concentration was varied from 25 to 300 mg/L. The dispersant to oil ratio (in weight) was kept constant to 1:10

**TABLE 1.** EXPERIMENTAL CONDITIONS FOR OSA FORMATION

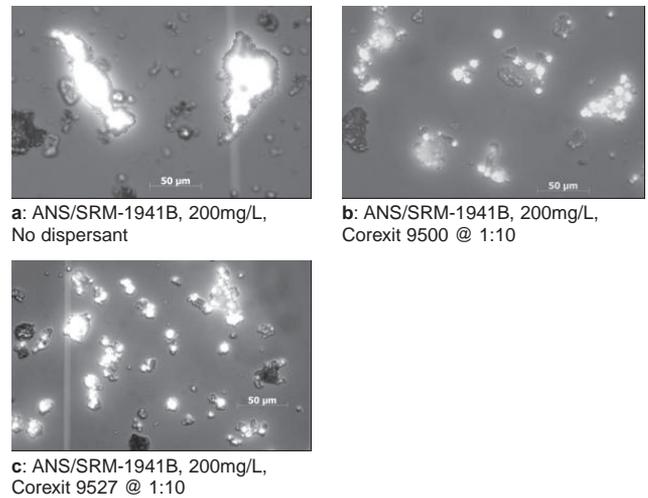
Reacting phase	Name	Density (g/mL)	Viscosity (mPa)	Surface tension (mN/m)	Oil-brine interfacial tension (mN/m)	Concentration (mg/L)
Sediment	SRM-1941b	2.57	-	-	-	25, 50, 100, 200, 300
Oil	Arabian Medium	0.876	28.1	26.5	20.0	200
	Alaska North Slope	0.873	17.0	26.4	19.0	
	South Louisiana	0.859	13.3	26.5	16.2	
Chemical Dispersant	Corexit9500	0.959	92.9	-	-	1:10*
	Corexit9527	1.002	67.5	-	-	

\* This concentration represents Dispersant-to-Oil Ratio in weight.

Direct observations of OSAs were performed using a UV-epi fluorescence setup equipped with a high resolution digital camera controlled by a computer (Figure 5). The setup is designed to measure droplet size of 0.1  $\mu\text{m}$  and larger. The observations reveal that OSAs do form readily with SRM-1941b sediment and the three oils with and without chemical dispersant. Examples of microphotographs of OSAs are shown in Figure 6. The data showed that OSAs formed with chemically dispersed oil may include several small droplets (Figures 6b,c).



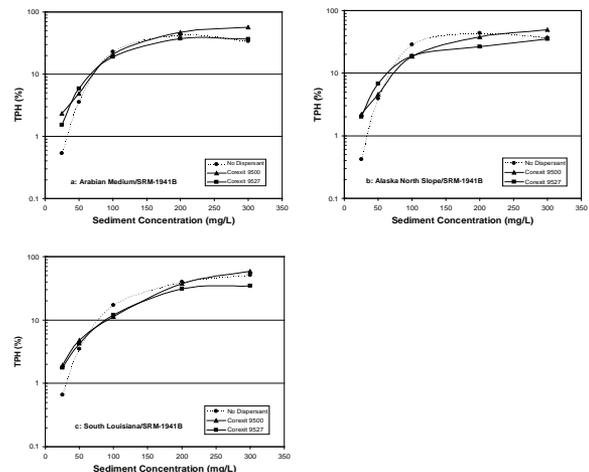
**FIGURE 5** IMAGING SETUP USED TO PICTURE OSAS USING UV EPI-FLUORESCENCE MICROSCOPY AND A HIGH RESOLUTION ZEISS DIGITAL CAMERA CONTROLLED WITH A COMPUTER. THE SETUP IS DESIGNED TO MEASURE DROPLET SIZE OF 0.1  $\mu\text{m}$  AND LARGER.



**FIGURE 6.** MICROPHOTOGRAPHS OF SINKING OSAS FORMED WITH ALASKA NORTH SLOPE OIL AND SRM-1941B SEDIMENT AT 200 MG/L WITHOUT CHEMICAL DISPERSANT (A), WITH COREXIT 9500 AT 1:10 RATIO (B) AND WITH COREXIT 9527 AT 1:10 RATIO (C). THE SCALES IN THE PICTURES ARE THE SAME.

#### 4. RESULTS AND DISCUSSION

Experimental results showed that OSAs form readily with both physically and chemically dispersed oils. Variations of oil concentration (or TPH) trapped in sinking OSAs presented typical trends for the three oils and the two chemical dispersants (Figure 7), as reported in previous studies (Guyomarch et al., 1999; Khelifa et al. 2004, 2005a,b; 2007b; Ajjolaiya, 2004, Khelifa, 2005; Ajjolaiya et al., 2006). The typical trend is characterized by a rapid increase in oil content when sediment concentration increases from 25 to about 200 mg/L. Further increase of sediment concentration does not increase significantly sedimentation of oil trapped in OSAs. This was expected as oil amount used in the experiments was kept constant to 50 mg.



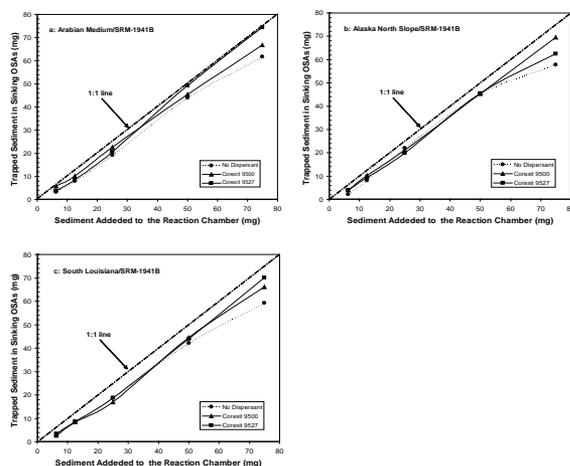
**FIGURE 7.** MEASURED TOTAL PETROLEUM HYDROCARBON (TPH) IN SINKING OSA FORMED WITH SRM-1941 SEDIMENT AT VARIOUS SEDIMENT CONCENTRATIONS AND CHEMICALLY DISPERSED ARABIAN MEDIUM CRUDE (A), ALASKA NORTH SLOPE CRUDE (B) AND SOUTH LOUISIANA CRUDE (C): COMPARISON WITH NO DISPERSANT CONDITIONS (PHYSICALLY DISPERSED OIL).

Overall, for the type of sediment and the three oils used in this study, application of the two type of chemical dispersant does not affect significantly oil sedimentation due to oil-sediment flocculation (Figure 7). However, it is interesting to note that oil sedimentation is 3 to 5 times higher with chemically dispersed oil than with physically dispersed oil at low sediment concentration (up to 50 mg/L). This trend is observed with both chemical dispersants and the three oils used in the study. This is possibly due to the fact that oil droplets formed with chemically dispersed oil are smaller and more abundant than those formed with physically dispersed oil. The probability for these small droplets to colloid with limited number (small concentration) of sediment grains becomes higher than with the large and less abundant physically dispersed droplets. This higher collision frequency between chemically dispersed droplets and sediment grains will produce a high oil sedimentation, as small oil droplets require less sediment grains to become negatively buoyant than large droplets.

For sediment concentration higher than 50 mg/L, the data showed a little difference in oil sedimentation obtained with and without chemical dispersant for both Corexit 9500 and 9527. The difference is within the uncertainty range. This observation is valid for the three oils (Figure 7). This finding is somewhat surprising and do not corroborate the hypothesis discussed in the introduction. Application of chemical dispersant was expected to increase oil sedimentation than in normal conditions. However, results shown in Figure 7 were obtained with the SRM-1941b sediment only (one type). This study is still ongoing. New results obtained with other types of natural sediments showed a different trend, as it will be discussed in subsequent communications.

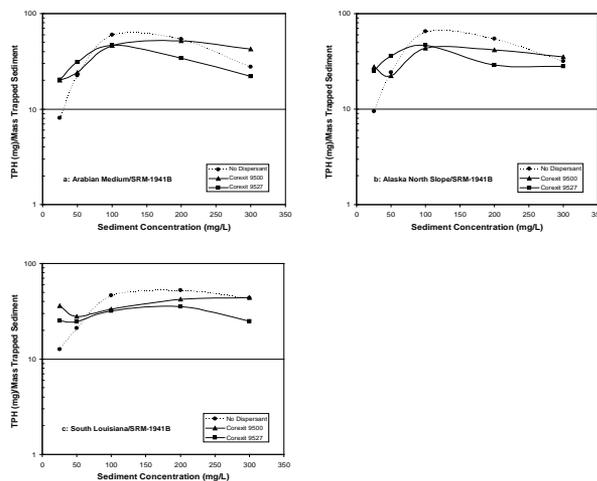
The small (or absent) difference observed between the results obtained with and without chemical dispersant at sediment concentration higher than 50 mg/L relates, possibly, to two factors. The first one is the high mixing energy used in this study and the second one is the abundance of fine grains in the SRM-1941b sediment. The high mixing energy helps formation of small oil droplets without chemical dispersant. The droplets are not as small as those obtained with chemical dispersant, but there are small enough to stay in suspension. This step of droplet formation is essential for OSA formation and, hence, oil sedimentation. If oil droplets are formed regardless of the application of chemical dispersant, the remaining key factor that controls formation of sinking OSAs is the abundance (concentration) of sediment fines in suspension. Several studies have shown that sediment fines less than about 5 microns in size are superior candidates for OSA formation (Ajijolaiya, 2004; Khelifa et al., 2004, 2005c; Ali, 2006). Results shown in figure 7 suggest that at sediment concentration higher than 50 mg/L (100 mg/L and higher), there are enough fines in the reaction chambers to sink the suspended oil droplets.

As discussed in section 3, sediment trapped in sinking OSAs was measured with care to evaluate the efficiency of OSA formation under the different conditions. Variations of the mass of sediment trapped in sinking OSAs are shown in Figure 8. All the curves showed linear (or almost) increase of the mass of sediment trapped in sinking OSAs with the amount of sediment added to the reaction chambers (or sediment concentration). The difference between the 1:1 curve represents probably the mass of sediment trapped in non-sinking OSAs. Positively and neutrally buoyant OSAs were observed visually and with the epi-fluorescence microscopy. The difference is slightly higher with the South Louisiana oil for sediment concentration up to 200 mg/L. At sediment concentration of 300 mg/L, the mass of sunken sediment is higher with application of chemical dispersant than without. However, the amount of sunken oil does not follow the same trend (Figure 7).



**FIGURE 8. VARIATIONS OF SEDIMENT TRAPPED IN SINKING OSAS WITH SEDIMENT CONCENTRATION, CHEMICAL DISPERSANTS AND OIL TYPES: ARABIAN MEDIUM CRUDE (A), ALASKA NORTH SLOPE CRUDE (B) AND SOUTH LOUISIANA CRUDE (C): COMPARISON WITH NO DISPERSANT CONDITIONS (PHYSICALLY DISPERSED OIL).**

Data shown in Figure 7 were normalized by the mass of sediment trapped in sinking OSAs shown in Figure 8 to study the trapping efficiency. The resulting variations are shown in Figure 9. Most of the curves presented maximum at which the formation of OSAs per unit mass of the trapping sediment is at its highest value. Presence of these maxima was reported by Khelifa et al., 2007b) and Guyomarch et al. (1999). Except for data obtained with South Louisiana oil, all the maxima are shown at sediment concentration of 100 mg/L, which in this study correspond to SOR of 2:1, regardless of application of chemical dispersant. As such, the equation proposed by Khelifa et al. (2007b) to predict the maxima (sediment concentration at which OSA formation is maximal) is also valid for chemically dispersed oils. The equation relates the maxima to the oil density and is expressed by “SOR at maximum OSR” =  $1367 \exp(-9.5 \rho_{oil} / \rho_{seawater})$ , where SOR represents the sediment to oil ratio in suspension and OSR the oil to sediment ratio in sinking OSAs.



**FIGURE 9. MEASURED VARIATIONS OF OIL (OR TPH) TO SEDIMENT RATIO (OSR) IN SINKING OSAS WITH THE CONCENTRATION OF SRM-1941B AND OIL TYPES.**

## 5. CONCLUSION

This paper presented a first part of an ongoing study that aims to verify the hypothesis that application of chemical dispersant in coastal waters may increase oil sedimentation due to oil-SPM aggregation. The findings revealed that: 1) chemical dispersant do not form a barrier to oil-SPM aggregation. OSAs formed readily under all the conditions discussed in this study. 2) Application of chemical dispersant did increase oil sedimentation by a factor of 3 to 5 than normal conditions at low sediment concentration (up to 25 mg/L). 3). However, no obvious effect of chemical dispersant was measured at sediment concentration of 100 mg/L and higher. As the third point does not corroborate the main hypothesis of this study, this finding was related to the facts that the experiments in this study were conducted with high mixing energy and a sediment type (SRM-1941b) that has high fine content. Verification of this explanation is expected from the findings of the ongoing experiments where three natural sediments (characterized by different fine contents) from three different locations are studied. Corresponding results will be discussed in subsequent communications.

## 6. ACKNOWLEDGEMENTS

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