Aerobic Degradation of Synthetic-Based Drilling Mud Base Fluids by Gulf of Guinea Sediments under Natural Environmental Conditions

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Abstract: Synthetic-based fluids (SBF), which are composed mostly of linear alpha Olephins, Esters and Paraffins are used in drilling mud to lubricate the drill bit, control reservoir pressure and bring rock chips and cuttings to the surfaces which are subsequently released into the marine environment as a residue on the cuttings as they are discharged. Aerobic biodegradation is a major criterion for selecting synthetic –based fluids for drilling mud. In the present study, sediments were collected from four different locations in the Gulf of Guinea measuring from 100-500m depth and were used in indoor basin benthic chamber tests to measure degradation rates of 4 different Ester based synthetic fluids at room temperature over a 120 day test period. At each 30 day interval, residual organic carbons were measured by gas chromatograph while microbial populations were quantified with the most probable plate number method (MPN). At the end of the 120-day monitoring period, the following % degradation rates were recorded for the different ester based fluids used in the study; BR-EST (94%), CH-EST (91%), PFB-009 (94.8%), PFB-008 (93.8%). This result indicate that the Ester based fluids used in the experiment are readily biodegradable and the Gulf of Guinea sediments harbour considerable populations of indigenous hydrocarbon utilizing microorganisms that are capable of degrading the exogenous ester based synthetic fluids. This study addressed the fate of the synthetic ester base fluid portion of the drilling mud in Gulf of Guinea sediments by determining the potential of indigenous marine sediment microbes to degrade representative SBF under natural conditions.

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1. Introduction

Three major types of cuttings can be defined depending on the drilling muds used to facilitate the boring process and also to carry the cuttings to the surface. They include:

- i. Water based muds containing for example KCl/Polymers or glycol.
- ii. ii. Pseudo-oil-based muds commonly comprising of olefins and Esters
- iii. Oil based muds comprising either clean mineral oil or in early stages, diesel.

The oil based muds are considered to have the most deleterious effect on the local environment especially diesel, so their use has been gradually phased out in some countries (Kjeilen *et al*, 1996). Alternative types of drilling fluids have been developed as a consequence of increasingly strong environmental protection legislation. The alternative drilling fluids have been designed to have less negative impact on the environment i.e., they are more easily degradable and less toxic than oil based drilling fluids. These alternative drilling fluids are pseudo oil based comprising mainly of Olefins, Esters and Paraffins and they have been proved to be very important in difficult deepwater drilling operations (Deborah and Alan, 2006). They also combine the technical advantage of oil base fluids and the low toxicity of water base fluids and are used mainly to lubricate the drill bit, control reservoir pressure and bring rock chips or cuttings to the surface.

Ester based fluids are well known for their high biodegradation potential but they are susceptible to calcium and acidic gas combination as well as thermal limitations (West et al, 2009), despite that, Ester based fluids deliver outstanding performance even under extreme bore hole and formation conditions (Tapavicza, 2005). Ester quality (EQ) stands for a new generation of drilling fluids, they are based on vegetable esters derived from natural raw materials like palm kernel oil. The overall benefits of Ester based drilling fluids include; faster drilling, reduced drilling costs, superior lubricity, excellent hole cleaning, protection of drilling formations and proven track record on performance (Tapavicza, 2005). Esters strongly protect the geological formations, preventing the swelling of the reactive clay and shale formations. The polar Ester groups and

the balanced vegetable C-chain are the main factors conferring these properties. The biodegradable Ester based muds are better alternatives to oil based muds because oil based muds are not environment friendly and often involve recovering and transportation of drill cuttings to onshore locations for treatment and disposal which is very costly. In contrast, vegetable ester drill cuttings can be safely discharged into the ocean without harming the ecosystem if they meet the local regulatory requirement, In Nigeria, the discharge limit is less than 50ppm of oil (DPR, 1991). When they are discharged, the cuttings and adherent synthetic base fluids settle to the sea floor and their concentration in the sea floor environment may decrease with time due to re-suspension, bed transport, bioturbation and biodegradation but biodegradation is expected to be the most significant mechanism of synthetic based fluid removal and subsequent environmental recovery (Deborah and Alan, 2006).

Aerobic biodegradation is a key criterion for selecting a base fluid for bioremediation though there may be other important factors such as availability of the base fluids, drilling environment and the operator's policy and local legislation. Numerous studies on petroleum degradation in marine environment and soil demonstrate that organic ingredients in oily cuttings are biodegradable under aerobic conditions (Prince, 1993, Kjeilen, 1997, Deborah and Alan, 2006) but in the floor of some sediments for instance the Gulf of Mexico, the average oxygen concentration is 6.8mg/L (0.21nm) and the oxygen only diffuses a few centimetres into the sediment, an indication that oxygen availability can be limiting in deep offshore sediments (Deborah and Alan, 2006). A wide variety of aerobic hydrocarbon degrading microorganisms have been isolated from the Gulf of Guinea sediments namely; Flavobacterium sp., Micrococcus sp., Alkaligenes sp., Corynebacterium sp., Aspergillus niger, Aspergillus fumigatus and Penicillium sp (Okoro, 2010a), in a related development, an investigation carried out by Okoro (2010_b) revealed that aerobic microorganisms are very active in the Gulf of Guinea sediment up to a depth of 2-5cm with total heterotrophic bacterial counts of 3.20 x 10^6 cfu/g and 2.20 x 10^4 cfu/g respectively. Other aerobic microorganisms implicated in hydrocarbon degradation in marine sediments by other researchers include; Pseudomonas sp. (Tagger et al, 1990), Flavobacterium sp. (Okpokwasili et al, 1984), and Vibrio sp. (West et al, 1984).

In the present study, the biodegradation potential of 4-Ester based drilling fluids (BR-EST,

CH-EST, PFB-009 and PFE-008) was tested under natural aerobic conditions using Gulf of Guinea sediments. The main objective of the study therefore is to determine the fate of these drilling fluids in the Gulf of Guinea sediments over time under natural aerobic environmental conditions.

2. Material and Methods: Experimental Design:

The experimental test set up consists of a series of 4 easily assessable rectangular shaped glass indoor basins called benthic chambers measuring approximately 18x30 inches (about 18 inches deep). Each of the glass containers was filled with the wet sediment collected from Escravos river (Located within the Gulf of Guinea) up to 12 inches depth followed by the introduction of 100mls of each of the Ester based fluids to the respective containers. The sediment/fluid mixture was mixed thoroughly by manual means using a metallic mixer. The experimental set up was allowed to settle for about 6hrs before the collection of the first sediment sample at day 0. The experiment was monitored for a period of 120 days and at each 30-day interval, sediment samples were collected and analysed for residual organic carbon and hydrocarbon utilizing bacteria. The entire set up was similar to the simulated sea bed experiment conducted by OGP (2003). The 4 sediment samples were labelled as follows; 1.SE-BR-EST, 2. SE-CH-EST, 3. SE-PFB-009, and 4. SE-PFB-008 depending on the type of ester based fluid added to the sediment.

Description of the Synthetic-based fluids (SBF) used for the study.

The SBF samples which were collected from the Nigerian Department of Petroleum Resources (DPR) were coded and have the following descriptions.

- 1. BR-EST (BAROID ESTER)
- 2. CH-EST (CHEVRON ESTER)
- 3. PFB-009 (Mixture of Ester and Olefin)
- 4. PFE-008 (Ester of Aliphatic acid).

Microbiological and Physicochemical Analysis of the Sediment samples

Enumeration of Total Heterotrophic Bacterial and Fungal Counts.

Heterotrophic bacteria and Fungi were enumerated by adopting the standard plate count technique using spread plate method. Appropriate dilutions of samples were plated out on nutrient agar plates for bacteria and potato dextrose agar (PDA) plates for Fungi. The plates for bacteria were made in duplicates and incubated aerobically at 29° C for 24hrs while that of Fungi were incubated aerobically for 3-4 days. $2\mu g/L$ of chloramphenicol was added to PDA plates to inhibit bacterial growth as described in Eaton *et al*, 1995.

Enumeration of hydrocarbon carbon utilizing bacteria

Hydrocarbon utilizing bacterial counts were obtained by plating out at low dilutions $10^{-1} - 10^{-3}$ of samples on mineral salt medium of Mills et al (1978). The composition of the medium in (g/L) is as follows NaCl (10), MgSO₄.7H₂0 (0.42), KCl (0.29), KH₂PO₄ (0.83), Na₂HPO₄ (1.25), NaNO₃ (0.42), Agar bacteriological (15), distilled water (1000 ml), and pH (7.2). The medium was autoclaved at 1.1 kg/cm² for 15 mins. The inoculated mineral agar plates were then inverted over sterile membrane filters moistened with crude oil (Escravos light) and held in the lid of the petri dishes. The dishes were wrapped round with a masking tape so as to increase the vapor pressure within the Petri dishes while the plates were incubated at 29°C for 6 days after which the growth of hydrocarbon degrading bacteria were observed and counted. For fungal plates, 0.1g of Penicillin was added to 250ml mineral salt medium to inhibit bacterial growth.

pH, Temperature measurement and Salinity

The pH of the sediment was measured with a portable water proof pH meter (Jenway, 3150, USA), Temperature was measured using portable thermometer (Hanana , H1-93510, USA). Salinity was measured as Chloride using the Argentometric method as earlier described in (Eaton *et al*, 1995).

Estimation of Background Nutrient Concentration of the sediment

Interstitial water samples were withdrawn with a simple apparatus as described in McKee *et al*, 1988. The collected interstitial water was filtered and inorganic nutrients such as Phosphorus and Potassium were analysed with ICP (Inductively coupled argon plasma emission spectrometer) as described in Eaton *et al*, 1995). Ammonium-Nitrogen was analysed with auto analyser as described in Eaton *et al*, 1995.

Detection of heavy metals:

Heavy metals were detected using the Atomic absorption Spectrophotometer (Perkin Elmer 5100PC, England) after sample preparation and digestion as previously described (Eaton *et al*, 1995).

Moisture content:

The moisture content of the sediment was measured by simple gravimetric analysis. 10grams of the sample containing water was dried in the oven at a temperature of 200°C after which, the sample was measured again and the difference in weight is the moisture content as previously described (Eaton *et al*, 1995)

Solvent extraction of Residual Oil

One gram of the sample was introduced into a separating funnel containing 50mls of Methylene chloride, this was followed by vigorous shaking for 10mins and filtration using Watman no.1 filter paper as previously described (Eaton *et al, 1995*) and the filtrate was collected in a clean conical flask.

Gas Chromatography of Oils

Degraded organic carbon were analyzed by gas chromatography using Hewlett Packard 5890 series 11 Gas chromatograph equipped with single flame ionization detector (FID) fitted with Perkin Elmer Nelson analog digital converter (900 series) and a Compaq deskpro computer. A J and W scientific DB-1 capillary column of 15 m length and an internal diameter of 0.32 mm wide bore of 1micron film thickness were used. A temperature program of 50-305°C increasing at 3.5°C per minute for 27.15min was employed. Hydrogen with a flow rate of 2ml per min was used as a carrier gas while the flow rate of air was 400ml per min. The detector temperature was 325° C while the injection port temperature was 305°C. 1 ml of the residual organic carbon extract was dissolved in methylene chloride at the ratio of 1:1 and a sample volume of 0.2 µl was injected into the GC.

Identification Microorganisms capable of utilizing SBF

The growth and morphology of bacterial isolates in minimal salts medium and on nutrient Agar plates were noted with regards to the following

Agar plates were noted with regards to the following characteristics; Form, Pigmentation,

Texture, Colour and Elevation. Fungal cultures were stained with Methylene blue and observed under a microscope (x40) and each fungal culture was identified based on its morphological characteristics. Bacterial cultures were stained using grams staining procedure and proper identification was done using a computerized BBL Enterotube identification test kits, manufactured by Becton Dickson Microbiology systems Inc. USA.

3. Results.

Microbiological and Physicochemical properties of Gulf of Guinea sediments

The total heterotrophic bacterial counts of the four sediments investigated ranged between 1.20-3.10 x 10^6 cfu/g while the hydrocarbon utilizing bacterial counts ranged from 0.011-0.080 x 10^6 cfu/g. Heterotrophic fungal and yeast counts in the sediments ranged between $0.0034 - 0.018 \times 10^6$ cfu/g while the hydrocarbon utilizing fungal and yeast counts ranged from $0.00016 - 0.00042 \times 10^6$ cfu/g.

The total organic carbon (TOC) in all the sediments tested were less than 10ppm suggesting that the sediment is pristine and have not undergone any significant pollution in the past. The levels of Nitrogen, Potassium and Phosphorus in all the sediments tested indicate that the sediments have sufficient nutrient that can sustain microbial growth and proliferation. The detailed results of the microbiological and physicochemical properties of the Gulf of Guinea sediments are shown on table 1.

Aerobic degradation of Ester based drilling fluids in the sediment

Aerobic microorganisms comprising of Bacteria and Fungi considerably degraded the Ester based fluids in the sediment within the 120 day experimental period. At day 60, all the ester based fluids tested achieved over 60 % degradation in the sediment and by the end of the 120 day period, almost all the residual ester present in the sediment (over 90%) were degraded by the microorganisms as shown in Figure 1.

Changes in the population dynamics of microorganisms with the capability to utilize the ester based fluids as the sole carbon source during biodegradation studies Changes in the population dynamics of microorganisms with the capability to utilize the ester based fluids as their sole carbon sources in the sediment were monitored during the 120 day experimental period and the results showed that the population densities of the microorganisms increased progressively from day 0 to day 60 which was the peak population density. The microbial population however showed a gradual decline after day 60 and this can be attributed to the considerable drop in the concentration of the ester based fluids as biodegradation progressed. The results are shown on table 2.

Microorganisms isolated from Gulf of Guinea Sediments with the capability to utilize the SBF.

A wide variety of microorganisms with the capability to utilize the synthetic based fluids were isolated from the Gulf of Guinea sediments during the 120 day period the experiment lasted. At day 0, various bacterial and fungal species were isolated from the various sediments tested and the predominant microbial flora comprised of Pseudomonas sp., Micrococcus sp., Alkaligenes sp., Corynebacterium sp., Actinomycetes sp., Enterobacter sp., Acinetobacter sp., Aspergillus niger, Penicillium sp., Penicillium crysogenum and *Candida* sp. but at the end of the experiment at day 120, the predominant microbial flora in the sediment declined to Pseudomonas sp., Alkaligenes sp., Micrococcus sp., Achromobacter sp., Aspergillus niger, Penicillium sp. and Penicillium crysogenum. The percentage of total heterotrophs in the sediment with the capability to utilize SBF ranged from 0.92-3.30%, the detailed results are shown on table 3.

 Table 1. Microbiological and Physicochemical properties of Gulf of Guinea sediments

		SE-BR-EST	SE-CH-EST	SE-PFB-009	SE-PFE-008
1	Total Heterotrophic Bacterial Counts (Cfu/g x 10 ⁶)	1.20	2.60	1.40	3.10
2	Hydrocarbon utilizing bacterial counts (Cfu/g x 10 ⁶)	0.011	0.086	0.023	0.041
3	Total Heterotrophic Fungi/Yeast Counts. (Cfu/g x 10 ⁶)	0.004	0.016	0.0034	0.018
4	Hydrocarbon Utilizing Fungi/Yeasts. (Cfu/g x 10 ⁶)	0.00016	0.00042	0.00016	0.00022
5	Total Organic Carbon (TOC) (ppm)	6.50	7.60	9.50	8.40
6	pH	6.70	6.80	6.70	6.90
7	Temperature (⁰ C)	23	24	24	23
8	Salinity (mg/g)	5280	5360	5540	5920
9	Moisture content (%)	56	58	60	58
10	Phosphorus (mg/g)	106	128	98	130
11	Potassium (mg/g)	98	83	76	82
12	Ammonia-N (mg/g)	3.20	3.11	2.80	3.0
13	Heavy Metals detected	Pb(0.032), Cr	Cd(0.05),	Fe(0.022),	Fe (0.031), Cd
		(0.10)	Pb(0.018)	Zn (0.04)	(0.006), Zn (0.03)



Figure 1. Degradation of Ester based drilling fluid in the sediment by aerobic microorganisms.

4. Discussion:

The degradation of surrogate ester based SBF under natural conditions by the indigenous microbial flora of the Gulf of Guinea sediments were examined under natural environmental conditions over a 120 day period. Incubation at hydrostatic pressure was not necessary because previous research have shown that incubation at such deep offshore pressure had no effect on the hydrocarbon substrate degradation (Benka-Coker and Olumagin, 1995, Alan *et al*,2006, Deborah and Alan ,2006).

The present study have demonstrated that the Gulf of Guinea sediments used in the study population harboured considerable of microorganisms with the capability to utilise and degrade the ester based fluids. Analytical study on the background nutrient composition of the sediment used in the study equally showed that the sediments had fairly good nutrient composition that can sustain microbial growth and proliferation. Previous investigations have shown that the Gulf of Guinea sediments have significant background nutrient composition and are populated with wide variety of microorganisms with the capability to utilize the organic carbon in the sediment (Okoro, 2010a). In the present study, the indigenous microbial populations of the sediment ranged between $1.20 - 3.10 \times 10^6$ cfu/g and only about 0.92-3.30% of the total heterotrophic population have the capability to utilize and degrade the ester based SBF in the sediment. Deborah and Alan (2006) have demonstrated that the indigenous

aerobic microbial populations in the Gulf of Mexico sediments ranged between $1.0 \ge 10^8 - 1.4 \ge 10^9$ cfu/g but less than 10% of the total heterotrophic population have the capability to utilize the SBF as their sole carbon and energy source.

In the present study, it was observed that the populations of microorganisms in the sediment that are capable of utilizing the ester based fluids increased in population when the ester based fluids were introduced but the populations declined gradually upon the degradation of the ester based fluids. A similar study conducted by OGD (2003) showed that biodegradation of esters increased with higher concentration of fluids in the sediment, the study also went further to demonstrate that sediment type (Sandy Vs Clay or Silt) for instance effects degradation rate and that degradation occurs more rapidly in Silt/Clay sediments than in sandier sediments. The sediment that was used in this study was sandy.

An investigation carried out Cavellier *et al*, 1999 with the Gulf of Mexico sediments showed that under aerobic and anaerobic conditions, about 50% of the esters in the sediments were degraded after 28 days and the residual concentration of esters were reduced to zero after 120 days of exposure. In the present study, only aerobic degradation was monitored and the average % degradation of the Ester based fluids in the sediment was, about 43% for a 30 day period and 94% for the 120 day period. A similar investigation carried out by OGD(2003) revealed that

loss of the SBF deposited on the cuttings which was measured over a period of 150-187 days was about 80%. Other investigators like Prince (1993), Swannel *et al*,(1996), Kjeilen (1997) and Paulsen *et al* (1997)

have equally demonstrated that the organic ingredients in the SBF are readily biodegradable over a considerable length of time.

Table 2. Population dynamics of Hydrocarbon utilizing bacteria during biodegradation of Ester based fluids (Bacterial Population x 10^6 cfu/g)

SEDIMENT	DAY 0	DAY 30	DAY 60	DAY 90	DAY 120
SE-BR-EST	0.011	0.580	1.260	0.360	0.086
SE-CH-EST	0.080	0.620	1.560	0.840	0.460
SE-PFB-009	0.023	0.180	1.040	0.960	0.210
SE-PFE-008	0.041	0.186	1.132	0.840	0.110

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	SE-BR-EST	SE-CH-EST	SE-PFB-009	SE-PFE-008
DAY 0	Micrococcus sp.,	Acinetobacter lwoffii,	<i>Vibrio</i> sp.,	Micrococcus sp.,
	Pseudomonas sp.,	Pseudomonas sp.,	Pseudomonas sp.,	Pseudomonas sp.,
	Enterobacter sp.,	Enterobacter sp.,	Acinetobacter	Enterobacter sp.,
	Corynebacterium	Alkaligenes sp.,	lwoffii. Alkaligenes	Corynebacterium sp.,
	sp., Actinomycetes sp.,	Flavobacterium sp.	sp. Pseudomonas	Achromobacter sp.,
	Achromobacter sp.,	Bacillus sp.,	mallei. Penicillium	Pseudomonas mallei
	Pseudomonas mallei,	Aspergillus niger	sp., Actinomycetes,	Rhodotorula sp.
	Aspergillus niger,	Aspergillus	Enterobacter,	<i>Candida</i> sp.,
	Candida sp.,	fumigatus	Penicillium	Cladosporium sp.,
	Penicillium sp.,	Penicillium	crysogenium,	Fusarium sp
	Rhizopus sp.	crysogenum	Aspergillus niger	
DAY 30	Micrococcus sp.,	Pseudomonas sp,	Pseudomonas sp	Micrococcus sp.,
	Pseudomonas	Alkaligenes sp.,	Alkaligenes	Pseudomonas sp.,.,
	sp., Actinomycetes sp.,	Flavobacterium sp.	sp., Pseudomonas	Corynebacterium sp.,
	Achromobacter sp.,	Bacillus sp.,	mallei. Penicillium sp.,	Achromobacter sp.,
	Pseudomonas mallei,	Aspergillus niger	Penicillium	Rhodotorula sp.
	Aspergillus niger,	Penicillium	crysogenium,	<i>Candida</i> sp.,
	Penicillium sp.,	crysogenum	Aspergillus niger	Cladosporium sp.,
				Fusarium sp.
DAY 60	Micrococcus sp.,	Pseudomonas sp,	Pseudomonas sp	Micrococcus sp.,
	Pseudomonas	Alkaligenes sp.,	Alkaligenes	Pseudomonas sp.,
	sp., Actinomycetes sp.,	Flavobacterium sp.	sp,. Penicillium sp.,	Achromobacter sp.,
	Achromobacter sp.,	Bacillus sp.,	Penicillium	Rhodotorula sp,.
	Aspergillus niger,	Aspergillus niger	crysogenium,	<i>Candida</i> sp.,
	Penicillium sp.,	Penicillium	Aspergillus niger	Cladosporium sp.,
		crysogenum		Fusarium sp.
DAY 90	Micrococcus sp.,	Alkaligenes sp.,	Pseudomonas sp	Micrococcus sp.,
	Pseudomonas	Flavobacterium sp.	Alkaligenes	Pseudomonas sp.,
	sp., Actinomycetes sp.,	Bacillus sp.,	sp,. Penicillium sp.,	Achromobacter sp.,.
	Achromobacter sp.,	Aspergillus niger	Penicillium	<i>Candida</i> sp.,
	Aspergillus niger,	Penicillium	crysogenium,	Cladosporium sp.,
	Penicillium sp.,	crysogenum	Aspergillus niger	
DAY 120	Micrococcus sp.,	Alkaligenes sp.,	Pseudomonas sp	Micrococcus sp.,
	Pseudomonas sp.,	Flavobacterium sp.	Alkaligenes	Pseudomonas sp.,
	Actinomycetes sp.,	Bacillus sp.,	sp,. <i>Penicillium</i> sp.,	Achromobacter sp.,.
	Achromobacter sp.,	Aspergillus niger	Penicillium	<i>Candida</i> sp.,
	Aspergillus niger,	Penicillium	crysogenium,	Cladosporium sp.,
	Penicillium sp.,	crysogenum	Aspergillus niger	
% of	0.92	3.30	1.64	1.32
Heterotrophs				
utilizing SBF				

One important issue in mixed culture microbial degradation is the issue of dominance of some microbial species over others. The population dynamics and the species diversity of the indigenous microorganisms that have the capability to utilize the ester based fluids as their sole carbon source was monitored and the dominant microbial flora in the Gulf of Guinea sediments were identified as Pseudomonas sp., Alkaligenes sp., Micrococcus sp., and Achromobacter sp. among the bacterial species and Penicillium sp., Aspergillus niger and Penicilluim crysogenum among the fungal species. A similar study in the Gulf of Guinea sediments by Benka-Coker and Olumagin (1995) showed the predominant microbial flora of the Gulf of Guinea sediments with the capability to utilize the SBF as Alkaligenes and Micrococcus, among the Bacterial species and Penicilium and Cladosporium sp. among the Fungal species. The study also showed that 6% of the total heterotrophic population possess the capability to utilize the SBF as their sole carbon and energy source.

Conclusion:

The present study have clearly demonstrated that all the ester based fluids used in the study are readily biodegradable and the Gulf of Guinea sediment is populated with diverse microbial flora with adequate nutrient composition which made the biodegradation of ester based SBF easier. The literature reviews conducted as part of this research revealed that ester based SBF are more readily degradable than others such as paraffins and olefins. In conclusion therefore, it can be deduced from the present study that the 4 ester based SBF used in the study are readily biodegradable by the Gulf of Guinea sediment and as such can be safely discharged into the ocean. The recommended ocean discharge is safer and far more economical than other disposal options like onshore thermal treatment process and land farming.

Acknowledgement

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