

# Evaluation of the Effectiveness of Combined Dissolved Air Flotation (DAF), Filtration and Activated Carbon Adsorption Processes in the Treatment of Oily Wastewater from Drilling Activities

<sup>1</sup>Amorin, R., <sup>2</sup>Agorhom, E. A. and <sup>1</sup>Mensah, F. K.

<sup>1</sup>Petroleum Engineering Department, University of Mines and Technology, Tarkwa

<sup>2</sup>Minerals Engineering Department, University of Mines and Technology, Tarkwa

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## Abstract

Treatment of drilling waste can be very ambitious depending on the type of waste treatment method and additives used during the fluid formulation. The success of a particular treatment method depends primarily on its ability to address the specific problems posed by the waste. This study evaluated the effectiveness of flotation process in the treatment of oily wastewater. The flotation experiments were carried out in a Denver flotation cell at a constant agitation speed of 600 rpm using different air flow rates of 2, 4, 6 and 8 L/min. The maximum flotation times for each of the air flow rates were also established. The flotation results showed that air flow rate has effect on the ultimate oil recovery, oil volume pull and the maximum flotation time. Air flow rate of 4 L/min recovered 130 ml of oil compared with 110, 110 and 120 ml for 2, 6 and 8 L/min, respectively. However, the flotation process used was not able to meet the EPA recommended oil retention of 10 mg/l for the waste water without further treatment for all the air flow rates. Simple filtration and activated carbon (0.25 g) adsorption of the wastewater produced after the flotation process at the 4 L/min air flow rate reduced the residual oil and grease content of 22.73 mg/l to 9.32 mg/l and 6.12 mg/l, respectively. Increasing the activated carbon concentration from 0.25 g to 0.5 g further reduced the oil and grease content to 4.05 mg/l. Again, addition of caustic solution to the wastewater raised the pH from 0.6 to 7.25. The flotation, filtration, adsorption and neutralisation processes make the wastewater environmentally safe for handling and disposal. A treatment option for an oily wastewater is proposed.

**Keywords:** Oily Wastewater, flotation, Activated Carbon, Filtration, Pollution

## 1 Introduction

Drilling operations produce significant amount of wastes which include drill cuttings, spent mud and oily wastewater (Onwukwe and Nwakaudu, 2012). Oily wastewater contains high level of dissolved and suspended total solids such as oil, grease, chromium, phosphate, lead, iron, chemical oxygen demand (COD), biochemical oxygen demand (BOD), nitrite, sulphide and copper (Yu *et al.*, 2017). Due to the high content of oil and residual organic pollution, the oily wastewater cannot be discharged directly into the environment without treatment. Therefore, wastewater must be treated to meet Environmental Protection Agency (EPA) requirement before is disposed into the environment (Table 1). The maximum allowable emission of oily wastewater

concentration before disposal for China is 10 mg/L, 2 – 10 mg/L (Germany), 3 mg/L (Iraq), and 5 mg/L (Switzerland) amongst others (Alwared and Faraj, 2015).

Oily wastewater may either be soluble or insoluble. Soluble oily wastewater is the emulsion of oil in water and contain three components which are the dispersed phase (oil), emulsifying agent and the external phase (Mohammed *et al.*, 2005). Treatment of these wastes may result in an improved oil/water separation, improved water quality, oil recovery, water reuse, protection of downstream facilities and environmental permit compliance (Bande *et al.*, 2008). For effective treatment of oily wastewater, both phases must be separated. Some techniques used in treating wastewater include gravity

separation, microfiltration, chemical coagulation, carbon adsorption, biological treatments and electro flotation (Yu *et al.*, 2013; Mohammed *et al.*, 2005). One suitable technique for the removal of oil from water is by flotation using a device called flotation cell in which bubble and oil collision is produced by agitation of the pulp (oil wastewater). The flotation process produces oily waste as scum and wastewater for further treatment. It has been used in the treatment of wastewater from mining industries, refineries and other industries (Kaya, 2009). This study therefore evaluates the application and effectiveness of bench-scale flotation cell in the treatment of oily wastewater from drilling activities in Ghana. The flotation process was complimented with simple filtration and activated carbon adsorption to meet EPA oil and grease content of a wastewater of not more than 10 mg/L.

**Table 1 EPA Requirement for Oily Wastewater**

Parameters or Composition	EPA Guidelines/ Threshold
pH	6 – 9
Conductivity	1500
Total Dissolved Solids (mg/L)	1000
Temperature (K)	650
Turbidity (NTU)	75
Colour (TCU)	250
Total Suspended Solids (mg/L)	50
COD (mg/L)	250
BOD (mg/L)	50
Oil (mg/L)	10
Sulphide (mg/L)	1.5
Nitrite (mg/L)	50
Total Phosphorus (mg/L)	2
Cadmium (mg/L)	0.10
Lead (mg/L)	0.10
Zinc (mg/L)	10
Copper (mg/L)	5
Iron (mg/L)	10
Arsenic (mg/L)	0.10

(Anon, 2017)

## 2 Materials, Methods Used

### 2.1 Sample Preparation

Oily waste was collected from one of the waste treatment company at Secondi-Takoradi. All experiments were carried out using solutions prepared from analytical grade reagents and Tarkwa tap water. 500 ml of oily wastewater was mixed with 50 ml of 0.5 M HCl and stirred thoroughly to obtain a homogeneous mixture. The HCl serving as an emulsifier breaker. 900 ml of fresh mixed water was

added to the mixture to make it possible to float in a 1.5 litre flotation cell. Four of such samples were prepared (A, B, C and D) as shown in Table 2. The temperature and pH of the samples were recorded.

**Table 2 Sample Treatments**

Sample	A	B	C	D
Flow rate (L/min)	2	4	6	8
Speed (rpm)	600	600	600	600
Volume of oily wastewater (ml)	500	500	500	500
Volume of mix water added (ml)	900	900	900	900
Initial pH of sample	5.60	5.68	5.68	5.64
Volume of acid (HCL) added (L)	0.05	0.05	0.05	0.05
pH after acid addition	0.50	0.49	0.49	0.47
pH after water addition	0.97	0.96	0.94	0.96
Temperature after acid addition (°C)	32.9	33.9	34.8	36.6
Temperature after water addition (°C)	29.8	30.6	31.1	32.2
Volume of base (NaOH) added (L)	0.05	0.05	0.05	0.05

### 2.2 Flotation

The flotation experiments were carried out in a 1.5 L Denver flotation machine. The prepared mixtures were put in the cell and agitated for 5 min to allow for uniform mixing and homogeneity. After which the samples were floated using different oxygen flow rates of 2, 4, 6 and 8 L/min at a constant speed of 600 rpm. The froth (scum) was cut and collected until no noticeable scum could be seen on the surface of the water in the cell. The maximum time required to cut and collect all the scum was noted and recorded for each of the different oxygen flow rates. The recovered oils (scums) was heated at a fixed temperature of 105.5 °C for 19 min to get rid of all physically trapped water. The oil was then allowed to cool after heating and its final weight as well as final volume determined.

### 2.3 Pulp Neutralisation

A base solution was prepared from adding 150 g of NaOH to 500 ml of water. 50 ml of 0.5 M NaOH solution was added to the water recovered (float

tailings) from each stage to neutralise the acid effect. The volume of water obtained from each sample were divided into two halves. One was used for oil and grease analysis and the other for further treated using filtration and adsorption. The pH of the solutions was then determined.

### 2.3 Adsorption

400 ml of the neutralised water obtained from sample B (recorded best results after flotation process) was further divided into two. Activated carbon of size 200 microns was used for the adsorption process. 0.25 g and 0.5 g of activated carbons were added to 200 ml each of the neutralised pulps and rolled on a bottle roller for 30 min. After 30 min of rolling, both samples were filtered to remove the activated carbons to obtain residues for further testing.

### 2.4 Filtration

400 ml of the neutralised water obtained from sample C (recorded second-best results after flotation process) was filtered using a funnel and filter paper. After 4 hours of filtration, the filtrate was collected and tested.

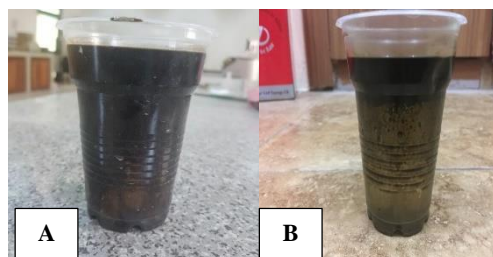
### 2.5 Oil and Grease Content Analysis

The neutralised water, filtered water and the water obtained after the adsorption was taken to AngloGold Ashanti laboratory to determine the oil and grease content using Atomic Absorption Spectroscopy (AAS).

## 3 Results and Discussions

### 3.1 Effect of Acid Addition on The Samples

The effect of acid digestion on the separability of oily wastewater is shown in Fig. 1. It was observed that addition of 50 ml of HCl to 500 ml of oily wastewater lightened the heavy oil (Fig. 1) and weakened the oil-water bond for effective oil-bubble interaction and effective collection via the flotation process.



**Fig. 1 Effect of Acid Addition (HCl) on Separability of Oily Wastewater (A) before HCl Addition and (B) After HCl Addition**

### 3.2 Effect of Oxygen Flow Rate Oil Separation and Recovery

The result of the efficiency of the flotation process (in terms of volume of oil recovered against time used) at different oxygen flow rates are presented in Table. 3. The results showed that an increase in oxygen flow rate from 2 L/min to 8 L/min had a positive effect on the flotation process by increasing the rate of oil recovery (Table 3).

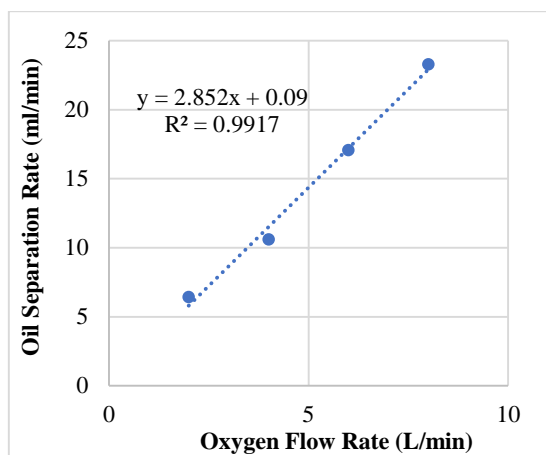
**Table 3 Froth Flotation and Activated Carbon Processes Results**

Sample	Units	A – 2 L/min	B – 4 L/min	C – 6 L/min	D – 8 L/min
<b>Oil Recovered Before Heating</b>					
Floating time	min	18.58	14.14	7.03	6.01
pH after flotation		0.57	0.64	0.59	0.6
Temperature of water obtained	°C	28.9	29.1	30.2	30.7
Volume of oil recovered	ml	120	150	120	140
Rate of oil separation (R)	ml/min	6.46	10.61	17.07	23.29
<b>Water Recovered Before Heating</b>					
Density of recovered water	lb/gal	9.8	9.8	9.8	9.8
Volume of water recovered	ml	924	921	918	937
pH of water after NaOH addition		7.2	7.29	7.32	7.29
<b>Oil Recovered After Heating</b>					
Heating temperature	°C	102	109	104	105
Heating time	min	13	25	15	23
Volume of oil after heating	ml	110	130	110	120
Volume of water lost	ml	10	20	10	20
<b>Residual Oil Left in Water (After Flotation Test)</b>					
Oil and grease content	ppm	34.56	22.73	25.95	32.47
<b>Residual Oil Left After Filtration and Carbon Treatments Rolled for 30 Minutes</b>					
Simple Filtration	ppm			9.32	
Activated Carbon (0.25g)	ppm		6.12		
Activated Carbon (0.50)	ppm		4.05		

This may be due to increase number of bubbles generated per unit area. At low flow rate of 2 l/min, less oxygen was bubbled through the sample restricting rapid upwards flow of the oil to the surface for collection requiring 18.58 min to float. Also, at higher flow rates of 4, 6 and 8 L/min it was observed that more scums were formed and this resulted in the higher volumes of oil recovered at the relatively shorter times. In order to compare the efficiencies of the different oxygen flow rates on the oil separation process, the volume of oil collected (ml) was normalized against the required time (min) using Equation (1):

$$\text{Rate} \left( R, \frac{\text{ml}}{\text{min}} \right) = \frac{[\text{volume of Oil Recovered (ml)}]}{[\text{Time Required (min)}]} \quad (1)$$

The results showed that rate of oil separation and recovery increased with increasing oxygen flow rate from 6.46 ml/min to 23.29 ml/min for 2 L/min and 8 L/min, respectively. A positive correlation between the oxygen flow rate and oil separation rate was observed as shown in Fig. 2.



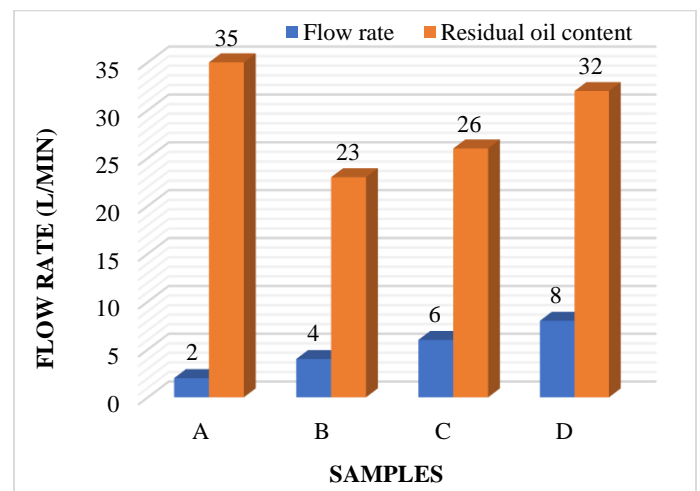
**Fig. 2 Oxygen Flow Rate and Oil Separation Rate Relationship**

### 3.3 Effect of pH

From Table 3, the pH of the samples decreased from about 5.6 to 0.6 upon the addition of 50 ml HCl making the sample very acidic for disposal. But upon the addition of the NaOH, the acidity of the samples was neutralised to give a pH range of 7.20 to 7.30; falling within the recommended EPA requirement of 6 – 9, therefore environmentally safe for handling and disposal.

### 3.4 Residual Oil Content after Flotation

A moderate residual oil content of 25.95 ppm was obtained from flow rate 6 L/min with the best residual oil content of 22.73 ppm at flow rate 4 L/min. This result shows that at flow rate of 4 l/min, almost all dissolved and suspended oils droplets were able to be collected under favourable conditions. But none of the flow rates was able to meet the residual oil volume of 10 mg/l (ppm) as stated by EPA hence further treatment (Figs. 3 and 4).



**Fig. 3 Effect of Flow Rate on Residual Oil Content**

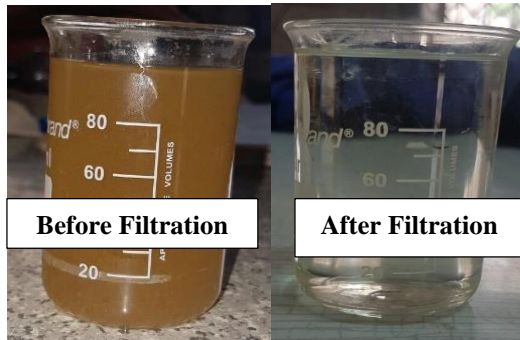


**Fig. 4 Nature of the Water Samples After Flotation**

### 3.5 Residual Oil Content after Simple Filtration

Based on the higher residual oil contents of 34.56 and 32.47 ppm obtained for samples A and D respectively (2 L/min and 8 L/min, respectively) after flotation, they were not recommended for further works. Further treatment was carried out on samples B and C (4 L/min and 6 L/min, respectively). From the simple filtration test conducted on sample C, the residual oil content decreased from 25.95 ppm to 9.32 ppm (64.08%

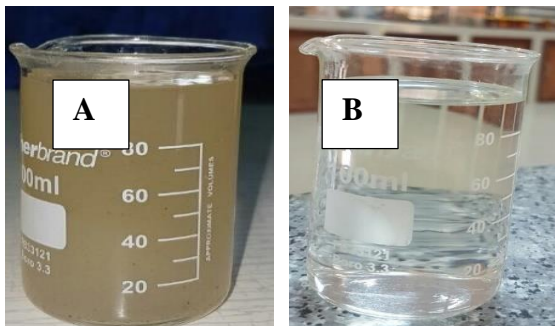
reduction) making it meet the EPA recommended oil and grease content of 10 mg/l of oil retention. Also, the simple filter paper filtration method produces less turbid and clear water as evident in Fig. 5.



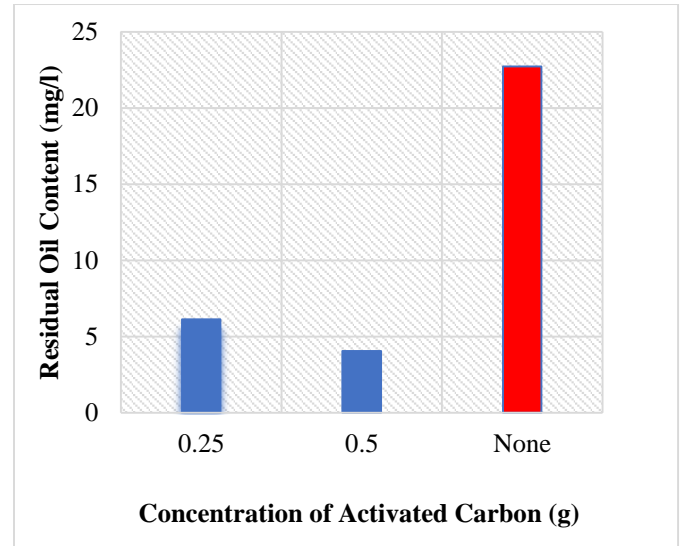
**Fig. 5 Clarity of Sample C Before and After Filtration**

### 3.6 Residual Oil Content after Adsorption Process

Applying activated carbon on sample B; by the addition of 0.25 g and 0.50 g of activated carbon rolled for 30 minutes, the residual oil content decreased from 22.73 ppm to 6.12 ppm (73.08%) and 4.05 (82.18%) ppm, respectively (Figs. 6 and 7). Based on these results, both recorded values were able to meet the recommended EPA standards of 10 mg/l of oil retention. The residual oil content removal for the activated is higher than that of simple filtration. The results showed an environmental acceptable and disposal waste water can be produced after flotation followed by either a simple filtration and/or after activated carbon adsorption.



**Fig. 6 (A) Sample 'B' Before and (B) Sample 'B' After Adsorption Process**



**Fig. 7 Effect of Activated Carbon Concentration on Residual Oil Removal**

## 4 Conclusions and Recommendation

Based on the study conducted the following conclusions were drawn;

- i. The addition of HCl to oil in water emulsions serves as a good demulsifier for breaking oil water bonds.
- ii. The froth flotation process employed was able to recover waste oil but could not produce waste water to meet the EPA recommended oil retention of 10 mg/l without further treatment by filtration and adsorption.
- iii. The choice of oxygen flow rate has an effect on oil separation and recovery rate. The highest oil separation rate was achieved at 8 L/min oxygen flow rate
- iv. Simple filtration and activated carbon adsorption after froth flotation was able to reduce residual oil by 64.08% and 82.18% respectively to meet the recommended EPA requirement of 10 mg/l.
- v. The addition of the base (NaOH), converted all acidic solutions (pH of 0.6) to a neutral solution (pH 7.25); making it environmentally safe for handling and disposal.

It is recommended that the treatment of oily waste water could be treated with floatation process and be accompanied by either simple filtration or adsorption to enhance oil recovery.



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## Authors



**Richard Amorin** is a Senior Lecturer at the department of Petroleum Engineering at the University of Mines and Technology, Tarkwa, Ghana. He holds MSc and PhD degrees in Petroleum Engineering. He is the Faculty Advisor for the Society of Petroleum Engineers (SPE) on campus. He is also a member of African Union Network of Sciences (AUNS), and International Association of Engineers (IAEng). His research focuses on gathering and transportation of oil and gas, drilling optimisation and HSE. He is the recipient of the 2019 SPE Africa-Region Distinguished Achievement Award for Petroleum Engineering Faculty.



**Eric A. Agorhom** obtained his BSc (Hons) and PhD degrees in Minerals Engineering from the University of Mines and Technology (UMaT), Tarkwa and the University of South Australia (UniSA), Adelaide, Australia. He is currently a Senior Lecturer in the Minerals Engineering Department at UMaT. His research interest is in the areas of Process Mineralogy, Geometallurgy, Copper-gold Ore Froth Flotation, Aqueous Extraction, Mine Waste Characterisation, and Process Plant Optimisation and Improvement. He is an associate member of the Australasian Institute of Mining and Metallurgy (AAusIMM) and a member of the West African Institute of Mining, Metallurgy and Petroleum (WAImm).



**Felicia Kukuwah Mensah** is an alumnus of University of Mines and Technology Tarkwa Ghana. She holds a BSc degree in Petroleum Engineering. She is currently an MSc student at Changzhou University studying Oil and Gas Storage and Transportation. Her research areas of expertise are centred on environment and safety and the transportation of oil and gas.