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Article

Removal of Hydrocarbons from Drill Cuttings Using Flotation Enhanced Stirred Tank (FEST)

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Abstract. Treatment of drill cuttings (DC) by washing processes consumes a considerable volume of solvents, resulting in high chemical wastes and operating cost. To minimize the chemical use, this work aims to develop an integrated process of dissolved air flotation (DAF) and mechanical stirring, called Flotation Enhanced Stirred Tank (FEST), as a pretreatment process for DC washing, in which total petroleum hydrocarbon (TPH) was a major pollutant of concern. The performance of an individual DC treatment process (stirring and DAF) was firstly investigated to determine the optimal experimental range. Then, response surface methodology with central composite design was applied to optimize three operational factors (saturated pressure (P_s), mixing speed (V_m), and treatment time (t)) for the integrated process, having TPH removal efficiency as the response output. Effects of hydrodynamic condition in terms of a/G ratio on the TPH removal performance were also analyzed. The experimental results revealed that mixing speed and saturated pressure were the significant factors affecting the TPH removal efficiency. FEST could yield the maximum TPH removal of 47% under the P_s of 4 bars, V_m of 400 rpm, and t of 70 min, showing its better performance than a single process from which less than 40% TPH removal was achieved. Combining DAF with stirring resulted in more turbulence in the system and thus improving the contact between hydrocarbon and bubbles. Therefore, better TPH removal could be obtained from FEST at lower a/G ratios compared to DAF. Furthermore, using saline water as a treatment medium was also possible. Overall, FEST exhibited its potential as an environmentally friendly process for the pretreatment of DC.

Keywords: Drill cuttings treatment, flotation enhanced stirred tank, hydrodynamic parameter, total petroleum hydrocarbons.

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1. Introduction

With the increase of oil and gas exploration and production to support the rapid growth of population and industrialization, it has led to an unavoidable increase of drilling wastes particularly a large volume of solid materials, known as drill cuttings (DC) [1, 2]. DC is oil-based byproducts generated from drilling operations, and they are brought to the surface together with drilling mud [3, 4], which contains toxic and poorly biodegradable contaminants such as petroleum hydrocarbons and a variety of heavy metals [5, 6]. DC are categorized as hazardous waste by the European Waste Directive [7] due to their high environmental risks. Therefore, it is necessary for oil and gas industries to properly manage DC and minimize potential negative impacts that might pose on the ecosystem.

A number of technologies have been utilized for DC treatment to date. Drying and thermal desorption are typical processes, in which a high-speed rotating centrifuge and heat are applied for removing hydrocarbons from DC, respectively [8-10]. Stabilization and solidification (S/S) involve the use of chemicals such as cement, lime, clay, fly ash, or other pozzolanic materials to limit the solubility or mobility of the contaminants [11, 12]. Supercritical fluid extraction and chemical washing promote the release of hydrocarbon contaminants from DC through the aid of extracting solutions [13-16]Bioremediation is another remedial alternative, in which hydrocarbons are degraded by micro-organisms [17, 18].

Among these technologies, chemical washing is of most interest as an easy and rapid process with low energy consumption. Moreover, various bio-based chemicals have recently shown their effective implication in DC treatment [19], making washing processes become more sustainable. Despite several advantages, chemical washing highly consumes extracting agents to achieve desired treatment targets, which further results in high chemical waste volumes and operating cost. Keeping these drawbacks in view, an initial removal of hydrocarbons from DC prior to chemical washing could be helpful. Still washing-based processes, water as the greenest and cheapest solvent is our first choice.

Contact between washing agents and contaminated solids is one of major mechanisms controlling the treatment efficiency. This mechanism generally involves the use of agitators or impellers, which might be inadequate in some cases. To improve the process, flotation could be one of possible options. Flotation has been reported as a promising technique for oily wastewater treatment [20-22]. It is a selective process that exploits the difference of particle surface properties. A particle surface is hydrophobic (repelled by water) like oil, the bubbles generated by flotation process collide and attach to the surface of oil droplets. This increases the rise rate of bubble-oil agglomerates and results in the removal of mineral and oils from oily wastewater [23-26]. Furthermore, the applications of flotation have been reported for the treatment of oil-contaminated soil [27-29]. In 2005, Urum et al. studied the removal of crude oil from soil using air sparging assisted stirred tank reactors with two surfactants (rhamnolipid and SDS). They claimed that the removal of crude oil from contaminated soil was not only dependent on the washing media properties, but also on the effect of air sparging [27]. This result suggests that the separation of hydrocarbons from DC may also be accomplished by the selective attachment of hydrophobic oil to bubbles generated by flotation process. Therefore, this study aims to develop a flotation enhanced stirred tank (FEST) as a pretreatment process for DC treatment. Design of experiment (DOE) using response surface methodology (RSM) based central composite design (CCD), which is more accurate compared to other designs and no need for a three-level factorial experiment for building a second-order quadratic model [30], is applied to identify the TPH removal mechanism and optimize the DC treatment conditions with three relevant parameters (i.e., saturated pressure, rotational speed, and treatment time). As a bubble-based process with stirring, the hydrodynamic parameter in term of the ratio of interfacial area to velocity gradient or a/G along with the calculation of collision efficiency (E_C) are applied in order to analyze the effectiveness of FEST in DC treatment. In addition, the effect of saline water on the DC treatment is investigated.

2. Materials and Methods

2.1. DC Samples

Soil samples were collected from an offshore petroleum drilling site in the Gulf of Thailand and were kept at 4°C before experiments. The DC samples were air-dried at room temperature and then analyzed for pH, total petroleum hydrocarbon (TPH), moisture content, and size distribution as shown in Table 1. Note that large particles (> 2000 μm) such as soil grains and gravels were removed and not subject to analysis because these bigger particles were hard to react with other constituents.

Table 1. The characteristics of DC studied in this work.

Parameters	Values
pH	7.74
TPH (mg/kg)	236,000
Moisture content (%)	5.26
Size distribution (%)	
Sand (50 - 2000 μm)	14.43
Silt (2 - 50 μm)	82.00
Clay (<2 μm)	3.56

2.2. DC Treatment Procedures

DC treatment was conducted in a laboratory scale setup. All experiments (stirring, DAF and FEST) were investigated in a cylindrical reactor made of transparent acrylic with 6 cm in diameter and 25 cm in height. Note that the solid-to-liquid ratio (S/L) of the reactor was fixed at 1:10 [31]. For stirring experimental setup, the cylindrical reactor was equipped with a stirred motor and a 4-pitched blade turbine with 45 degrees (4-blade PBT). For dissolved air flotation (DAF) experimental setup, this cylindrical reactor without the stirring parts was connected with an air compressor and a pressure vessel at the reactor's bottom for generating fine bubbles. The combination of both stirring and DAF setups was applied as a Flotation Enhanced Stirred Tank (FEST) setup as shown in Fig. 1. Tap water and synthesized saline water were used as treatment reagents. The saline water contained 30 g/L NaCl, which referred to the salinity found in the Gulf of Thailand [32]. All experiments were carried out under room temperature of approximately 28±1°C.

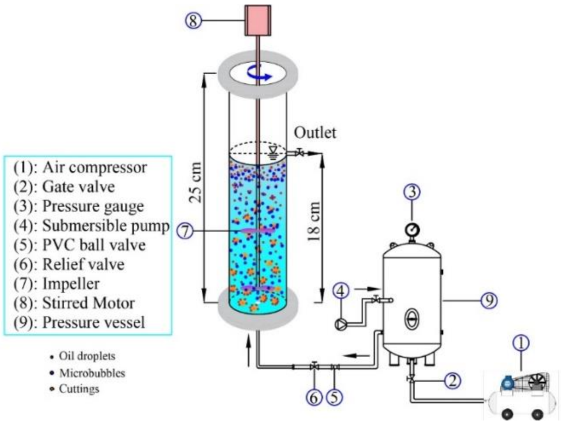

Fig. 1. Lab-scale experimental setup of FEST.

2.3. Experimental Design of DC Treatment Using FEST

Response surface methodology (RSM) based a central composite design (CCD) is a tool used for creating significantly good models with a smaller number of experiments [33]. RSM-CCD was employed to better understand the relationship of related factors and optimize the DC treatment conditions using FEST. Saturated pressure (P_s), mixing speed (V_m), and treatment time (t) were selected as independent variables X_1, X_2, and X_3, respectively. In CCD, the selected experimental points included 8 cubic points, 6 axial points ($\alpha = \pm 1.68$), and 6 replicates at the center point ($\alpha = 0$); hence there was totally 20 runs for conducting the optimization. These variables were assessed at five coded levels (–1.68, –1, 0, +1, and +1.68), as shown in Table 2. In detail, the experimental values of P_s, V_m, and t were in a range of 2–4 bar, 200–600 rpm, and 20–60 min, respectively. Minitab 17 statistical package (MINITAB Inc., PA, USA) was used for the statistical analysis of the results.

Table 2. Experimental conditions designed from the matrix of RSM-CCD.

Trial No.	Coded variables			Experimental variables		
	X_1^a	X_2^b	X_3^c	X_1^a	X_2^b	X_3^c
1	-1	-1	-1	2	200	20
2	1	-1	-1	4	200	20
3	-1	1	-1	2	600	20
4	1	1	-1	4	600	20
5	-1	-1	1	2	200	60
6	1	-1	1	4	200	60
7	-1	1	1	2	600	60
8	1	1	1	4	600	60
9	-1.68	0	0	1.32	400	40
10	1.68	0	0	4.68	400	40
11	0	-1.68	0	3	63.64	40
12	0	1.68	0	3	736.36	40
13	0	0	-1.68	3	400	6.36
14	0	0	1.68	3	400	73.64
15[d]	0	0	0	3	400	40
16[d]	0	0	0	3	400	40
17[d]	0	0	0	3	400	40
18[d]	0	0	0	3	400	40
19[d]	0	0	0	3	400	40
20[d]	0	0	0	3	400	40

[a] Saturated pressure (P_s) (bar)
[b] Rotational speed (V_m) (rpm)
[c] Treatment time (t) (min)
[d] Center points

2.4. Analytical Parameters

2.4.1. TPH removal efficiency

The performance of FEST on DC treatment was examined in term of TPH removal efficiency as expressed in Eq. (1). The TPH concentrations were measured using gas chromatography equipped with a flame ionization detector (GC-FID) following the US EPA method 8015B [34].

$$\text{TPH removal (\%)} = \frac{\text{Initial TPH-Residual TPH}}{\text{Initial TPH}} \times 100 \quad (1)$$

where initial TPH and residual TPH were the TPH concentration of DC before and after treatment process, respectively.

2.4.2. Hydrodynamic parameters

In this study, the bubble-related hydrodynamic parameter was expressed as a ratio of interfacial area to velocity gradient (a/G). Bubble interfacial area (a) is a significant hydrodynamic parameter affecting the separation efficiency of hydrophobic materials. This parameter can be calculated from the ratio of total gas bubble surface to total volume in the system (including bubble gas and liquid phases), which can be rewritten in term of bubble formation frequency (f_B), liquid height (H_L), bubble rising velocity (U_B), bubble surface area (S_B), total volume of liquid and gas (V_{Total}) as shown in Eq. (2), where D_B, N_B, and V_B are bubble diameter, total amount of bubbles in the system, and bubble volume, respectively [22].

$$a=f_B\times\frac{H_L}{U_B}\times\frac{S_B}{V_{Total}}=f_B\times\frac{H_L}{U_B}\times\frac{\pi D_B^2}{AH_L+N_BV_B} \quad (2)$$

Note that the average diameter of the generated bubbles (D_B) was calculated by [35]:

$$d_b=382.52P_s^{-1.09} \quad (3)$$

where P_s was Saturated pressure (bar).

From Stoke's law, bubble rising velocity (U_B) was calculated by:

$$U_B=\frac{gD_B^2}{18\vartheta} \quad (4)$$

where ϑ is a kinetic viscosity of water (m^2/s) and g is gravitational acceleration (9.81 m/s^2)

Another significant parameter is a velocity gradient (G), which relates to the mixing or turbulence level in the system and can be determined using Eq. (5) [36].

$$G=\left(\frac{P}{\mu V}\right)^{0.5} \quad (5)$$

where P is the power required, μ is the dynamic viscosity, and V is the volume of the reactor.

Since FEST is a combined flotation–stirring process, the P value should be determined from the sum of P from stirring device and P from bubble generator. For P from the stirring process (P_{mixing}) with turbulent flow region (Re > 10,000), P_{mixing} can be defined as a function of impeller constant for turbulence flow (K_T), rotational speed (n), impeller diameter (D), and density of the liquid (ϱ) as expressed in Eq. (6). Note that the K_T value of the pitched blade turbine used in this work is 1.00 [37]. Meanwhile, the P from bubbles (P_{bubble}) can be estimated by pneumatic mixing parameters as shown in Eq. (7), where C_1 is the constant value of 3,904, Q_g is the air flow rate, and H is the depth from the water surface to the diffuser [36].

$$P_{mixing}=K_Tn^3D^5\varrho \quad (6)$$

$$P_{bubble}=C_1Q_g\log\left(\frac{H+10.4}{10.4}\right) \quad (7)$$

The TPH removal efficiency tends to be enhanced as increasing the chance of bubbles colliding and contacting to DC. Therefore, a collision efficiency (E_C) is applied for better understanding about the impact of hydrodynamic parameters on the performance of DC treatment. At intermediate flow condition, it can be determined using Eq. (8) [38]

$$E_C=\left(\frac{D_{DC}}{D_B}\right)^2\left(\frac{3}{2}+4\frac{Re^{0.72}}{15}\right) \quad (8)$$

where D_{DC} and Re is DC diameter and Reynolds number, respectively. Note that average D_{DC} is equal to 21.6 μm in this work.

Re is a dimensionless value that measures the ratio of inertial forces to viscous forces and describes the degree of turbulence in the system. The higher Re, the greater turbulence. Since FEST is a combined flotation–stirring process, the turbulence is caused by a stirring device (Re_{mixing}) and bubble generator (Re_{bubble}) and it can be calculated from Eq. (9) [39] and Eq. (10) [40].

$$Re_{mixing}=\frac{nD^2\varrho}{\mu} \quad (9)$$

$$Re_{bubble}=\frac{\rho U_BD_B}{\mu} \quad (10)$$

3. Results and Discussion

The results are divided into four main parts: (1) investigation of the FEST effectiveness on DC treatment, (2) optimization of the FEST process using RSM-CCD, (3) impact of hydrodynamic parameters (i.e., a/G) on the TPH removal efficiency, and (4) effect of saline water on DC treatment performance.

3.1. The Effectiveness of FEST as DC Treatment Process

In this part, the performance of an individual DC treatment process (stirring and DAF) on the TPH removal was firstly investigated. For the stirring process, three different rotational speeds (V_m), including 200, 400, and 600 rpm, were chosen for 1-h DC treatment with 10-min sampling interval. This experimental range was chosen because the lower V_m (<200 rpm) could not provide strong-enough turbulence, while the higher V_m (>600 rpm) provided too-strong turbulence, causing the rise of water level and then water flowed out of the reaction tank. As presented in Fig. 2a, the TPH removal increased with the increase of rotational speeds, achieving the maximum efficiency at V_m of 600 rpm and treatment time of 60 min.

For DAF, since few generated bubbles were observed in the flotation cell for the P_s less than 2 bars, so three different saturated pressures (P_s), including 2, 3, and 4 bars were chosen and operated under the same treatment time as stirring experiments. Figure 2b demonstrates that increasing P_s could improve the TPH removal efficiency, and the maximum TPH removal was obtained from P_s of 4 bar at 60 min treatment time.

Both results indicated that V_m and P_s significantly affected the TPH removal efficiency. Increasing V_m generated greater turbulence in the system and further reduced the attraction between TPH and DC surface. On the other hand, higher P_s provided smaller sizes and more numbers of microbubbles. These microbubbles related to a greater interfacial area which effectively enhanced the detachment of TPH from DC surface due to the hydrophobic interaction.

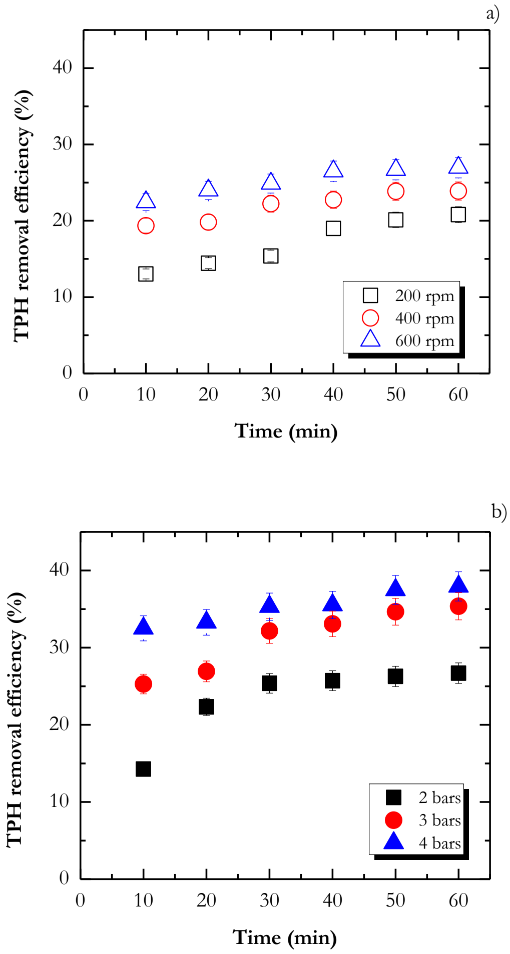

Fig. 2. TPH removal efficiency of a) stirring and b) DAF at different operating conditions.

Afterward, FEST was examined in order to maximize the TPH removal efficiency. The designed parameters, including P_s, V_m, and treatment time (t), were varied into three conditions: (1) 2 bars, 200 rpm, 20 min, (2) 3 bars, 400 rpm, 40 min, and (3) 4 bars, 600 rpm, 60 min. The results showed that FEST was more effective than a single process. As displayed in Fig. 3, the TPH removal of FEST was nearly 45% at P_s of 4 bars, V_m of 600 rpm, and t of 60 min, meanwhile that of a single stirring and DAF process was 25% and 38%, respectively. High turbulence caused the suspension of DC particles and helped the microbubbles easily attached and removed TPH from DC via the hydrophobic interaction. Additionally, higher turbulence generated a greater shear force that can accelerate the detachment of TPH from DC. Therefore, FEST could provide greater TPH removal efficiency compared to a single process due to the combined effects of generated microbubbles and mixing turbulence. To gain more understanding of FEST, the process was also optimized through the RSM-CCD as described in the next section.

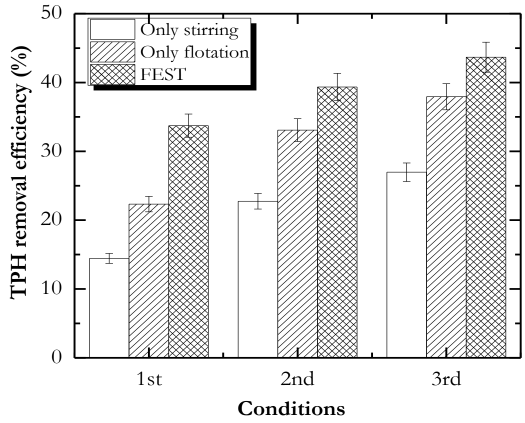

Fig. 3. TPH removal efficiency obtained from different processes and operating conditions.

3.2. Optimization of FEST through RSM-CCD

The RSM-CCD was applied for optimizing the FEST process for DC treatment. Three operational parameters of study included P_s (DAF), V_m (stirring), and t, indicated as an independent variable X_1, X_2, and X_3, respectively. The TPH removal efficiency was determined as a response variable.

The DC samples were washed following the experimental conditions designed by RSM-CCD shown in Table 2. The TPH removal efficiency obtained from the designed experiments were in the range of 27 to 44%. The relationship between the independent variables (P_s, V_m, and t) and the response variable (TPH removal efficiency) displayed a satisfied result of the model fit to the experimental data with a coefficient of determination (R^2) of 0.869 as expressed in Eq. (6).

$$\begin{aligned}\%\text{TPH removal} &= 10.6 + 7.93\,P_s + 0.0711V_m \\ &- 0.309t - 0.219\,P_s^2 - 0.000030\,V_m^2 + 0.00217t^2 \\ &- 0.01179\,P_sV_m + 0.0355\,P_st + 0.000112\,V_mt\end{aligned} \tag{11}$$

To consider the influence of each operational factor on the TPH removal efficiency, probability value (p-value) with 90% confidence level was determined. If p-value was less than or equal to 0.10 (p-value ≤ 0.10), it indicated that the factor significantly affects the TPH removal efficiency. Table 3 represents the analysis of variance (ANOVA) of TPH removal efficiency. The model p-value was 0.002, which was considered significant. However, only P_s, V_m, and P_sV_m were found to exhibit their significant role on the removal of TPH from DC using FEST. So, the Eq. (11) could be modified by removing the insignificant terms as expressed in Eq. (12)

$$\begin{aligned}&\%\text{TPH removal} \\ &= 10.6 + 7.93\,P_s + 0.0711V_m - 0.01179\,P_sV_m\end{aligned} \tag{12}$$

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Table 3. ANOVA of TPH removal efficiency.

Source	DF	Adj SS	Adj MS	p-value
Model	9	382.413	42.49	0.002
Linear	3	297.092	99.031	0
P_s	1	149.928	149.928	0.000
V_m	1	145.78	145.78	0.001
t	1	1.385	1.385	0.634
Square	3	35.17	11.723	0.173
Ps^2	1	0.694	0.694	0.736
Vm^2	1	20.511	20.511	0.088
t^2	1	10.828	10.828	0.2
2-ways interaction	3	50.151	16.717	0.088
$Ps*V_m$	1	44.515	44.515	0.019
Ps*t	1	4.022	4.022	0.423
V_m*t	1	1.614	1.614	0.608

To evaluate the precision and the competency of the model, the predicted TPH removal efficiency obtained from the model was plotted against the actual TPH removal efficiency obtained from the experiments (Fig. 4). It was shown that the data plots were in the error of ±10%. This could confirm the precision and the competency of the model.

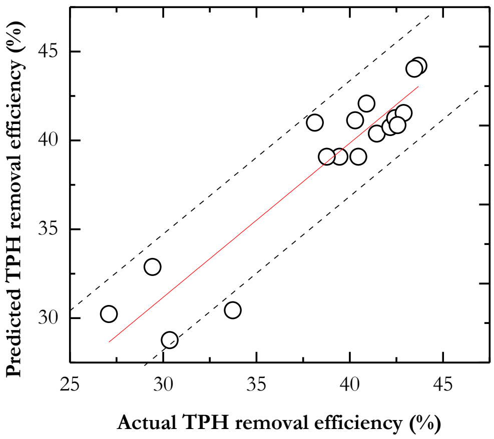

Fig. 4. The comparison between experimental results and predicted results of TPH removal efficiency.

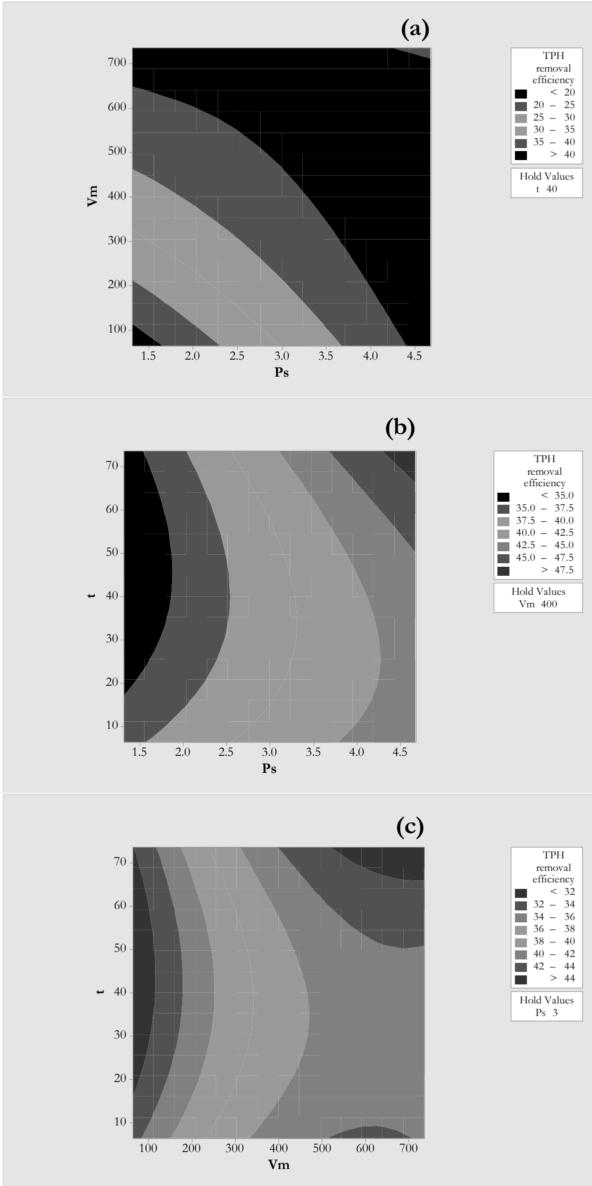

Fig. 5. Contour plots of the TPH removal efficiency as a function of operating factors: (a) P_s and V_m, (b) P_s and t, and (c) V_m and t.

The results are also presented as the contour plot (Fig. 5). These plots correlated well with the ANOVA results. According to the interaction between P_s and V_m shown in Fig. 5(a), more than 40% of TPH removal efficiency could be obtained when P_s was operated up to or higher than 4 bars with any V_m started from 200 rpm. The treatment time (t) was not significant as much as P_s and V_m. Operating at high P_s and high V_m provided quite similar range of TPH removal efficiency at any t between 20–60 minutes as shown in Figs. 5(b) – 5(c). According to the obtained mathematical model, the optimum operational conditions of FEST were P_s of 4 bars, V_m of 400 rpm, and t of 70 minutes, providing the maximum TPH removal around 50%. To verify the result, an additional experiment was then performed under the predicted optimal conditions. From the test, the TPH removal efficiency was achieved at 47.02%. This confirmed that the model attained from this work is reliable for predicting the TPH removal efficiency of FEST for DC treatment.

3.3. Impact of Hydrodynamic Parameters on the TPH Removal Efficiency

In this section, the impact of hydrodynamic parameters on the TPH removal efficiency was investigated and described in term of a/G. Since bubble interfacial area, a, is a bubble-based parameter, so the impacts of a/G on the TPH removal efficiency were investigated only in DAF and FEST. The calculated a/G values of FEST compared with DAF are displayed in Fig. 6. The a/G values of FEST seemed to be smaller than those of DAF since the G values of FEST were the sum of G caused by microbubbles in the flotation process together with G from stirring. At the quite similar a/G values, it is obvious that FEST showed a better performance on the TPH removal efficiency. The TPH removal of DAF and FEST at a/G around 10^5 s/m was 26.28% and 46.05%, respectively. This might be explained that the stirring caused well suspension and distribution of DC in the reactor, providing a greater chance of bubbles to attach DC particles and remove TPH from the DC surface via the hydrophobic interaction.

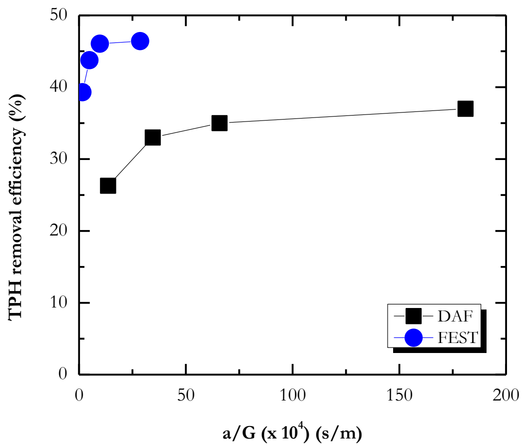

Fig. 6. The a/G values of DAF compared to FEST against the TPH removal efficiency.

Our assumption could be supported by an axial flow regime of a pitched-blade turbine together with the calculation of collision efficiency (E_c) of FEST and DAF. Note that E_c in this case refers to an instance of bubbles colliding against DC. Higher E_C tended to provide better TPH removal efficiency due to the increase of contact probability between DC and bubbles. Figure 7 presents the suspension and distribution of DC in the reactor caused by DAF and FEST process. It is clearly observed that there was more DC suspended throughout the reactor of FEST (Fig. 7b). This was because the pitched-blade turbine which was used as a mixing device in this work provided an axial flow regime or top-to-bottom mixing (Fig. 8b). The impeller could drive the DC particles and generated bubbles along with the fluid flowing in a downward angle until the fluid met the bottom of the reactor and was then deflected from the bottom. The fluid spread out over the bottom and flowed up along the walls before being drawn back to the impeller, leading to both DC and bubbles being re-circulated in the reactor [41]. This mechanism enhanced the chance of bubbles to collide and contact with DC surface as proven by hundred times greater E_C value of FEST (10.05) compared to that of DAF (0.10). For FEST, the combination of pneumatic mixing together with mechanical top-to-bottom mixing generated stronger turbulence (high Re), leading to greater E_C. Whereas, generated bubbles in DAF could not produce enough turbulence (lower Re) to make DC suspended and distributed well in the reactor, and bubbles also only flowed up vertically to the medium surface (Fig. 8a). This was the reason that higher TPH removal efficiency was obtained from FEST. Moreover, within the same process, the TPH removal efficiency could be improved at higher a/G.

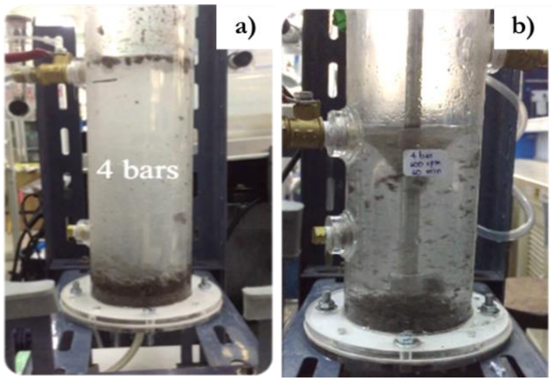

Fig. 7. The suspension and distribution of DC in the reactor using (a) DAF and (b) FEST.

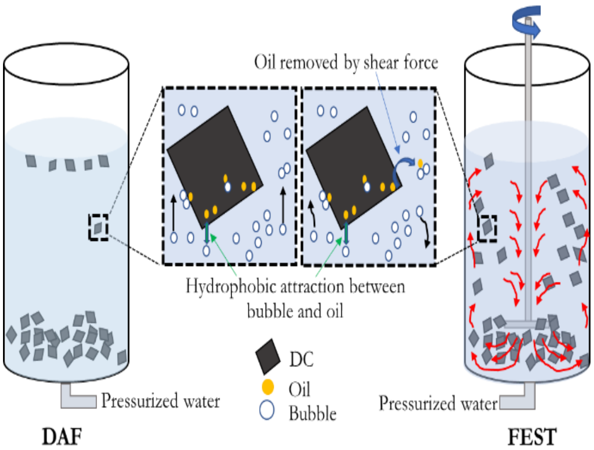

Fig. 8. Possible oil removal mechanisms for (a) DAF and (b) FEST.

3.4. Effect of Saline Water on DC Treatment

As saline water is the most convenient and cheapest water resource for offshore petroleum fields, the synthesized saline water with NaCl of 30 g/L, which referred to the salinity found in the Gulf of Thailand, was then tested as a treatment reagent to remove TPH from DC compared to tap water.

According to Fig. 9, the change of treatment medium from tap water to saline water showed the remarkable result. The TPH removal efficiencies were 37.94% and 46.42% for DAF and FEST using tap water, while 48.51% and 50.32% were respectively achieved from saline water. It signified that the DC treatment performance was improved due to the effects of NaCl solutions. The presence of NaCl in water kept the microbubbles separate from each other due to the increase of electrostatic force between bubbles, making them stable for a longer time [42]. Additionally, salinity (particularly cations) increased the thickness of electrostatic double layer [43], elevating the electrostatic repulsion between DC and oils and resulting in the greater DC treatment performance. Therefore, saline water could be another potential reagent for removing TPH from DC.

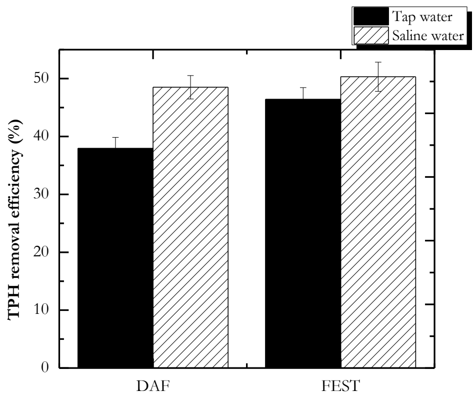

Fig. 9. The comparison of tap water and saline water as DC treatment medium.

4. Conclusions

In this study, a combined flotation–stirring process, called Flotation Enhanced Stirred Tank (FEST), was developed and proposed as a pretreatment technology for TPH-contaminated DC. According to RSM-CCD, the TPH removal around 47% was achieved at P_s of 4 bars, V_m of 400 rpm, and t of 70 minutes. Among these three parameters, P_s and V_m exhibited the strongest effects on the treatment performance. Furthermore, using saline water as a treatment medium instead of tap water could improve the TPH removal efficiency due to the increase of electrostatic repulsion between bubble–bubble and DC–TPH. These findings suggest the potential use of FEST as a pretreatment to remove TPH from DC prior to further treatment steps either for disposal or material recovery, e.g., chemical washing.

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