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Optimization of Washing Conditions and Adsorption Process for Petroleum Hydrocarbon Removal from Drill Cuttings Byproduct

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Abstract

Drill cuttings contaminated with total petroleum hydrocarbon (TPH) are generated from oil and gas exploration and production. The treatment of drill cuttings through washing process has been applied due to its high efficiency and less energy consumption. However, this process generates washing solution containing TPH as a petroleum waste, which requires further management. Therefore, the purpose of this study is to optimize the drill cuttings washing process using ethyl lactate (EL) as a green washing agent. Afterwards, the spent washing solution was purified through an adsorption process using two adsorbents: coal-based and coconut shell-based granular activated carbon (GAC). The result showed that liquid-to-solid (L/S) ratio was the most influential factor affecting the removal of TPH from drill cuttings by EL. The higher volume of EL used, the higher TPH extraction capacity. The mixing speed followed by washing time and rinse-to-solid (R/S) ratio also significantly affected the TPH removal efficiency, whereas the rinsing time was statistically insignificant. The optimum washing conditions were L/S ratio of 10 mL/g, washing time of 20 min, mixing speed of 100 rpm, R/S ratio of 10 mL/g, and rinsing time of 1 min, from which the TPH removal of 87.6% was achieved. For the adsorption experiments, coal-based GAC performed better in adsorbing TPH from the spent washing solution compared to coconut shell-based GAC. Thus, the overall results suggest that EL was a promising agent for removing TPH from drill cuttings, and coal-based GAC could be a potential adsorbent for spent EL purification and recovery.

Keywords : Drill cuttings; ethyl lactate; washing process; total petroleum hydrocarbon; activated carbon

Introduction

In oil and gas exploration and production, drilling is a key process conducted by the aid of drilling mud, which is a synthetic oil containing several additive chemicals for controlling and stabilizing the borehole. During drilling operation, a drill bit is used to grind the rocks while drilling mud is pumped down the well through the hollow drill string and returned carrying the drill cuttings (i.e., crushed rock pieces). Once drill cuttings are taken to the surface, the drilling mud is then separated through the solid-control equipment for reusing in drilling process and drill cuttings become wastes. Drill cuttings contaminated with drilling mud and petroleum hydrocarbon are recognized as hazardous wastes. Generally, a single drilling well produces more than a thousand tons of drill cuttings [1, 2]. Therefore, the proper management and disposal of drill cuttings are necessarily required. Currently, washing process has been widely applied for remediating hydrocarbon-contaminated soil due to its simple and rapid operation, less power consumption, and high removal efficiency [3]. Washing process can be an in-situ or ex-situ remediation process in which an extracting agent is used to remove the contaminants from soils. Green solvent, ethyl lactate (EL) has drawn much attention in hydrocarbon-polluted soil washing process due to its high efficiency, biodegradability, and low toxic properties [4]. Hence, EL is expected to exhibit high removal efficiency for hydrocarbon liberation from drill cuttings. However, several factors might affect the washing performance including liquid-to-solid ratio, mixing rate, and washing time [5]. Optimizing these parameters is necessary, and design of experiment (DOE) could be a helpful statistical tool for achieving the optimal washing conditions [6].

Despite cleaned drill cuttings as a product, the process remains washing waste (or washing solution) holding high concentration of solvent and hydrocarbon pollutants. Thus, it is essential to recover and reuse the washing solution in order to minimize the chemical waste volume as well as the overall operational cost. Various treatment techniques have been studied on hydrocarbon removal from non-aqueous solutions such as membrane filtration [7], distillation [8], and solvent extraction process [9]. Nevertheless, several problems are commonly found from these methods such as high treatment cost, high energy consumption, and complex operation [10]. Besides these technologies, an adsorption process could be another technique to remove oil from the spent washing solution [11, 12]. Moreover, the process exhibits its easy operation, low maintenance cost, and small footprint, compared to other treatment processes [13]. In adsorption processes, activated carbon is the most commonly used adsorbent for removing a variety of organic compounds due to its low cost, local availability, easy maintenance, and regeneration potential [14]. Furthermore, it is produced from the natural carbonaceous materials including coal, peat, lignite, and wood by physical and chemical activation, which generates high porosity and large surface area for the solute adsorption [15]. Therefore, this work aims to optimize the washing conditions for total petroleum hydrocarbons (TPH) removal from drill cuttings using EL as an extracting agent. Subsequently, the washing solution is purified through an adsorption process, in which coal-based and coconut shell-based granular activated carbon are investigated in order to determine the optimal adsorbent type for solvent recovery.

Materials and Methods

Drill Cuttings

Drill cuttings samples were collected from offshore petroleum exploration and production. The samples were prepared by air-dried for two days at room temperature before screening by a standard sieve #7 (equivalent to 2.8 mm) to remove large grains as it is hard to react with other constituents. Physicochemical properties of drill cuttings including heavy metals, chloride, pH, electrical conductivity (EC), cation exchange capacity (CEC), TPH concentration, bulk density, moisture content, organic matter, and particle size distribution, were analyzed.

Design of Experiment and Data Analysis

Response Surface Methodology with Central Composite Design (RSM-CCD) was used to design the experimental conditions and investigate the effects of the washing parameters on the treatment performance. There are several influenced factors on the TPH removal from drill cuttings washing process including liquid-to-solid ratio (L/S), washing time, mixing speed, rinse-to-solid ratio (R/S), and rinsing time. The low, middle and high levels of each factor were coded as -1, 0 and +1, respectively as shown in Table 1. According to five factors with three levels each, the 32 experimental runs consisting of 16 cube points, 10 axial points, and 6 replicated at the center points, were provided for performing the experiments. The TPH removal percentage calculated by Equation (1) was a response variable to be optimized.

$$\text{TPH removal (\%)} = \frac{C_o - C_e}{C_o} \times 100 \qquad (1)$$

Where C_o is the initial TPH concentration (mg/kg) and C_e is the residual TPH concentration (mg/kg).

Analysis of variance (ANOVA) was also employed to validate the predicted model and evaluate the statistical effect of each factor. The experimental results were analyzed using Minitab software and fitted into the empirical second-order polynomial equation as given in Equation (2) in order to optimize the drill cuttings washing conditions [6]. The model quality is estimated by the correlation coefficient (R^2) and the result analysis is carried out using F-test and p-value with 95% confidence level of statistical analysis.

$$y = \beta_0 + \sum_{i=1}^{5}\beta_i x_i + \sum_{i=1}^{5}\beta_{ii} x_i^2 + \sum_{i=1}^{4}\sum_{j=i+1}^{5}\beta_{ij} x_i x_j \qquad (2)$$

Where y is the predicted response by the model, x_i and x_j are the independent factors, β_o is a constant, β_i is linear coefficients, β_{ii} is second-order coefficients, and β_{ij} is interaction coefficients.

Table 1 Experimental design for washing process

Factors	Unit	Factor levels		
		-1	0	+1
L/S ratio	mL/g	3.0	6.5	10.0
Washing time	min	1.0	10.5	20.0
Mixing speed	rpm	50	100	150
R/S ratio	mL/g	1.0	5.5	10.0
Rinsing time	min	1.0	5.5	10.0

Washing Procedures

Two grams of drill cuttings were added with a pre-determined EL volume, and the washing process was conducted under room temperature following the conditions obtained from RSM-CCD using a shaker. The washing solution was drained out after settling down for a certain time. Then, drill cuttings were rinsed with tap water by shaking at the previous speed of EL washing. Once rinsing water was removed, cleaned drill cuttings were dried in the oven at 60°C for 6 hours and analyzed for the residual TPH concentration through solvent extraction and detection by gas chromatography equipped with flame ionization detector (GC-FID, Agilent 6890N, USA), following the U.S. EPA 8015 method [16].

Adsorbent Materials

To remove TPH from the washing solution, two types of commercial granular activated carbon (GAC), i.e., coal-based (0.60-2.36 mm) and coconut shell-based (1.18-2.36 mm) were employed. The GAC samples were washed four times with deionized water and then dried at 60°C overnight prior to use.

Adsorption Experiment

In this part, the synthetic solutions representing of the washing solution generated from drill cuttings washing process were prepared by dissolving diesel oil in EL at the calculated amount in order to get three solution concentrations. The adsorption experiment was conducted in a 150 mL glass vial screw-top holding 50 mL of washing solution and 0.2 g of GAC following the optimum dosage reported previously [13]. The bottles were therefore sealed and agitated in a water bath shaker under a speed of 150 rpm at 25°C for 24 hours to reach the equilibrium. After adsorption, the solutions were filtrated and analyzed for the residual TPH concentration using UV-Vis spectrophotometer at 260 nm wavelength.

Results and Discussions

Drill Cuttings Characterization

The characteristics of offshore drill cuttings are displayed in Table 2. The drill cuttings sample contained an acceptable level of heavy metals except for the arsenic concentration, which was found beyond the limit of residential and agricultural soil quality (3.9 mg/kg) based on the standard of Thailand's Pollution Control Department (PCD). Iron (1.73%) and calcium (0.06%) were two dominant elements found in drill cuttings similar to general soils. In addition, the CEC of 7.29 cmol/kg was measured and fallen in a range of 6-12 cmol/kg, indicating that the drill cuttings were low reactive in nature [17]. The TPH concentration of approximately 236,000 mg/kg was detected and regarded as the initial TPH concentration used in this study. The particle size distribution analysis showed that the drill cuttings particles mostly existed as silt, sand, and clay, respectively. Moreover, a specific surface area up to 389.8 m^2/kg was measured from the sample.

Table 2 Physicochemical properties of drill cuttings

Parameters	Value	Parameters	Value
Fe (mg/kg)	17,330	Chloride (mg/L)	271
Ca (mg/kg)	596	pH	8.0
Hg (mg/kg)	0.521	EC (mS/cm)	0.106
Pb (mg/kg)	30.870	CEC (cmol/kg)	7.29
As (mg/kg)	25.550	TPH concentration (mg/kg)	236,000
Cd (mg/kg)	0.183	Bulk density (g/cm^3)	1.24
Cr (mg/kg)	25.430	Moisture content (%)	5.0
Mg (mg/kg)	4,685	Organic matter (%)	15.31
Mn (mg/kg)	1,254	Sand (2000 – 50 µm) (%)	12.88
Ni (mg/kg)	23.430	Silt (50 – 2 µm) (%)	83.45
Zn (mg/kg)	60.490	Clay (< 2 µm) (%)	3.67

Optimization of Washing Conditions

Based on the experimental results, the optimum washing condition was found at L/S of 10 mL/g, washing time of 20 min, mixing speed of 100 rpm, R/S ratio of 10 mL/g, and rinsing time of 1 min, in which the maximum TPH removal efficiency of 87.6% was achieved. The validity of the model is evaluated by ANOVA and the second-order polynomial equation of the TPH removal efficiency as a function of L/S ratio (A), washing time (B), mixing speed (C), R/S ratio (D), and rinsing time (E) are given in Table 3 and Equation (3), respectively. According to the F-test with 95% confidence level, the "p-value" less than 0.05 designates the parameter statistically significant. As shown in Table 3, the F-value of 12.91 and the p-value well below 0.05 indicate that the model prediction was statistically significant. Moreover, the linear terms (A, B, C, D), square term (C^2), and 2-way interaction terms (AB, AD, BC, DE) had significant impact on the washing performance since the p-values were less than 0.05. It can be observed that L/S ratio had the highest influence on the TPH removal efficiency followed by mixing speed, washing time, and R/S ratio, respectively. The large p-value of Lack-of-Fit (0.177) also indicates that the experimental error was not statistically significant. Additionally, the fitness of the model R^2 = 0.9591 was obtained.

$$\begin{aligned}\%\text{TPH removal} = {} & 32.78 + 5.14A - 0.426B + 0.565C + 0.65D - 1.14E - 0.228A^2 + 0.0398B^2 \\ & - 0.003738C^2 + 0.0459D^2 + 0.1313E^2 - 0.0727AB + 0.00828AC - 0.1093AD \\ & + 0.0676AE + 0.00406BC + 0.0002BD - 0.0271BE + 0.00562CD + 0.00157CE - 0.1199DE \end{aligned} \quad (3)$$

Table 3 ANOVA result for the TPH removal percentage

Source	DF	Adj SS	Adj MS	F-value	p-value	
Model	20	1870.06	93.503	12.91	0.000	Significant
Linear	5	1098.20	219.640	30.32	0.000	
A: L/S ratio	1	889.72	889.717	122.82	0.000	
B: Washing time	1	62.53	62.533	8.63	0.013	
C: Mixing speed	1	100.02	100.017	13.81	0.003	
D: R/S ratio	1	45.38	45.379	6.26	0.029	
E: Rinsing time	1	0.55	0.555	0.08	0.787	
Square	5	375.51	75.303	10.39	0.001	
A^2	1	19.24	19.235	2.66	0.131	
B^2	1	31.79	31.788	4.39	0.060	
C^2	1	214.94	214.936	29.67	0.000	
D^2	1	2.12	2.125	0.29	0.599	
E^2	1	17.40	17.401	2.40	0.149	
2-Way Interaction	10	395.34	39.534	5.46	0.005	
AB	1	93.36	93.364	12.89	0.004	
AC	1	33.61	33.611	4.64	0.054	
AD	1	47.44	47.438	6.55	0.027	
AE	1	18.13	18.126	2.50	0.142	
BC	1	59.48	59.483	8.21	0.015	
BD	1	0.00	0.001	0.00	0.992	
BE	1	21.41	21.414	2.96	0.114	
CD	1	25.58	25.578	3.53	0.087	
CE	1	2.00	1.995	0.28	0.610	
DE	1	94.33	94.333	13.02	0.004	
Error	11	79.69	7.244			
Lack-of-Fit	6	59.21	9.868	2.41	0.177	Not significant
Pure Error	5	20.48	4.096			
Total	31	1949.74				

The predicted responses (%TPH removal) given by Equation (3) were plotted against the experimental values as displayed in Figure 1 to evaluate the precision and competency of the model. The high correlation R^2 = 0.9574 indicated a great goodness-of-fit of the model to the experimental data. Additionally, the main effects plot of each factor and the surface plot of interactive effects were illustrated in Figures 2 and 3, respectively. The results exhibited that L/S ratio was the main influential factor on the TPH removal efficiency as confirmed by the least p-value shown in Table 3. The increase of L/S ratio from 3 to 10 had a positive effect on the washing performance, indicating that the amount of TPH removed was directly influenced by the volume of EL added. This can be explained that the higher L/S ratio, the greater capacity of EL to mobilize the TPH molecules existing in drill cuttings.

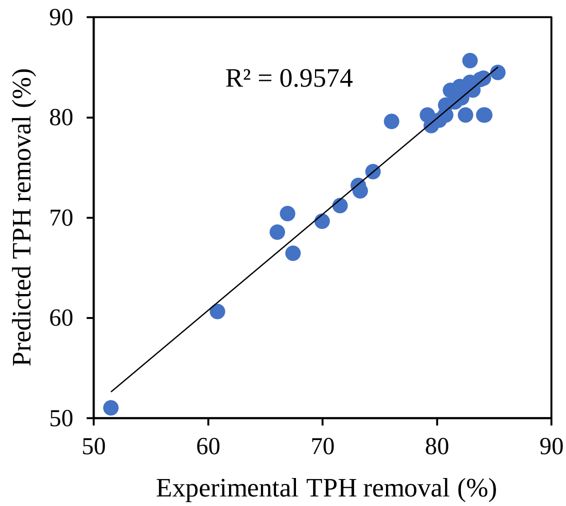

Figure 1 Experimental values against predicted values of the TPH removal efficiency

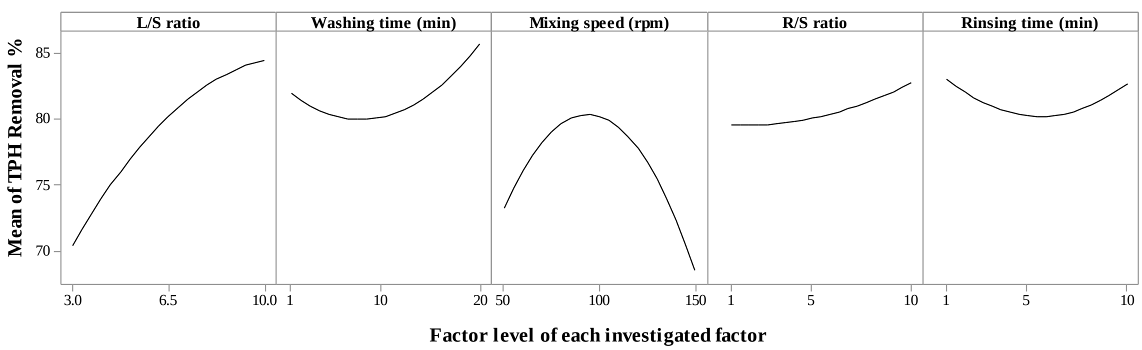

Figure 2 Main effects plot of each factor

Despite the high impact of L/S ratio, the effects of mixing speed on TPH removal could not be neglected. An effective washing performance could be reached at a velocity that the surface area of drill cuttings was completely contacted with EL. It can be observed in Figure 2 that the TPH removal efficiency has a tendency to increase with increasing mixing speed until it raised to 100 rpm. However, when the speed continued to increase more, the TPH removal was dropped significantly. This result indicates that a mixing speed of 100 rpm was adequate to obtain the maximum TPH extraction potential. The excessive mixing speed might cause the extracted TPH molecules to liberate from the EL and re-attach to the drill cuttings particles, resulting in reduced TPH removal. For the washing time, it corresponds to the degree of mass transfer rate between TPH and EL. The process could be enhanced by prolonging washing time to 20 min.

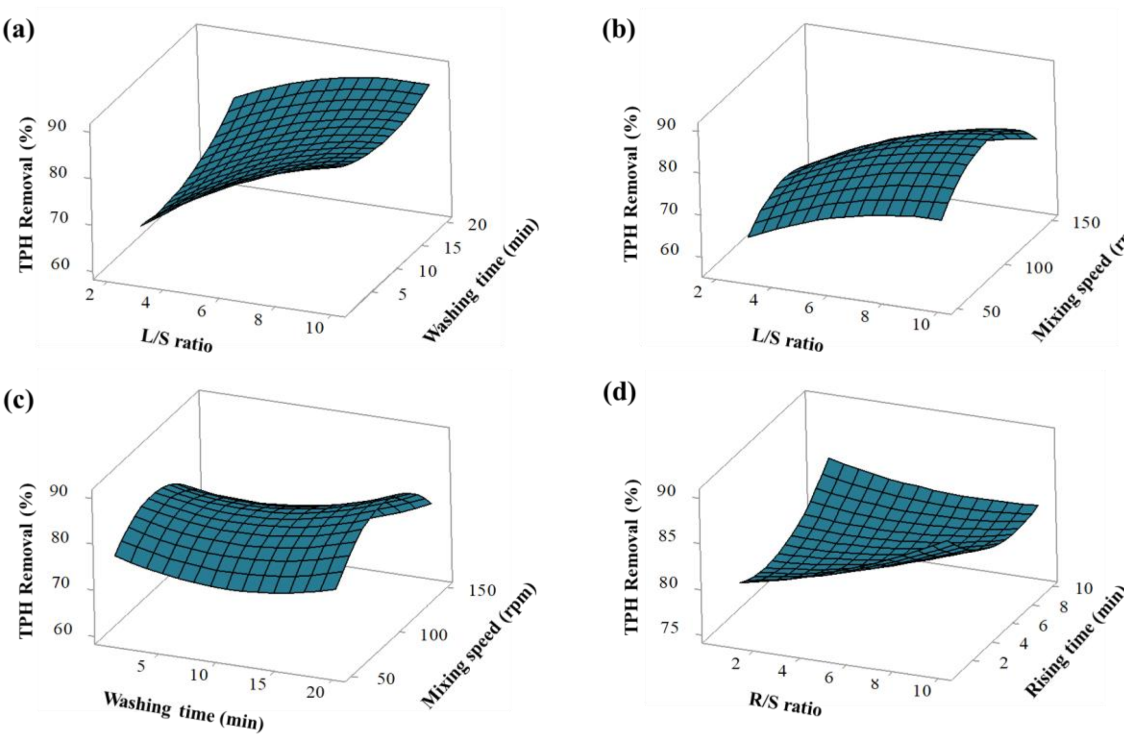

Figure 3 Surface plots of (a) L/S ratio and washing time, (b) L/S ratio and mixing speed, (c) washing time and mixing speed, and (d) R/S ratio and rinsing time against %TPH removal

Another finding to point out is the effect of R/S ratio, rinsing drill cuttings with water is required in order to remove the remaining TPH and EL solution adhered to drill cuttings. Since EL contains both polar and nonpolar properties, it is completely miscible in hydrocarbons and water [18]. Therefore, after water rinsing, the cleaned drill cuttings would become more purified. Based on the main effects plot, the TPH removal was improved as increasing R/S ratio from 1 to 10 mL/g. Hence, the higher R/S ratio resulted in a larger volume of water used for liberating the residual solution from drill cuttings. Nevertheless, the rinsing time was found statistically insignificant as the p-value was higher than 0.05 (Table 3). A prolonged rinsing time normally exhibits better exposure between drill cuttings and washing solution. However, in this experimental result, an extended rinsing time from 1 to 10 min did not noticeably improve the TPH removal. This is probably due to the emulsified mixture that formed from the co-exist of TPH, EL, and water. The longer rinsing time might produce more emulsions which were difficult to remove from drill cuttings, and thus resulting in less TPH removal efficiency.

Characterization of Adsorbents

The chemical element composition of coal-based GAC and coconut shell-based GAC were analyzed through energy dispersive x-ray spectrometer (EDS) as shown in Table 4. Both coal-based and coconut shell-based GAC contained carbon and oxygen as the main compositions. According to the BET analysis, the specific surface area of 850 m^2/g and 1000-1130 m^2/g were measured from the coconut shell-based and coal-based GAC, respectively.

Table 4 EDS analysis of coal-based and coconut shell-based GAC

GAC	Element analysis (wt%)				
	C	O	Ca	K	Other
Coal	84.61	12.19	1.43	-	1.77
Coconut shell	90.13	6.47	0.82	2.58	-

Adsorption of TPH from Washing Solution

Considering on TPH adsorption performance, Figure 4 clearly demonstrates that the TPH removal efficiencies of coal-based and coconut shell-based GAC decreased from 33.3% to 13.7%, and from 20.2% to 6.1%, respectively, as increasing TPH concentration from 1600 mg/L to 6300 mg/L. Moreover, the amount of TPH adsorbed with coal-based GAC was higher than that of coconut shell-based for all concentrations. As presented in the adsorbent characteristics, the coal-based GAC holding larger specific surface area which could enhance the interaction between TPH molecules and adsorbent. Therefore, at the TPH concentration of 1600 mg/L, the coal-based GAC could remove TPH from the washing solution about 33.3%, whereas only 20.2% of TPH was removed by coconut shell-based GAC. Additionally, the visual appearance of the washing solution before and after adsorption experiment by coconut shell-based and coal-based GAC is also displayed in Figure 5. The washing solution changed from yellow to clear solution since the TPH in solution had been removed by GAC. Furthermore, the solution adsorbed by coal-based GAC was much clearer, indicating the higher TPH removal than that of coconut shell-based GAC. Thus, the coal-based GAC was considered as an effective adsorbent for EL recovery. However, the TPH removal of the spent EL could be enhanced only 33.3%. The process should be further optimized to improve the adsorption capacity and TPH removal efficiency. In addition, the regeneration and reuse of the exhausted adsorbent contaminated with the petroleum hydrocarbon should be considered in order to minimize the hazardous wastes as well as the overall treatment cost.

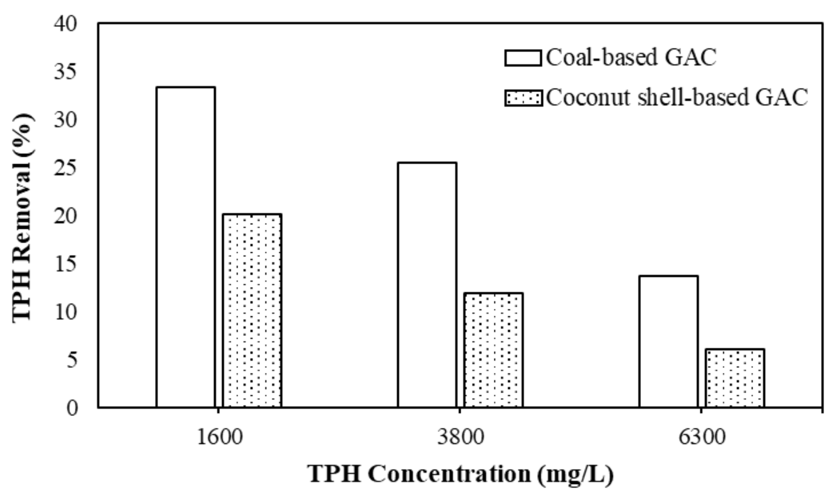

Figure 4 TPH removal efficiency at the different concentrations for both GAC

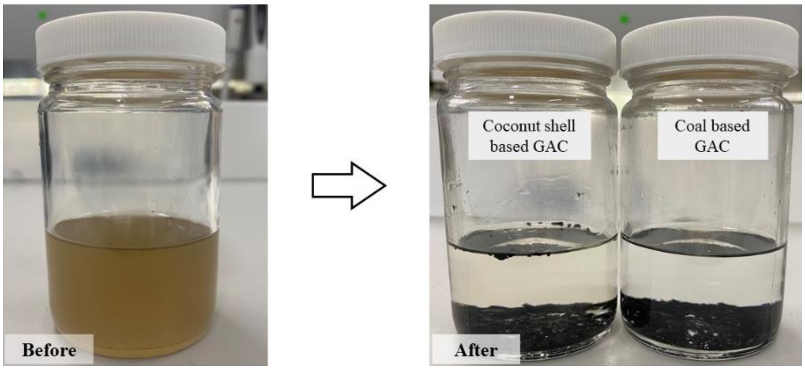

Figure 5 Appearance of washing solution before and after adsorption with GAC

Conclusions

The treatment of drill cuttings using EL washing process and subsequent adsorption of TPH from washing solution was investigated. The result indicated that drill cuttings washing performance was largely influenced by L/S ratio followed by mixing speed, washing time, and R/S ratio. The maximum TPH removal efficiency of 87.6% was achieved at the optimum conditions: L/S ratio of 10 mL/g, washing time of 20 min, mixing speed of 100 rpm, R/S ratio of 10 mL/g, and rinsing time of 1 min. Moreover, the coal-based GAC exhibited better performance for removing TPH from the spent washing solution compared to coconut shell-based GAC for all solution concentrations. It was due to the coal-based GAC containing higher specific surface area than that of coconut shell-based GAC. Therefore, coal-based GAC is recommended as an alternative material to recovery the spent EL generated from drill cuttings washing to reduce the chemical waste volume as well as the treatment cost.

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