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TPH removal from oily wastewater by combined coagulation pretreatment and mechanically induced air flotation

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ABSTRACT

The objective of this study was to combine coagulation and mechanically induced air flotation in order to determine the optimum operational condition for the removal of total petroleum hydrocarbons (TPHs) from oily wastewater. A mixture of water, gas oil, and emulsifier was used to generate a stable emulsion as the simulated wastewater. Aluminum sulfate, ferric chloride, and polyaluminum chloride were employed as coagulants to destabilize the emulsions, and the subsequent separation of hydrocarbons was carried out in a mechanical flotation cell. Considering the multiplicity of factors in both stages of coagulation and flotation, the effects of each factor were studied using Taguchi design of experiments technique, and the optimum conditions were revealed. The results showed that at the optimal conditions, about 93% TPH removal was achieved by addition of 50 mg/l aluminum sulfate at pH 4, flotation time of 10 min, impeller speed of 1,000 rpm, and airflow rate of 4.5 l/min.

Keywords: Coagulation; Mechanically induced air flotation; Oil-in-water emulsion; Taguchi method; TPH

1. Introduction

Oil production and refining processes generate a large amount of oily wastewaters [1], which are one of the most serious environmental issues faced by the oil-related industries [2]. These wastewaters may contain emulsified oil in water as a main pollutant [3]. Oil-in-water emulsions, which can be formed by the emulsifying agents [4], are quite permanent in the environment and often resistant to be separated by conventional gravitational processes [5,6]. The discharge of oily wastewaters directly to the environment has been controlled according to the limits regulated by environmental agencies. In addition to environmental restrictions, economic interest may cause oil industries to reuse of such wastewaters [7].

Various physical, chemical, and biological techniques are used for the treatment of oily wastewaters before being reused or discharged to the environment [8]. Among these techniques, flotation has been demonstrated as an effective and applicable method for

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removal of oil and grease as well as suspended solids from wastewaters. In flotation, rising gas bubbles cause to float very small oil droplets by being attached to the surface of droplets [3]. Consequently, floating oil particles are skimmed off from the liquid surface. Flotation systems can be essentially classified based on the method used for bubble generation [9]. A type of flotation system offering the advantages of significantly lower capital and operational cost, smaller space requirements and high separation efficiency for oil-in-water emulsion separation is induced (dispersed) air flotation in which air bubbles are formed and introduced into wastewater mechanically by the combination of a high speed rotating impeller and an air injection system [10,11].

Coagulation pretreatment using various chemical reagents such as the aluminum and ferric salts, known as coagulant, can destabilize the oily phase by reducing the charge on the surface of oil droplets, functioning to break the emulsion and enhance droplet coalescence [12]. While coagulation is coupled with induced air flotation, separation of emulsified oil droplets from wastewater becomes more effective [13] since the coagulated droplets can easily absorb or entrap air bubbles [14].

In order to optimize operating conditions of the treatment process, and considering the multiplicity of factors in both stages of coagulation and flotation, design of experiments (DOE) can be employed to study all combinations of the factors and their interactions included in experimental study [15]. Taguchi method, a form of DOE which utilizes fractional factorial designs, can be used to study the influence of design factors with a minimum number of experiments [16,17]. Further, the results of the Taguchi experiments are analyzed to establish the optimum condition, identify the significant factors and interactions, and estimate the response under the optimum conditions [18].

Although mechanically induced air flotation has been widely used in the mineral processing and wastewater treatment, but according to our knowledge, only a few literatures are available on the combined coagulation and mechanical flotation for the separation of oil from oily wastewater without using any the flotation collectors or frothers. Furthermore, no one has ever studied the effect of various operating conditions on coagulation and mechanical flotation performance for emulsified oil separation simultaneously by using an experimental design approach.

The objective of this study is to evaluate effects of various factors involved in coagulation process combined with mechanically induced air flotation system on the removal efficiency of total petroleum hydrocarbons (TPHs) from oily wastewater. Accordingly, the effects of factors influencing on emulsion stability in the coagulation process such as type and concentration of coagulants and pH and the effects of operational factors in flotation process including flotation time, impeller speed, and airflow rate were investigated by using the Taguchi experimental design method. The experimental results were statistically investigated by analysis of variance (ANOVA) to evaluate the effects and relative importance of controlling factors and significant interactions on the response.

2. Experimental

2.1. Materials and methods

The gas oil was selected as the oil phase to prepare simulated wastewater, since it is not evaporated during the tests (with boiling point about point ranging from 230 to 400˚C), and its solubility in water is negligible (with number of carbon atoms about C13-C25) [19]. The nonionic surfactant Tween 80 (Polyoxyethylene sorbitan monooleate) used as an emulsifier was purchased from Oleon, Belgium. Three different coagulant agents, i.e. aluminum sulfate ($Al_2(SO_4)_3$, $16H_2O$), ferric chloride ($FeCl_3$, $6H_2O$), and polyaluminum chloride (PACl) with industrial purity were examined. Tap water was used to prepare all the oil-in-water emulsions. The pH of oil-in-water emulsions was adjusted by addition of a 5 M solution of hydrochloric acid (HCl) and sodium hydroxide (NaOH) obtained from Merck, Germany. All experiments were conducted at ambient temperature.

The oil-in-water emulsions were prepared by adding a predetermined amount of gas oil and Tween 80 to 1.5 l of tap water placed in a 2 l glass container. All the emulsions initially contained gas oil concentrations of 500 mg/l. The concentration of Tween 80 added was also adjusted to 0.002 wt.%, a bit above its critical micelle concentration which is about 0.0013 wt.% [20]. The mixture was stirred with a mechanical stirrer at 1,500 rpm for 15 min. This procedure could form a stable emulsion with average oil droplet size of about 20 μm as measured by an optical microscope (Olympus BH2, Japan).

The flotation experiments were conducted using a Denver D-12 mechanical flotation machine equipped with a 2 l cell. Air bubbles are formed by introducing the air directly into the system through a rotating impeller (rotor). The impeller speed can be varied from 0 to 3,000 rpm. The impeller forces the liquid through the disperser openings and creates a negative pressure within the standpipe surrounding the impeller shaft. It pulls the air into the liquid through an air suction opening on the standpipe, thereby creating fine bubbles via the disperser. In this research, an air pump with a range of 0–6 l/min equipped with a flowmeter was used to control the air entering the cell so that the air pump was connected to the air suction opening on the standpipe using a silicone hose. Therefore, any air bubbles were not formed before the air pump was started.

For performing the experiments, the prepared emulsion was transferred to the flotation cell. A desired amount of coagulant was subsequently added to the emulsion after the pH adjustment and agitation occurred at 600 rpm for 3 min. After that, the impeller speed was adjusted to the desired speed, and flotation process was carried out by inducing the required airflow into the cell for a specified time interval. All flotation experiments were repeated two times.

In this research, turbidity was used to determine the TPH of emulsions, since the preliminary experiments showed that there is a relationship between their TPH and turbidity. In order to obtain a standard reference line, the TPH and turbidity measurements of several emulsion samples with different known gas oil concentrations were performed using an Agilent 6890 N Gas Chromatography with a flame ionization detector (GC-FID) and a HACH 2100 N turbidimeter with the highest sensitivity of 0.1 NTU, respectively. Therefore, the standard line (TPH vs. turbidity) plotted with correlation coefficient (*r*2) of 0.99 was used to determine the TPH of each emulsion sample based on a measured turbidity. The removal efficiency of TPH by flotation process was calculated according to the following equation:

$$\text{Re}\% = [(C_0 - C)/C_0] \times 100 \quad (1)$$

where C_0 and C are the TPH concentrations of emulsions before and after flotation, respectively.

2.2. Experimental design

In this research, a Taguchi experimental design based on a L_{27} orthogonal array (OA) involving six factors, namely coagulant type and concentration, pH, flotation time, impeller speed and airflow rate at three levels, was employed. The factor levels were selected according to their effective ranges on preliminary experiments. In addition, the interactions between coagulant type and pH as well as coagulant type and concentration were investigated, since they were found to be effective on the response based on the primary data analysis. The percent of TPH removal was selected as the response variable. The studied factors and their levels are listed in Table 1.

Table 1
The studied factors and their levels in experiments

Factors	Levels		
	1	2	3
Coagulant type	PACl	Alum	$FeCl_3$
pH	4	6	8
Coagulant concentration (mg/l)	20	50	80
Flotation time (min)	2	6	10
Impeller speed (rpm)	600	800	1,000
Airflow rate (l/min)	1.5	3	4.5

Each trial was repeated twice at the similar conditions to evaluate the signal to noise ratio (S/N). S/N analysis specifies a robust set of operational conditions from variations within the results [18], leading to a process performance which is minimally affected by external sources of variability [21]. In S/N analysis, the response quality is represented as the ratio of the controllable factors (signal) compared to uncontrollable factors (noise) [22]. The experimental designs and analysis were performed using Minitab 15 software.

3. Results and discussion

3.1. Taguchi results

The design of the L_{27} OA and corresponding S/N ratio for each trial is presented in Table 2. Effect of each factor level on the response can be independently determined by calculating the average S/N ratio of all trials in which that level of the factor is applied [20,23]. In this study, since a higher treatment efficiency is desired, the "larger is better" criterion was preferred for the S/N ratio. Thus, the ultimate goal of the experiments was to maximize the S/N ratio. The S/N ratio for "larger is better" criterion was calculated by the following equation [24]:

$$\frac{S}{N} = -10\log\left(\frac{1}{n}\sum_{i=1}^{n}\frac{1}{y_i^2}\right) \quad (2)$$

where y_i and n are the response value (i.e. percentage of TPH removal) and the number of repetition for a trial, respectively.

The S/N ratios computed for each level of factors are presented in Table 3 and Fig. 1. Corresponding to the "larger is better" criterion, the levels of factors with higher S/N ratios give more desirable performance characteristics, and the level maximizing the

Table 2
Experimental results for TPH removal and corresponding S/N ratios

Trial	Coagulant type	pH	Coagulant concentration	Flotation time	Impeller speed	Airflow rate	TPH removal		S/N ratio
							Run1	Run2	
1	1	1	1	1	1	1	54.4	53.8	34.7
2	1	1	2	2	2	2	71.7	69.8	37.0
3	1	1	3	3	3	3	64.1	59.6	35.8
4	2	2	1	1	1	2	51.9	54.8	34.5
5	2	2	2	2	2	3	74.9	79.3	37.7
6	2	2	3	3	3	1	68.5	70.7	36.8
7	3	3	1	1	1	3	40.8	43.8	32.5
8	3	3	2	2	2	1	65.6	61.2	36.0
9	3	3	3	3	3	2	54.3	57.4	34.9
10	2	3	1	2	3	1	69.9	72.7	37.1
11	2	3	2	3	1	2	74.7	72.1	37.3
12	2	3	3	1	2	3	63.1	68.6	36.3
13	3	1	1	2	3	2	77.9	80.0	37.9
14	3	1	2	3	1	3	69.7	70.9	36.9
15	3	1	3	1	2	1	53.4	55.0	34.7
16	1	2	1	2	3	3	63.4	67.4	36.3
17	1	2	2	3	1	1	62.8	66.7	36.2
18	1	2	3	1	2	2	34.6	31.1	30.3
19	3	2	1	3	2	1	77.9	74.3	37.6
20	3	2	2	1	3	2	65.3	70.3	36.6
21	3	2	3	2	1	3	66.1	69.9	36.6
22	1	3	1	3	2	2	73.8	77.0	37.5
23	1	3	2	1	3	3	54.1	59.5	35.1
24	1	3	3	2	1	1	51.9	51.6	34.3
25	2	1	1	3	2	3	92.5	93.3	39.4
26	2	1	2	1	3	1	78.0	77.5	37.8
27	2	1	3	2	1	2	87.1	86.6	38.8

Table 3
S/N ratio for levels of each factor

	Factors					
	Coagulant type	pH	Coagulant concentration	Flotation time	Impeller speed	Airflow rate
Level 1	35.2	37.0	36.4	34.7	35.8	36.1
Level 2	37.3	35.9	36.7	36.9	36.3	36.1
Level 3	36.0	35.7	35.4	37.0	36.5	36.3
Delta	2.1	1.3	1.3	2.2	0.7	0.2
Rank	2	4	3	1	5	6

S/N ratio is optimal for each factor. In addition, as shown in Table 3, the factors are ranked based on Delta values representing the difference between the highest and lowest mean S/N ratio of the levels for each factor. A factor with a higher Delta value has a more significant effect on the response. Therefore, flotation time and airflow rate are the most and the least effective factor on the response with regard to having the highest and the lowest Delta value, respectively.

3.1.1. Effect of coagulant type

In oil-water emulsions, the oil droplets are stabilized by forming micelles with the emulsifier. Oily

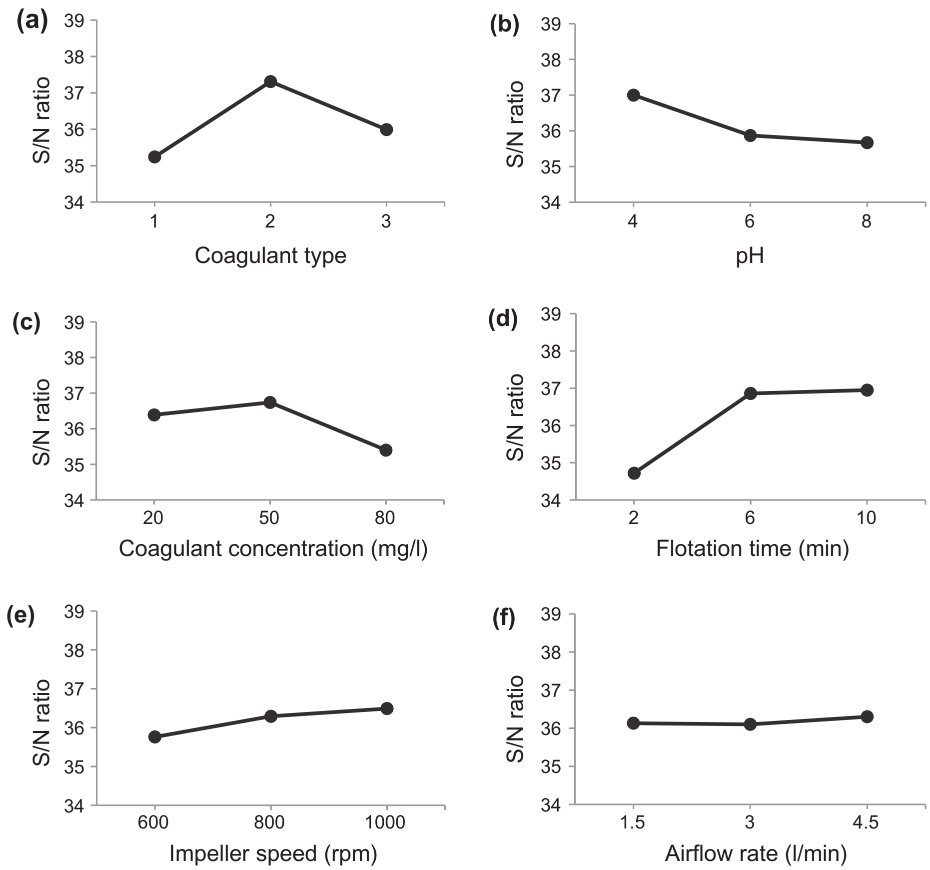

Fig. 1. Effect of operational factors on TPH removal.

emulsion containing micelles cannot be effectively treated by flotation process, unless the emulsion is unstable [25]. Therefore, the emulsions was destabilized by using three coagulants, i.e. $FeCl_3$, $Al_2(SO_4)_3$ and PACl. As shown in Fig. 1(a), the higher value of S/N ratio as the transformed response of system is achieved when second level of coagulant ($Al_2(SO_4)_3$) is used. It was observed that $Al_2(SO_4)_3$ could form less dense and smaller flocs than ones formed by $FeCl_3$ and PACl, probably because of differences between sulfate and chloride-based coagulants in the bonding types of the hydrolysis products. On the other hand, highly turbulent conditions in a mechanical flotation cell can lead to disintegrate the flocs just formed, possibly causing restabilization of the emulsion. However, the flocs formed by $Al_2(SO_4)_3$ may not break up into tiny pieces as much as the ones formed by PACl and $FeCl_3$, since the velocity gradient required to break apart a small floc is much higher than that required to break apart a larger floc. Therefore, the best performance for removal of TPH seems to be obtained by utilizing $Al_2(SO_4)_3$.

3.1.2. *Effect of pH*

Three pH levels of 4, 6, and 8 were applied to investigate the effect of pH on TPH removal of emulsions. The analysis of S/N ratio showed that maximum S/N ratio is obtained in first level (pH of 4) as illustrated in Fig. 1(b). With increasing pH to 6 and 8, it was observed decreasing trend in the S/N ratio. When metal coagulants are used, pH played very important role because of its affecting on polymeric species upon dissolution of the coagulants in water [26]. The measurement of zeta potential has represented that oil droplets in water are negatively charged over the studied pH range (4–8) because of hydroxyl ions adsorption on their surface [3,27]. On the other hand, at a higher pH, the coagulant particles are less positively charged; therefore, their attraction to the anionic oil droplets decreases [28]. Accordingly, it seems that with decreasing pH value, the coagulants particles can cause to form larger oil droplets as a result of charge neutralization and the electrical double layer compression. Also, at lower pH conditions, a higher foam volume is observed on the water surface. It may be due to increasing release of adsorbed surfactant corresponding to the relatively lower ratio of the surface area to the volume of oil droplets. Consequently, oil droplets separation can be improved with increasing the collision possibility between oil droplets and air bubbles because of forming larger oil droplets as well as enhancing the attachment probability of air bubbles to oil droplets by surfactant molecules creating a stable liquid film around each bubble resulting in prevention of bubbles coalescence [29].

3.1.3. Effect of coagulant concentration

The effect of coagulant concentration on TPH removal of the emulsions was investigated in three levels of 20, 50, and 80 mg/l regarding their effective ranges obtained from pretests. As presented in Fig. 1(c), with increasing coagulant concentration from level 1 to level 2, S/N ratio increased. A further increase in coagulant concentration to level 3 was associated with a decrease in S/N ratio. As a result, it can be stated that the more removal efficiency was obtained in coagulant concentration of about 50 mg/l. It has been reported that while zeta potential of oil droplets tends to zero, flotation process is enhanced [3]. At less than critical coagulation concentration (CCC), demonstrated a discontinuity in physical characteristics as a function of coagulant concentration, there are not enough oppositely charged ions present to adequately neutralize the negative charges of the system. However, the restabilization of the system occurs at over the CCC due to reversal of the charge caused by the presence of excess counter ions [29]. It seems that such phenomenon leads to reducing the treatment efficiency at third level of coagulant concentration (80 mg/l).

3.1.4. Effect of flotation time

As shown in Fig. 1(d), the effect of flotation time on TPH removal of the emulsions was examined. With increasing flotation time from level 1 to level 2, S/N ratio mainly increased representing significant improvement of TPH removal. With further increasing flotation time to level 3, the S/N ratio was a bit improved. Therefore, it can be stated that better treatment performance was achieved at flotation time of 6 min; however, no significant improvement of efficiency was observed with further increasing of time. It appears that with increasing of flotation time, probability of oil droplets coalescence, and attachment of gas bubbles to oil droplets were enhanced, thereby improving the rising of oil droplets to the surface of water.

3.1.5. Effect of impeller speed

Three impeller speeds of 600, 800, and 1,000 rpm were employed to investigate its effect on TPH removal of emulsions. As presented in Fig. 1(e), with increasing the speed of impeller to higher levels, the values of transformed response increased. Although some researchers have stated that the formed flocs could be disintegrated under turbulence and high shear forces, other findings suggest that turbulence and agitation can also cause to enhance the contact between oil droplets and air bubbles. Accordingly, the observed increase in the removal of petroleum hydrocarbons with increasing the impeller speed can be justified. This is consistent with results reported by Wells et al. [29].

3.1.6. Effect of airflow rate

The effect of airflow rate on the removal efficiency of TPH by mechanical flotation is shown in Fig. 1(f). It was reported that the high airflow rate resulted in more available bubbles could increase the collision probability between air bubbles and oil droplets, enhancing the removal efficiency [2,30]. In this study, airflow rate was investigated at three levels of 1.5, 3, and 4.5 l/min. With increasing the airflow rate from first (1.5 l/min) to second level (3 l/min), almost no change was observed in transformed response of system. With further increasing to third level (4.5 l/min), very slight improvements were observed in S/N ratio. Therefore, the study of effect of airflow rate on the removal of petroleum hydrocarbons indicated that there are almost no significant differences between the levels of airflow rate considered in this study.

3.1.7. Effect of interactions

The interaction plots for coagulant type and pH as well as coagulant type and concentration are shown in Fig. 2.

If a strong interaction exists between two factors, the effect of a factor on the response will change with varying the levels of the other factor. In the interaction plots, the non-parallel and the intersecting lines represent the existence of a possible and a strong interaction between two factors, respectively [31]. Therefore, it can be stated that there is a strong interaction between coagulant type and pH (Fig. 2(a)) and a moderate interaction between coagulant type and concentration (Fig. 2(b)).

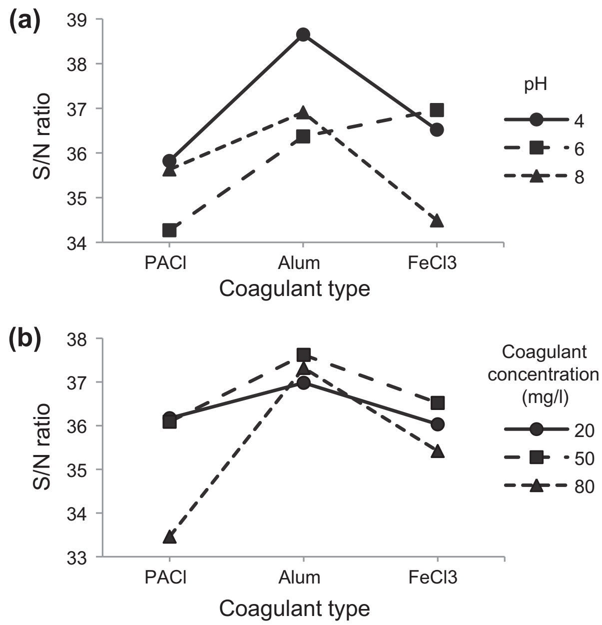

Fig. 2. Interaction effects between (a) coagulant type and pH and (b) coagulant type and concentration.

Table 4
The optimum operating conditions obtained from the analysis of S/N ratio

Factor	Level	S/N ratio
Coagulant type	2	37.3
pH	1	37.0
Coagulant concentration	2	36.7
Flotation time	3	37.0
Impeller speed	3	36.5
Airflow rate	3	36.3

3.2. Optimum conditions

Based on the results obtained from the analysis of S/N ratio, the optimum operating conditions were determined. As mentioned, the level of each factor maximizing the S/N ratio specifies the optimum condition for that factor. Therefore, the optimum conditions within the selected factor levels were defined at the second level of coagulant (aluminum sulfate), the first level of pH (pH = 4), the second level of coagulant concentration (50 mg/l), the third level of flotation time (10 min), the third level of impeller speed (1,000 rpm,) and the third level of airflow rate (4.5 l/min) as shown in Table 4.

3.3. ANOVA results

The experimental results were statistically investigated by ANOVA to evaluate the effects and relative importance of controlling factors on the response [23]. Table 5 demonstrates the results of ANOVA for S/N ratios. As shown, the degrees of freedom (df), sum of squares (SS), mean square (MS), F value, and p value for each factor were given. P value represents the significance probability value of each factor [32]. A p value of 0.05 or smaller for each factor indicates that the considered factor is statistically significant at the 95 percent confidence level chosen for this study. The results showed that coagulant type and concentration, pH, flotation time, and the interaction between coagulant type and pH, which had p value less than 0.05, were identified as having significant effect on the response at a confidence level of 95% within the studied levels of factors. It should be noted that the lack of significant effect of other factors at the confidence level of 95% does not offer that they are not important. It indicates that changing their level to another

Table 5
Results of ANOVA for S/N ratio

Factor	df	SS	MS	F	P
Coagulant type	2	19.8	9.9	14.55	0.005
pH	2	9.2	4.6	6.78	0.029
Coagulant concentration	2	8.7	4.4	6.43	0.032
Flotation time	2	28.6	14.3	21.08	0.002
Impeller speed	2	2.5	1.3	1.85	0.237
Airflow rate	2	0.2	0.1	0.15	0.865
Interaction of coagulant type & pH	4	14.0	3.5	5.15	0.038
Interaction of coagulant type & concentration	4	8.0	2.0	2.93	0.116
Error	6	4.1	0.7		
Total	26	95.1			

Note: df = Degrees of freedom, SS = Sum of squares, MS = Mean square, $F = F$ value, and $P = P$ value.

Table 6
Confirmation experiments for optimum conditions

Run	Confirmation		Prediction	
	S/N ratio	TPH removal (%)	S/N ratio	Mean response (%)
1	39.3	92.7	40.2	95.2
2	39.5	93.9		

one would not cause a significant variation of the response while in turn these factors could be important in the process [33]. Flotation time (with p value of 0.002) was the most important factor on the response corresponding to having the greatest contribution to the total SS. In addition, the other factors, in order of importance, were coagulant type, the interaction between pH and coagulant type, pH, coagulant concentration, the interaction between coagulant type and concentration, impeller speed, and airflow rate.

3.4. Confirmation experiment

Finally, a confirmation experiment was performed to verify the optimum conditions using the combination of the optimal levels as determined by Taguchi analysis [34]. The results of the confirmation experiment with two replications and the predicted performance by Minitab software are presented in Table 6. It was observed that there is a good compatibility between the predicted and measured results.

4. Conclusion

The effects of six factors on the removal efficiency of TPH from the oily emulsion by mechanical flotation following coagulation were investigated by applying Taguchi design of experiment method. Based on the analysis of S/N ratio, optimum conditions for TPH removal of oil-in-water emulsions were obtained by using aluminum sulfate at pH of 4 and concentration of 50 mg/l, flotation time of 10 min, impeller speed of 1,000 rpm, and airflow rate of 4.5 l/min. Under these conditions, about 93% of TPH was removed.

ANOVA showed that, in the range of the examined levels of factors, flotation time and coagulant type have significant effects on the response at the confidence level of 95%. Flotation time (with p value of 0.006) and airflow rate (with p value of 0.948) are the most and least effective factors, respectively. In addition, the other factors, in order of importance, were coagulant type, pH, coagulant concentration, and impeller speed.

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