PRE-TREATMENT OF INDUSTRIAL MINERAL OIL WASTEWATER USING RESPONSE SURFACE METHODOLOGY

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ABSTRACT

This study focuses on the process optimization of coagulation floatation by cross-interaction effects of the following multiple factors: pH, coagulant dosage and floatation time on the response of chemical oxidation demand (COD), soap oil and grease (SOG), turbidity and total suspended solids (TSS). The results from the response surface methodology incorporated with Box Benken design (BBD) models reveals significant correlations and interactions between the manipulated and response variables. To establish the optimum pre-treatment conditions, the experiment used the design BBD, using Design Expert (Design Expert 10.0.3) software. This ensures that the process engineers and scientists gain a better understanding of the practical application of the experimental results over the conventional method of one-factor-at-time (OFAT). The recent trend in industrialization and population growth has indeed deepened the demand on the world's energy (oil) and water resources. However, the need to meet the significance of energy and water for sustainable social economy growth and development has resulted in extremely adverse effects on water and environmental pollution. South Africa, well known as a water scarcity country, has also intensified its policy and increased fines for offenders who do not meet its regulations and discharge limits. In this context, the treatment of industrial mineral oil wastewater, which is regarded as hazardous and harmful to the environment, derived from petrochemical and oil refinery industries, has a recovery value. This has raised attention for systematic technology and approach in recovering oil and water for reuse to conserve the supply of fresh water and energy resources.

Keywords: floatation, response surface methodology, soap oil and grease, oil water separation.

1 INTRODUCTION

Modernisation has shown that the rate of a country's economy and development can be measured by the utility of energy and water per household. For instance, crude oil provides 60% of South Africa's energy followed by coal, synthetic fuels and natural gases, where specific water intake (SWI) of local refineries vary between 0.51 and 0.67 m^3/t of crude. In addition, most of these industries discharge approximately 46% of the water intake as mineral oil wastewater (MOW) with the major contaminant such as chemical oxidation demand (COD), soap oil and grease (SOG), turbidity and total suspended solids (TSS) [1]. However, the oil droplets in the MOW contains emulsified, dispersed, dissolved and spilled oils generated from the petroleum industry during the primitive refining of the crude oil, ships slops, petroleum off spec and during transportation [2], [3].

These excess amounts of MOW causes severe pollution, oxygen depletion, imbalance ecosystem and human health risks when discharged into the environment without treatment. In addition, the unrecovered oil contributes to the presence of a high content of degradable organic compounds with great capacity of penetration into the ground that pose threats to the ground water [4], [5]. Tir and Moulai-Mostefa [6] reported that depending on the pollutants and the source of the MOW; there are several wastewater treatment methods to ensure good effluent quality before discharge into the sewer system. However, there are some limitations associated with some of these methods such as operational cost and inefficient operation leading to downstream problems such as an increase in chemical oxygen demand (COD) and SOG [7], [8]. An improper operation of a municipal wastewater treatment plant (WWTP) led

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to the pollution of various water bodies thereby posing health and social economic threat to the needy [9]. In South Africa, to address and balance the environmental protection, economic viability of recovered oil (lubricant oil) and water (irrigation) has led to strict regulation for the discharge of the effluent [1]. Therefore, the need for one or combination of many methods to achieve effective purification, separation efficiency and lower the effluent quality and cost is attracting the attention of many scholars [10], [11].

Coagulation, which is a chemical treatment method, followed by a separation process such as dissolved air floatation (DAF) are mostly used in the MOW treatment [6]. The chemical treatment is usually done to improve the water quality via the addition of coagulants with small amount of acid (H+). The breaking of emulsions chemically to remove oils can be done via addition of salts (such as Al^{3+}, Fe^{3+} or Ca^{2+}), polymers, bentonites and pH adjustment [8], [12]. In addition, pH adjustment by either increasing (lime, NaOH) or decreasing (HCl, H_2SO_4) pH is very sensitive, where overdose or underdose can lead to inefficient treatment and corrosion of the piping system and equipment [13]. This destabilises the oil emulsion, breaks and slows down the interfacial force between the oil droplet and the continuous dispersed phase. The significance of chemical pre-treatment before floatation is to neutralise the oil charge, absorb the oil-dispersed phase, and hence increase the interfacial bridging to precipitate. This results in stability and increases the coagulated oil droplet size to float for separation [14].

The application of DAF works under the principle of rising velocity, which is a cost-effective alternative to sedimentation. In this process air is induced at the bottom of the floatation column, where the abundant free micron bubbles spontaneously boils out and adheres to the oil droplet, coagulate then cause rise up to float to the surface to be separated. According to Li et al. [2], to achieve 95–97% separation of oil water containing emulsified oil within a floatation time of 5 minutes, the contact time between the oil droplet and the air bubbles is significant. This is due to the complex mechanism between the bubbles–oil droplet attachment process; where the fluid viscosity, oil droplet and bubble size and the interfacial force are affected by the induction and hold up time [15].

To adapt this concept from a bench scale and implement on a full scale, to achieve high treatment efficiency with low chemical usage and desirable water qualities, there exist some limitations to control the process. This is due to variations of the water quality (inlet and outlet). In addition, lack of a predictive model to incorporate the operating conditions (coagulant dosage, pH and floatation time) to compensate the series of treatment processes as well as for decision making [3].

The response surface methodology (RSM) predictive model provides a closer result of the response towards the desirable water quality and that makes it the alternate option for process optimisation. The RSM is a statistical tool that integrates both the independent variables and the experimental data input and then finally generate a predictive model as the output or response. Therefore, its application is widely acceptable due to rapidity and fewer number of experimental runs required, well-designed regression analysis, evaluation and identification of the most significant input factors that can affect the process and help the researcher to focus on identifying and controlling [16]. In addition, the use of one-factor-at-time (OFAT) method is difficult to determine the interactions between the factors and establish the relationship between the input and output variables which is very crucial in the application of multiple factors.

The optimisation of multivariate procedure requires two variables viz. the responses and the factors. The responses are the dependent variables, their values depend on the levels of the factors. The arrangement of the Box Behnken Design (BBD) is based on the selected points from the three-level factorial [17]. This allows the coefficient of first-second order estimation to be the significant eqn (1).

$$Y = \beta_0 + \sum_{i=1}^{n} \beta_i X_i + \sum_{i=1}^{n} \beta_{ii} X_{ii}^2 + \sum_{i<j}^{n} \beta_{ij} X_i X_j + \varepsilon, \quad (1)$$

where $X_i X_j$ represents the interaction terms, and β_{ii} and β_{ij} represents the coefficients of the interaction factors respectively.

Therefore, implementing the RSM-BBD to evaluate the relationship, and interactions of the factors involves the following steps:

- Design of the experiment: three-level designs such as the Box Behnken Design (BBD).
- Regression and statistical analysis, developing of models and graphical representation of the response surface (3D, contour plots).
- Optimisation of the variables using the response model to achieve the desirable target.
- The use of the analysis of variance to check the validity of the models by comparing the predicted model and the experimental values.

In this study the goal is to optimise multiple factors on coagulation floatation treatment process, improve a polymeric coagulant dosage and stabilise the outlet water quality. Pre-treatment optimisation to serve this purpose was carried out using DAF jar test with polymeric chloride (Z553D) coupled with the response surface methodology. The BBD was used to evaluate the effects and interactions of pH, coagulation dosage and floatation time on a local South African oil refinery WWTP effluent quality in the KwaZulu-Natal province.

2 MATERIALS AND METHODS

2.1 Pre-chemical treatment mechanism (Jar test)

The DAF jar test used in this study consists of six identical Perspex conical bottom floatation jars, with each volume capacity of 1200 ml, diameter of 100 mm and a sample point on each side of the jars. The pH of the sample obtained from the oil refinery was first adjusted with either 1.0 M H2SO4 or 1.0 M NaOH stock solution to suit the runs requirement. Then the required dosage of the Z553D was also added to the sample with rapid mixing of 250 rpm for 2 minutes. To enhance the formation of the oil droplet flocs, the stirring speed was then reduced to 30 rpm for 15 minutes. Immediately after the slow mixing time overlap, compressed air was introduced at the bottom via an air release nozzle from an 8 L air saturator at the desired pressure of 350 kPa. The mixture was left to float per the floatation time required.

2.2 Optimisation using Box Behnken Design (BBD)

The first step was to identify the optimum range for the chemical additives (pH and coagulant dosage) at a fixed floatation time of 15 min and coagulant dosage of 50 mg/L. The pH values of 2, 4, 6, 8 and 10 were tested. From the optimum pH obtained and the fixed floatation time as stated earlier, the coagulant dosage was also evaluated at 10, 20, 30, 40, 50 and 60 mg/L.

Lastly, to overcome the limitations of OFAT and determine the overall optimum conditions, the BBD in the RSM (Design Expert 10.0.3) was used to generate the experimental matrix and the outcome was used to find the optimal variable and the response model equation to enhance the performance of the Z553D for the treatment of the MOW.

The responses identified for this study were COD (Y1), SOG (Y2), TSS (Y3) and turbidity (Y4). The COD, turbidity and TSS were measured with the Hach DRB 3900 spectrophotometer, Hach 2100N and Hach DR/890 portable colorimeter respectively. The South African Bureau of Standards (SABS) technique 1051 was adapted for the analysis of SOG using Dichloromethane for the oil extraction. Three distinct factors were also identified as the manipulating variables affecting the responses. This included pH (A), coagulant dosage (B) and floatation time (C) with their levels shown in Table 1.

The percentage removal of the response was calculated using eqn (2):

$$\mathrm{Y_n}(\%) = \frac{\mathrm{y_0} - \mathrm{y_n}}{\mathrm{y_0}} \times 100, \tag{2}$$

where $\mathrm{Y_n}$, $\mathrm{y_0}$ and $\mathrm{y_n}$ represent the demanded response (water quality), initial and final water quality respectively.

3 RESULTS AND DISCUSSION

The chemical pre-treatment before floatation allows the oil droplet, colloids, organics and suspended solids to agglomerate into larger aggregates for stability and buoyancy in order to float. In addition, it was observed that the air bubble size depended on the air saturator pressure applied. This increased the air bubble–oil droplet attachment via the mechanism of collision, adhesion and stabilisation [14].

Evaluation of pH, coagulant dosage and floatation time to identify the most important factor and their state of interest was seen as important, hence reducing downstream treatment cost and adding value to recovered oil and water. The response COD, SOG, TSS and turbidity were found to be dependent on the input factors. Where the change in response was found to be directly proportional to the effect caused by the change in level of the factor and where the change was caused by two factor levels it was deduced that there is interactional effects. The results obtained from the BBD matrix is represented in Table 2.

3.1 Effects of pH

The adjustment of the pH has significant effect on the separation due to the negative charge of the oil droplet. This affects the nature and the occurrence of the flocs, thus neutralising the surface charge. It was found that lower pH resulted in the oil droplet flocs becoming unstable and enlarged resulting in them being easily separated. Fig. 1 represents the effect of pH on the removal efficiency of the response contaminants. It can be seen that the charge neutralisation and precipitation of the oil droplet and other particles were feasible for a narrow range of pH 4–6 for effective separation. Thus above 85% of COD, SOG and turbidity were removed at a floatation time of 15 min and coagulant dosage of 50 mg/L. In the case of TSS above 70% was removed. However, pH has a lesser significant influence on TSS and turbidity removal unlike, COD and SOG. Increasing the pH decreases the efficiency removal hence the lower the pH the better the removal. Therefore, the pH was adjusted to within 4 and 6 for the process optimisation.

Table 1: RSM – Box Behnken Design matrix.

Input factors	*Range or levels*		
Coded values	–1	0	1
A: pH	4	5	6
B: Coagulant dosage (mg/L)	30	40	50
C: Floatation time (min)	10	15	20

Table 2: Box Behnken Design matrix.

Standard run	pH	Coagulant dosage (mg/L)	Floatation time (min)	COD (mg/L)	SOG (mg/L)	Turbidity (NTU)	TSS (mg/L)
1	0	0	0	87	89	80	71
2	0	0	0	84	90	83	70
3	0	–1	1	80	70	73	63
4	–1	1	0	88	91	87	76
5	1	0	–1	79	75	78	66
6	0	0	0	84	86	81	69
7	0	1	1	89	88	86	74
8	1	1	0	88	88	83	73
9	–1	0	1	77	83	83	68
10	0	0	0	82	90	85	68
11	1	0	1	77	84	84	67
12	–1	–1	0	75	73	71	61
13	–1	0	–1	79	84	80	66
14	1	–1	0	78	72	72	65
15	0	1	–1	89	90	84	73
16	0	–1	–1	79	81	74	62
17	0	0	0	83	88	82	61

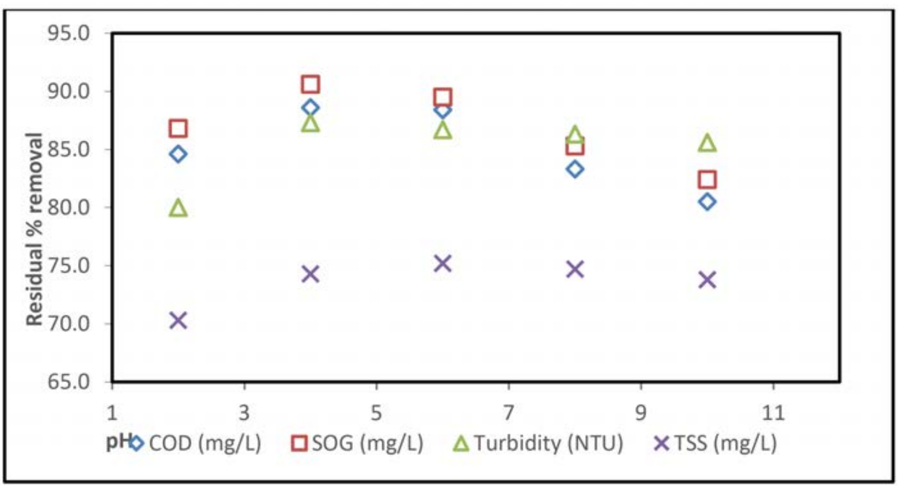


Figure 1: Effects of pH on response % removal; coagulant dosage 50 mg/L and floatation time 15 min.

3.2 Effects of coagulant dosage

To enhance oil water separation there must be a strong interfacial force between the oil-water, bubbles-water and bubbles–oil. However, the floatation mechanism depends solely on the collision that occurs between the air bubbles and the oil droplet. Hence, the addition of the Z553D intensifies the bridging and adsorption action, resulting in increased collisions and agglomeration as well as forming larger flocs.

In contrast, when the dosage exceeded the centre of limits stability resulted and the flocs easily broke thereby reducing the tendency to float rather settle as sludge [2]. Fig. 2 shows that the removal of the contaminants were more efficient within the coagulant dosage of 40–50 mg/L, thus above 75% for TSS and that of COD, SOG and turbidity over 85% were being removed. On the other hand, at a coagulant dosage of 60%, there is likelihood of an overdose or excess Z553D, which might have caused the increase of air-floc to agglomerate with slow rising velocity thereby decreasing the Z553D efficiency.

3.3 Optimization using RSM

The optimum conditions and the region of interest was determined using the RSM coupled with the BBD. On this basis, the data obtained from the experiment was used to study the effects and interaction towards the response. To justify the correlation that exists between the input and response variables, the analysis of variance (ANOVA) was used to test the statistical significance and the response model generated. The following are the empirical response regressions with their significant coded model terms:

$$Y_1 = -19.75 + 40.35A - 1.275B + 2.325C - 4A^2 + 0.0225B^2 - 0.08C^2, \quad (3)$$

$$Y_2 = 87.37 - 1.5A + 7.63B - 4.33A^2 - 3.58B^2, \quad (4)$$

$$Y_3 = 80.35 + 6.25B, \quad (5)$$

$$Y_4 = 67.82 + 5.63B. \quad (6)$$

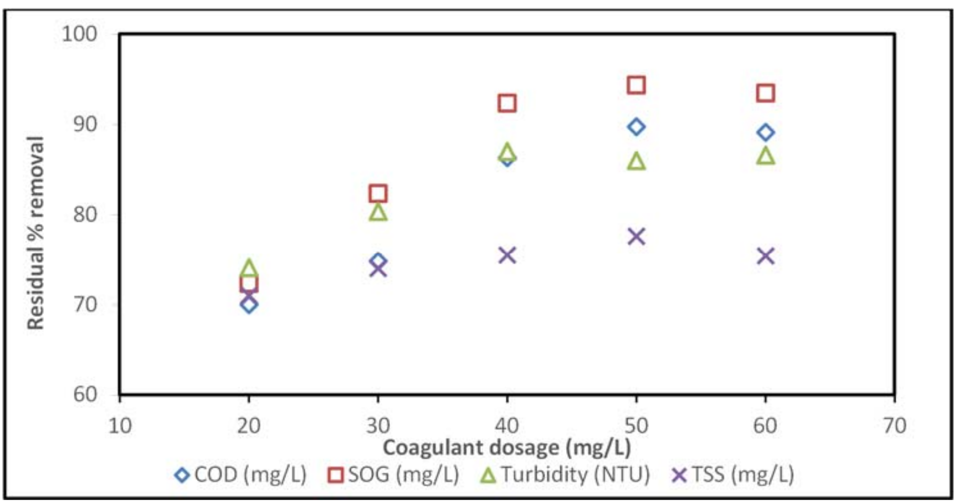


Figure 2: Effects of coagulant dosage on response % removal; pH 5 and floatation time 15 min.

To predict the response using the given level of each factor and to identify the relative response by comparing their coefficients, all the input terms must be in a coded form. The positive coefficient terms in eqns (3)–(6) signifies an increase on those terms will increase the percentage removal of the response. Whilst the negative terms suggest if not maintained at an optimum value, will lower the efficiency of the removal.

The quadratic and linear regressions selected were all found to be significant with low standard deviation and a high coefficient of determination (R2) values as shown in Table 3. The actual R2 for each of the response models were 0.93, 0.90, 0.77 and 0.74 for Y1, Y2, Y3 and Y4 respectively. The ANOVA for all the responses in Table 4 also depicts that all the input variable terms in the response empirical models were all significant with *P*-values less than 0.05. Also the lack of fit errors has no significant effects on the models.

Table 3: Summary of all response model statistics.

Responses	Model Source	Std. Dev.	Adeq. Precision	Actual R^2	Adjusted R^2	*P*-value	*P>F*-value
COD	Quadratic	1.51	13.69	0.93	0.90	19.23	0.0009
SOG	Quadratic	3.65	10.64	0.90	0.80	6.54	0.0194
TSS	Linear	2.5	14.61	0.77	0.76	3.43	0.1237
Turbidity	Linear	2.41	13.59	0.74	0.73	2.6	0.1854

Table 4: ANOVA for fits of all responses percentage removal.

Source / input variable	Sum of squares	df	Mean square	*F*-value	*p*-value Prob > *F*	
Y_1-model	326.31	6	54.38	23.91	< 0.0001	significant
A	68.28	1	68.28	30.01	0.0003	
B	10.61	1	10.61	4.66	0.0562	
C	15.58	1	15.58	6.85	0.0257	
A^2	67.37	1	67.37	29.61	0.0003	
B^2	21.32	1	21.32	9.37	0.012	
C^2	16.84	1	16.84	7.4	0.0215	
Residual	22.75	10	2.27			
Lack of Fit	8.75	6	1.46	0.42	0.8381	not significant
Pure error	14	4	3.5			
Cor total	349.06	16				
Y_2-model	624.03	4	156.01	11.71	0.0004	significant
A	18	1	18	1.35	0.2677	
B	465.13	1	465.13	34.92	< 0.0001	
A^2	79.12	1	79.12	5.94	0.0313	
B^2	54.08	1	54.08	4.06	0.0669	
Residual	159.85	12	13.32			
Lack of Fit	148.65	8	18.58	6.64	0.0426	not significant
Pure Error	11.2	4	2.8			
Cor Total	783.88	16				
Y_3-model	312.5	1	312.5	50.2	< 0.0001	significant

Table 4: Continued.

Source / input variable	Sum of squares	df	Mean square	F-value	p-value Prob > F	
B	312.5	1	312.5	50.2	< 0.0001	
Residual	93.38	15	6.23			
Lack of Fit	78.58	11	7.14	1.93	0.2755	not significant
Pure Error	14.8	4	3.7			
Cor total	405.88	16				
Y_4-model	253.12	1	253.12	43.47	< 0.0001	significant
B	253.13	1	253.13	43.47	< 0.0001	
Residual	87.35	15	5.82			
Lack of Fit	24.55	11	2.23	0.14	0.9954	not significant
Pure error	62.8	4	15.7			
Cor total	340.47	16				

3.4 The three-dimensional (3D) plot

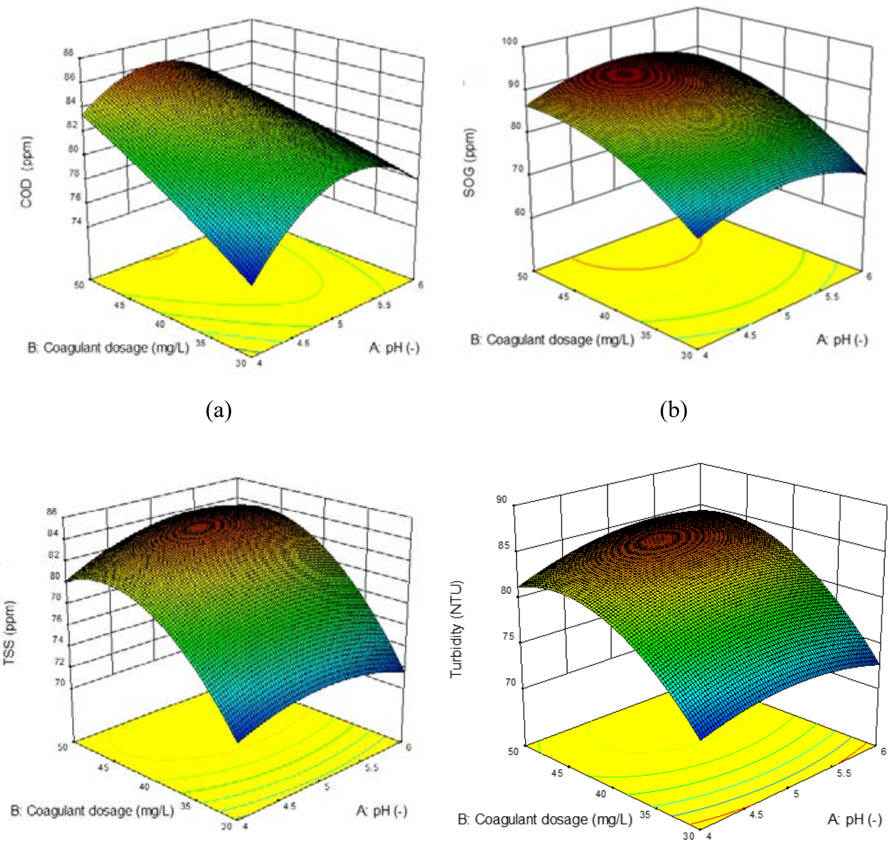
The sensitivity of the input variables towards the responses were represented graphically in 3D response surface plots. On this basis, the floatation time was kept constant (15 min) while the coagulant dosage and pH were varied within their designed range. This helped to identify the main factor, interactional effects and the optimum condition for decision making onto the large scale WWTP. The mutual interaction and high peak region between the coagulant dosage and pH was located at 45–50 mg/L and the pH within the range of 4.5–5.5, where over 80% of the contaminants were removed.

Fig. 3(a) shows the graphical representation of how the COD response varies as a function of the input variables (coagulant dosage and pH). The peak of the curvature suggest that the optimum conditions to maximise the COD removal is well inside the design boundaries. In addition, COD removal increases with an increase in coagulant dosage at fixed pH and floatation time (5 and 15 minutes).It was found 91% of the COD was removed at coagulant dosage of 49 mg/L.

Fig. 3(b) shows the relative effect of the input variables (coagulant dosage and pH) on the removal of SOG. The removal of SOG increases with an increase in coagulant dosage at fixed pH (5). And a maximum removal of 92% was attained by using a coagulant dosage of 49 mg/L.

Fig. 3(c) shows the 3D response plot for TSS removal showing the interaction effect of coagulant dosage and pH. It was noted increasing the coagulant dosage had much impact on the removal than the pH. And at coagulant dosage of 49 mg/L, maximum removal of 86% TSS was attained.

Fig. 3(d) also shows the 3D response plot for the turbidity removal denoting the relative effect of the two input variables. It was noted that increasing the coagulant dosage had no significance effects to maximise the removal whiles the pH had no significant effect on the removal. And 73% as the maximum removal was attained.



(a) (b)

(c) (d)

Figure 3: (a) 3D plot for COD response – effects of pH and coagulant dosage at constant floatation time (15 minute); (b) 3D plot for SOG response – effects of pH and coagulant dosage at constant floatation time (15 minute); (c) 3D plot for TSS response – effects of pH and coagulant dosage at constant floatation time (15 minute); (d) 3D plot for turbidity response – effects of pH and coagulant dosage at constant floatation time (15 minute).

4 CONCLUSION

The operating conditions suitable for the chemical changes and the specific water quality was successfully performed by using a complete mimic WWTP (DAF jar test). The acidification and coagulation before DAF helped improve the effluent treatment, water quality and value added through the recovered oil and water recycled with less downstream cost. The use of the RSM coupled with the BBD provided efficient metrics with a smaller number of experimental data fitted on regression equations with accuracy. The empirical response model predictions were consistent with the experimental data and thus there exists a correlation between the operating factors and response water quality. The 3D response surface helps to identify the effect of the main factor in the system by visualising their interactions towards the response. To assist the process engineer in decision making on a large scale, the main factor to control was the coagulant dosage while the optimum pH and floatation time were kept constant at 5 and 15 minutes respectively. In addition, the optimum coagulant dosage using the RSM was 49 mg/L while that of the OFAT was 50 mg/L with over 80% of the contaminants removed. Therefore, application of RSM for optimisation of industrial WWTP was seen to be significant over OFAT due to the option to evaluate and visualise the interactional effects between the input variables towards the responses with fewer resources required.

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