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Coagulation–flocculation process for POME treatment using *Moringa oleifera* seeds extract: Optimization studies

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Abstract

The treatment of palm oil mill effluent, POME is an important issue for the minimization of water pollution. The coagulation–flocculation process using *Moringa oleifera* seeds after oil extraction as natural coagulant is reported for POME treatment in the present study. The important process parameters pH, settling time, *M. oleifera* (after oil extraction) dosage and flocculant (NALCO 7751) dosage were optimized using design of experiments (DOE). A full factorial composite experimental design and response surface methodology (RSM) were used to obtain the optimum values of the parameters. The suspended solids of the raw POME were reduced from 17.927 mg/L to 181 mg/L and % recovery of the sludge as 87% was obtained at an optimum pH 5, settling time 114 min, *M. oleifera* dosage of 3469 mg/L and 6736 mg/L of flocculant (NALCO 7751) dosage in the present study.

Keywords: *Moringa oleifera* seeds; POME; Wastewater treatment; Response surface methodology; Coagulation–flocculation process

1. Introduction

The rapid development of the palm oil industries in Malaysia over the years should be in tandem with the development of its environmental technical know-how. There are still many environmental pollution issues that need to be addressed on a dynamic basis even though the existence of environmental laws and regulations since the implementation of the Environmental Quality Act (EQA) [1]. A new and improved POME treatment technology would be required in order to meet the requirements of Department of Environment discharge limits.

Palm oil mill effluent (POME) is a voluminous, high biochemical oxygen demand (BOD) liquid waste, generally discharged at 75–85 °C. It is a colloidal dispersion of biological origin and with an unpleasant odour. It has total solids content of 5–7% which a little over half is dissolved solids, and the other half being a mixture of various forms of organic and inorganic suspended solids. This property, coupled with its high BOD loading and low pH, makes it not only highly polluting but also extremely difficult to treat by conventional methods [2]. Based on the statistics of total crude palm oil production in May 2001, the crude palm oil production of 985,063 tonnes used 1,477,595 m^3 of water, and 738,797 m^3 was discharged as POME. Without proper treatment of POME, the effluent will pollute watercourses where this effluent is discharged [3].

Since the ultimate goal of wastewater management is towards zero discharge, the best wastewater treatment scheme is inevitably a treatment that allows 100% reuse and recycling of the water. However, if this is the solution, a higher quality of treated water is required especially when it is to be used as boiler feed water in the palm oil mill. The industry should adopt new green and clean technologies to reduce or prevent waste generation at source [4].

The current treatment technology of POME typically consists of biological aerobic and anaerobic digestion or facultative digestion. Biological treatment systems need proper maintenance and monitoring as the processes solely rely on microorganisms to break down the pollutants. The microorganisms are very sensitive to the changes in the environment and thus great care has to be taken to ensure that a conducive environment is maintained for the microorganisms to thrive in. It requires skilful attention, commitment and long retention time. Besides, it also generates a vast amount of biogas. This biogas contains methane, carbon dioxide and trace amounts of hydrogen sulphide. Some of these gases are corrosive and odorous.

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Nomenclature

A	first factor or input variable, pH
b_{ij}	the first-order interaction effect
b_j	the linear effect
b_{jj}	the squared effect
b_0	the offset term
B	second factor or input variable-settling time (min)
C	third factor or input variable—*Moringa oleifera* (after oil extraction) dosage (mg/L)
D	fourth factor or input variable—flocculant (NALCO 7751) dosage (mg/L)
R^2	determination coefficient
x_i	the coded value of the *i*th independent variable
X_i	the natural value of the *i*th independent variable
X_i^x	the natural value of the *i*th independent variable at the center point
ΔX_i	the step change value
Y	the predicted response

The treated water produced also cannot be recycled back to the plant [5].

The treatment of POME using different methods has recently increased the interest of many researchers [6,7]. Oswal et al. [6] treated POME using tropical marine yeast, *Yarrowia lipolytica* NCIM 3589 and 95% of chemical oxygen demand (COD) reduction was achieved with a retention time of 2 days. Nik Nurulaini et al. [7] reported that the chemical coagulation is the fastest way to reduce the organic load of the POME to an acceptable and economical level. Up to 60% removal of the BOD and COD and 90% of the suspended solids (SS) are within the reach with proper selection of chemical coagulant and its optimum dosage.

POME treatment requires a sound and efficient system in facing the current challenges. There are many processing plants failed to comply with the standard discharge limits even though they have applied biological treatment system. Therefore, an alternative POME treatment system is required to meet standard discharge limits (400 mg/L of suspended solids). A technological shift from biological and chemical treatment to coagulation–flocculation process with environmental friendly coagulants could result to the improved the POME treatment system.

Coagulation–flocculation followed by sedimentation and filtration is used worldwide in the wastewater treatment process before discharge of the treated water to the river. Many coagulants are widely used in the conventional wastewater treatment processes [8]. These coagulants can be inorganic coagulants (e.g., aluminium sulfate and polyaluminium chloride), synthetic organic polymers (e.g., polyacrylamide derivatives) or naturally occurring flocculants (e.g., microbial flocculants). These coagulants and flocculants are used for different purposes depending on their chemical characteristics [9].

Aluminium sulphate (Alum) an inorganic salt is the most widely used coagulant in water and wastewater treatment [10]. High level of aluminium concentrations in water may have human health implications However, recent studies have pointed out several serious drawbacks of using aluminium salts, such as '*Alzheimer*'*s*' disease and similar health related problems associated with residual aluminium in treated waters [11]. Aluminium has also been indicated to be a causative agent in neurological disease like '*per-senile dementia*' [10]. Many developing countries can hardly afford the high costs of imported chemicals for water and wastewater treatment [11]. In this context, environmental friendly coagulant presents a viable alternative for the treatment of wastewater.

Moringa is a tropical plant belonging to the family *Moringacae* (syns. *Moringa pterygosperma Gaertn.*), that is a single family of shrubs with 14 known species. *Moringa oleifera* is native to India but is now found throughout the tropics. Commonly known as the '*horse-radish*' tree or '*drumstick*' tree [12–14]. *Moringa* is full of nutrients and vitamins and is good as a human food as well as in the food of animals. This multipurpose tree also helps to clean dirty water and is a useful source of medicines [9,14–15].

Earlier studies recommended the use of *M. oleifera* seed extract as coagulant for water treatment in African and South Asian countries [16]. The seed cake, produced after extraction of active ingredients of seed contains high levels of protein and makes a good fertilizer for use in agriculture [14–15] or as an animal feed. *M. oleifera* coagulant (MOC) as primary coagulant was used for raw waters and synthetic turbid waters for turbidity removal in the range of 80–99% [17–19].

The activity of *M. oleifera* seeds as a coagulant is due to the presence of water soluble cationic proteins in the seeds. These proteins are densely charged cationic dimmers (protein complex made up of two subunits with a positive charge) with a molecular weight of about 13 kDa and isolelectric pH value of 10 and 11, respectively [18]. Adsorption and neutralization of charges are the main mechanisms of coagulation. It is possible to extract an edible vegetable oil from the *Moringa* seeds before using as the coagulant. In coagulation, *Moringa* hardly affects the pH and conductivity [11]. Therefore, the application of *M. oleifera* seeds in wastewater treatment can reduce the cost of chemicals used for pH adjustment. The volume of sludge produced using *Moringa* as coagulant is considerably less compare to alum [11] and do not offer any disposal problem.

Thus, *Moringa* seed after oil extraction is recommended as a suitable resource material for POME treatment because it has excellent properties as an environmental friendly coagulant.

2. Design of experiment (DOE)

The traditional experimental method, one factor at a time approach, can hardly be used to establish relationships among all the experimental input factors and the output responses. Even though the traditional approach can be useful in finding predominant factors in this situation, it is time and energy consuming method. Furthermore, since the results are valid only under fixed experimental conditions so the prediction for other conditions is uncertain [20]. To solve this problem, design of experiment (DOE) offers better alternative to study the effect of variables and their responses with minimum number of experiments.

Using design of experiment based on response surface methodology (RSM), the aggregate mix proportions can be arrived with minimum number of experiments without the need for studying all possible combinations experiment. Further the input levels of the different variables for a particular level of response can also be determined. RSM is a collection of statistical technique for designing experiments, building models, evaluating the effects of factors and searching for the optimum conditions of the factors. RSM also quantifies relationships among one or more measured responses and the vital input factors [21]. Therefore, DOE could be used in POME wastewater treatment with minimum numbers of experimental runs, and to determine the optimum conditions using RSM [22].

In order to determine if there exist a relationship between the factors and the response variables investigated, the data collected must be analyzed in a statistically manner using regression. A regression design is normally employed to model a response as a mathematical function (either known or empirical) of a few continuous factors and 'good' model parameter estimates are desired [22]. In developing the regression equation, the test factors were coded according to Eq. (1):

$$x_i = \frac{X_i - X_i^x}{\Delta X_i} \quad (1)$$

where, x_i is the coded value of the *i*th independent variable, X_i the natural value of the *i*th independent variable, X_i^x the natural value of the *i*th independent variable at the center point, and ΔX_i is the value of step change.

Each response Y can be represented by a mathematical equation that correlates the response surface. The four independent variables can be represented as second-order polynomial equation:

$$Y = b_0 + \sum_{j=1}^{4} b_j x_j + \sum_{i,j=1}^{4} b_{ij} x_i x_j + \sum_{j=1}^{4} b_{jj} x_j^2 \quad (2)$$

where Y is the predicted response, b_0 the offset term, b_j the linear effect, b_{ij} the first-order interaction effect and b_{jj} is the squared effect.

They are few papers reported in the literature on the application of DOE in wastewater treatment, such as application of the central composite design and response surface methodology to the advance treatment of olive oil processing wastewater using Fenton's peroxidation [23], empirical modeling of *Eucalyptus* wood processing [24] and the optimization of ozone treatment for colour and COD removal of acid dye effluent using central composite design experiment [25].

The RSM have several classes of designs, with their own properties and characteristics. Central composite design (CCD), box-Behnken design and three-level factorial design are the most popular designs applied by the researchers. The CCD was used to study the effects of the variables towards their responses and subsequently in the optimization studies [22].

The objective of the present study is to find out the optimum values of the process parameters using RSM in POME treatment. In the present research *M. oleifera* seed extract as a natural coagulant is utilized for the POME treatment. The design of experiments is used to study the effects of pH, settling time, *M. oleifera* dosage (after oil extraction) and flocculant (NALCO 7751) dosage as the process variables on the treatment of POME. RSM technique was used to optimize the responses of suspended solids (mg/L) and % recovery of the sludge formed after the treatment of POME against four input process variables.

3. Experimental

3.1. POME sample collection

POME sample was collected from United Palm Oil Mill, Nibong Tebal, Penang and are cooled to room temperature. The characteristics of POME were obtained following APHA Standard Methods of Examination of Water and Wastewater [26] as shown in Table 1. Although the characteristics of POME could vary but, in order to minimize the effect of different characteristics of POME, the experiments were repeated with different samples of POME to obtain the average results that can be applied to the treatment of different POME samples.

3.2. Coagulant preparation

The dry *M. oleifera* seeds were obtained from Nibong Tebal, Penang, Malaysia. The seed wings and coat were removed manually, good quality seeds were then selected, and the kernel was grounded to a fine powder. Oil content of dry *M. oleifera* seeds was extracted with *n*-hexane as a solvent using Soxhlet apparatus. The extraction was carried out for about 8 h. Stock solution of the *M. oleifera* cake after extraction of oil was prepared by dissolving 5 g of this cake in 100 mL distilled water. The mixture was stirred in a blender (Model National MX-798S) for 2 min to extract the active ingredients. The resulting suspension was filtered through a muslin cloth. The flocculant (NALCO 7751) was obtained from Merck Sdn. Bhd., Malaysia. The chemical description of milky white liquid flocculant (NALCO 7751) is a formulation of water-soluble polymer, ammonium sulfate and inorganic acid(s). This flocculant was chosen in this research because the material safety data sheet (MSDS) of this flocculant shows that it is non-hazardous and biodegradable. Its also did not contain aluminium in the flocculant. Thus, this flocculant is safe and suitable for POME treatment.

Table 1
Typical characteristic of palm oil mill effluent

Parameters	Range	Average value
Temperature (°C)	75–90	80
pH	4.0–4.8	4.5
Suspended solid, SS (mg/L)	11,500–22,000	17,927
Total solid, TS (mg/L)	36,500–42,600	39,470
Chemical oxygen demand, COD (mg/L)	30,000–50,400	40,200
Oil and grease (mg/L)	1300–4700	2658
Total kjeldahl nitrogen, TKN (mg/L)	660–890	800

Table 2
Experimental factors and their levels

Factors	Range and levels				
	−α (−2)	Low level (−1)	Zero level (0)	High level (+1)	−α (+2)
A, pH	3	4	5	6	7
B, Settling time (min)	30	60	90	120	150
C, *Moringa oleifera* dosage (mg/L)	1000	2000	3000	4000	5000
D, Flocculant dosage (mg/L)	5000	6000	7000	8000	9000

3.3. Coagulation–flocculation process

The 600 mL beaker was filled with 500 mL of POME for each test run. The pH value of each jar test was adjusted to the desired value by using either sulphuric acid (3 M) or potassium hydroxide (5 M) within the range of 3–7; *M. oleifera* dosage (after oil extraction) was added varying from 1000 mg/L to 5000 mg/L and POME sample was agitated at 150 rpm for 5 min (rapid mixing). The mixing speed was reduced to 30 rpm for 30 min after adding the flocculant dosage (NALCO 7751) varying from 5000 mg/L to 9000 mg/L. The contents of each beaker were then allowed to sediment with the settling time of 30–150 min.

3.4. Determination of the response

The suspended solids content of the supernatant was determined by a turbidity measurement with the help of Turbidity meter, model WTW Turb 350 IR. While, the percentage recovery of the sludge (after POME treatment) produced was calculated from the mass of sludge after drying 24 h at 105 °C divided by the dry mass of sludge present from raw POME. The suspended solids (mg/L) and % recovery of the sludge were used as the responses for optimization using the design of experiments.

3.5. Model fitting and statistical analysis

The regression and graphical analysis with statistical significance were done using Design-Expert software (version 6.0.6, Stat-Ease, Inc., Minneapolis, USA). In order to visualize the relationship between the experimental variables and responses, the response surface and contour plots were generated from the models. The optimum values of the process variables were obtained from the response surface.

4. Results and discussion

4.1. Experimental design

Response surface methodology was used in order to obtain the relationship between the variables and responses. The range and levels of the variables investigated in the present study are given in Table 2. Generally, these parameters range have been arrived based on the preliminary experiments. It was found from the preliminary studies that the coagulation–flocculation efficiency decreased when operating the process at pH more than 7 and therefore the pH range studied was between 3 and 7.

A 2^4 factorial design with eight star points and six replicates at the central points were employed to fit the second-order polynomial model, which indicated that 30 experiments are required and presented in Table 3. The central values (zero level) chosen for experimental design were: pH 5; settling time, 90 min; *M. oleifera* dosage (after oil extraction), 3000 mg/L; and flocculant dosage (NALCO 7751), 7000 mg/L and experimental runs 3, 4, 5, 14, 17 and 27 were repeated in the random order at these conditions for obtaining the experimental error. By using multiple regression analysis, two responses were correlated with the four

Table 3
Experimental factors and experimental responses

Run	Experimental factors				Responses	
	A	*B*	*C*	*D*	Suspended solids (mg/L)	Recovery of the sludge (wt%)
1	0	−2	0	0	368	82
2	−1	−1	−1	1	276	84
3	0	0	0	0	183	87
4	0	0	0	0	211	87
5	0	0	0	0	200	87
6	1	1	1	−1	228	85
7	−1	1	−1	−1	218	87
8	0	0	0	−2	244	84
9	0	0	−2	0	222	85
10	2	0	0	0	194	82
11	0	0	0	2	306	84
12	1	−1	1	−1	259	84
13	1	1	−1	1	246	84
14	0	0	0	0	194	87
15	1	1	1	1	198	85
16	−1	1	1	1	205	84
17	0	0	0	0	205	86
18	0	2	0	0	229	85
19	−2	0	0	0	278	84
20	1	1	−1	−1	262	82
21	−1	1	1	−1	213	87
22	−1	−1	−1	−1	246	84
23	−1	−1	1	1	425	83
24	0	0	2	0	231	86
25	−1	−1	1	−1	379	83
26	−1	1	−1	1	194	86
27	0	0	0	0	203	87
28	1	−1	1	1	302	86
29	1	−1	−1	−1	234	82
30	1	−1	−1	1	280	84

variables studied using the polynomial Eq. (2). Table 3 shows the coded experiments conducted as per experimental design along with the response values.

4.2. Process models

By using multiple regression analysis, the responses (suspended solids and percentage recovery of the sludge) were correlated with the four variables studied using the second-order polynomial as represented by Eq. (2). The coefficients of the model equation and their statistical significance were evaluated using Design-Expert 6.0.6 software. The quadratic regression model for the suspended solids (mg/L^{-1}) and % recovery of the sludge in terms of coded factors are given by Eqs. (3) and (4), respectively:

$$\text{suspended solids (mg/L)} = +199.33 - 13.13A - 38.13B + 11.29C + 8.79D + 9.39A^2 + 25.01B^2 + 7.01C^2 + 19.14D^2 + 22.19AB - 20.19AC - 0.062AD - 25.31BC - 15.19BD + 0.94CD \quad (3)$$

$$\%\ \text{recovery of the sludge} = +86.83 - 0.42A + 0.67B + 0.25C + 0.083D - 0.90A^2 - 0.77B^2 - 0.27C^2 - 0.65D^2 - 0.63AB + 0.75AC + 0.62AD + 0.000BC - 0.38BD - 0.25CD \quad (4)$$

where A, B, C and D are the coded values of the process variables pH, settling time, *M. oleifera* dosage (after oil extraction) and flocculant (NALCO 7751) dosage, respectively. The coefficients with one factor represent the effect of the particular factor, while the coefficients with two factors and those with second-order terms represent the interaction between the two factors and quadratic effect, respectively. The positive sign in front of the terms indicates synergistic effect, while negative sign indicates antagonistic effect.

The statistical analysis gives several comparative measures for the model selection. Ignoring the aliased model, the quadratic model seems to be the best: based on low standard deviation and high R^2 statistics. The value of R^2 for the suspended solids and percentage recovery of the sludge were 0.9560 and 0.9630, respectively. This is also evident from the fact that the plot of predicted versus experimental suspended solids and percentage of the sludge in Figs. 1 and 2, respectively are close to $y = x$ showing that the prediction of experimental data is quite satisfactory. These plots therefore visualize the performance of the model.

4.3. Effects of process variables

The graphical representations of the models (Eqs. (3) and (4)) facilitate an examination of the effects of the experimental factors on the responses, 3D surface graphs and contour plots between the factors were obtained using the Design-Expert software and are presented in Figs. 3–6. These figures illustrate the responses of different experimental variables and can be used to identify the major interactions between the variables [27].

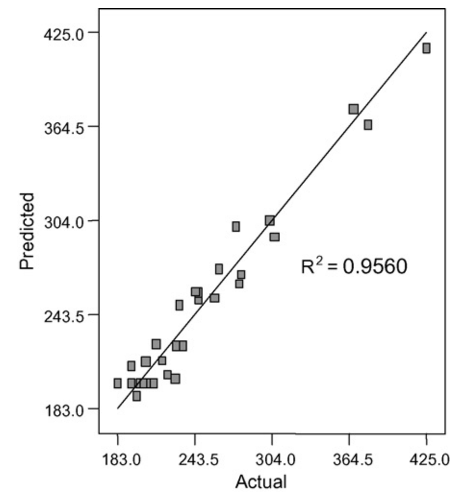

Fig. 1. Predicted vs. actual data for suspended solids (mg/L).

The 3D surface graph and contour plot in Fig. 3 shows that the reduction of suspended solids increased with the increase in settling time 90–105 min and *M. oleifera* dosage 3000–4000 mg/L. The response surface in Fig. 3 shows a curvature. This indicate that the interaction effect between settling time and *M. oleifera* dosage on suspended solids is greatly pronounced, as confirmed by significance test. *M. oleifera* is a short chain low molecular weight and high charge density compound. The positive charged proteins bind to the surface of the negatively charged particles. This led to the formation of negatively and positively charged particle surfaces. Due to particle collision enhanced by agitation, inter-particle interactions between the differently charged sectors took place and resulted in the formation of flocs [28]. The isoelectric pH of these proteins is 10–11 [18]. *M. oleifera* gave nearly 25 wt% of edible oil as a side product, which makes this coagulant more economical in its usage after oil extraction.

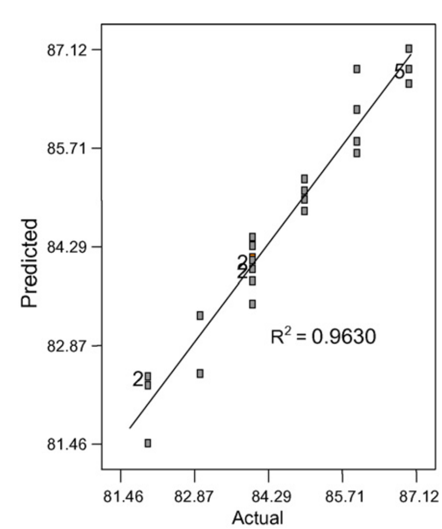

Fig. 2. Predicted vs. actual data for % recovery of the sludge.

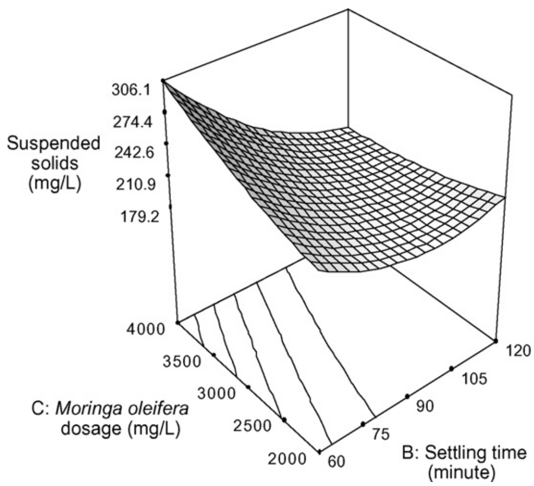

Fig. 3. Effect of *Moringa oleifera* (after oil extraction) dosage and settling time on suspended solids; 3D surface graph and contour plot.

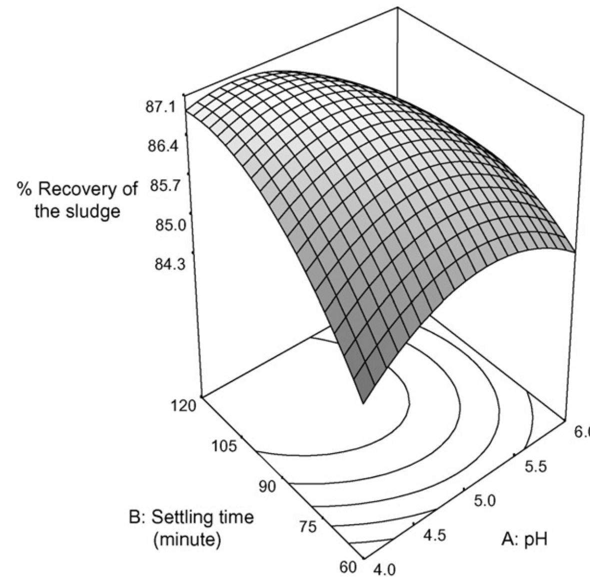

Fig. 5. Effect of pH and settling time on % recovery of the sludge; 3D surface graph and contour plot.

Fig. 4 shows the effect of flocculant (NALCO 7751) dosage in 3D response surface plot. The plot shows that the flocculant (NALCO 7751) dosage of 6500–7500 mg/L and settling time of about 90–105 min reduced the suspended solids. The high molecular weight of flocculant (NALCO 7751) has a high cationic charges or potentially ionisable functional group. The destabilization of particle by large polymeric molecules involves the bridging of particles by the polymer chain, hence forming larger structure units.

Fig. 5 shows the changes in the % recovery of the sludge with varying pH and settling time. An increase in the settling time with the lower pH value will increase the % recovery of the sludge. The response surface plot reveals that the pH had little interaction with the settling time. In the range of pH 4.5–5.5 and settling time at 90–120 min, the maximum percentage recovery of the sludge was 87.1 wt%.

Fig. 6 shows a result of the contribution of the interaction effect between the pH and flocculant (NALCO 7751) dosage. It can be seen that the percentage recovery of the sludge increased with an increase in the flocculant (NALCO 7751) dosage at 6500–7500 mg/L when pH was decreased from 5.5 to 4.5. Fig. 6 indicates that the highest percentage of the sludge is obtained at the intersection of zero levels of the other two factors. Because, the 3D surface and contour plot of the RSM are drawn as a function of two factors at a time, while the other two factors were fixed at the zero level.

Although high dosage required from *M. oleifera* coagulant and flocculant (NALCO 7751) for coagulation–flocculation process, the removal of suspended solids and % recovery of the sludge was improved in POME wastewater. Furthermore this treatment avoid of using chemical based like alum and PAC which have a number of disadvantages. Since the POME treat-

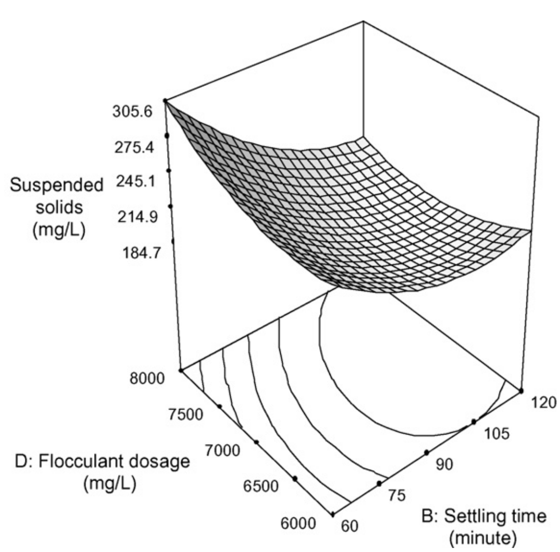

Fig. 4. Effect of flocculant (NALCO 7751) dosage and settling time on suspended solids; 3D surface graph and contour plot.

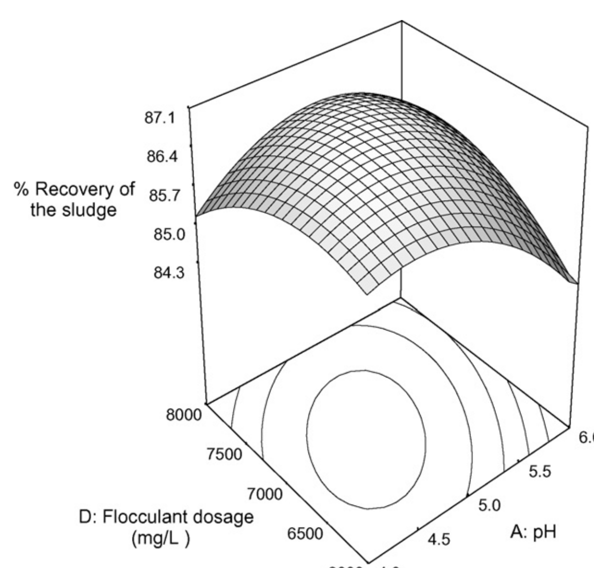

Fig. 6. Effect of pH and flocculant (NALCO 7751) dosage on % recovery of the sludge; 3D surface graph and contour plot.

Table 4
Optimum values of process variables

Run	pH	Settling time (min)	*M. oleifera* dosage (mg/L)	Flocculant (NALCO 7751), dosage (mg/L)	Suspended solid (mg/L)		Recovery of the sludge (wt%)	
					Experimental	Predicted	Experimental	Predicted
1	5	114	3469	6736	181	184	87	88
2	5	107	3374	6944	187	186	86	88
3	5	119	3761	6671	183	184	88	89
4	5	109	4075	6517	188	187	89	90
5	6	101	3342	7000	191	187	88	86

ment, the sludge obtained after treatment was biodegradable would not pose any disposal problem and could be used as an organic fertilizer.

4.4. Optimization analysis

In order to verify of the model developed, 5 more experiments were performed for suspended solids (mg/L) and percentage recovery of the sludge as shown in Table 4 by using the numerical optimization of the Design-Expert software based on the models proposed. A set of solution was generated by the Design-Expert 6.0.6 software to determine the optimum conditions of the process. The experiments were conducted at these conditions and comparison between the experimental results with the predicted results from the model was made. The minimum values of suspended solids and highest % recovery of the sludge were selected as an optimum values. The results demonstrated that the model prediction from Eqs. (3) and (4) for both responses agreed reasonably well with the experimental data. Thus, the optimum values of the process variables were: pH, 5; settling time, 114 min; *M. oleifera* dosage, 3469 mg/L and flocculant (NALCO 7751) dosage, 6736 mg/L. The suspended solids value was 181 mg/L and % recovery of the sludge was 87 wt% at the optimum conditions.

5. Conclusions

Response surface methodology involving an experimental design was used to evaluate the effects of pH, settling time, *M. oleifera* (after oil extraction) dosage and flocculant (NALCO 7751) dosage on the suspended solids (mg/L) and percentage recovery of the sludge and obtained the optimum value of the four process variables. The adequacy of the model was verified effectively by the validation of experimental data. Additionally, the optimum results show that the suspended solids removal from the raw POME (SS = 17,927 mg/L) was high after using the *M. oleifera* seeds (after oil extraction) and flocculant (NALCO 7751) with the 99% removal of suspended solids. A further advantage in the usage of *Moringa* seeds includes as safe, natural and environmental friendly coagulant. It could replace the conventional chemical coagulants such as alum and PAC. Additionally, 25 wt% an edible oil can be extracted from the seeds as a valuable product and oil extracted cake can be used as an effective coagulant in POME treatment. The sludge recovered can be disposed and used as an organic fertilizer. As a conclusion, the application of DOE technique was successfully applied in POME treatment. This technique could be extended for other research activities.

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