

Original paper

## Application of response surface methodology (RSM) for optimization of ammoniacal nitrogen removal from palm oil mill wastewater using limestone roughing filter

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### ABSTRACT

The creation of very pollute palm oil mill waste water has resulted in semiserious environmental hazards. The reason for the current study is to test the optimal removal of ammonia nitrogen ( $\text{NH}_3\text{-N}$ ) from palm oil mill waste water by filtration using inexpensive filters media in place of current methods, to remove ammonia nitrogen from palm oil mill effluent. A series of batch and column studies were conducted using a different particle size of limestone (4, 12 and 20 mm) at various filtration rates of 20 ml/min, 60 ml/min and 100 ml/min. An experimental model design was conducted using Central Composite Design (CCD) in Response Surface Methodology (RSM). RSM was used to calculate the outcomes of process variables and their role in reaching ideal conditions. Equilibrium isotherms in this study were evaluated using the Langmuir and Freundlich isotherm. Using statistical analysis, the  $\text{NH}_3\text{-N}$  removal model proved to be very significant with very low probability values (0.0001). The column study showed that ideal  $\text{NH}_3\text{-N}$  removal was attained using a lower flow rate and smaller sized limestone (LS). The ideal conditions found when using 4 mm limestone and a 20 ml/min flow rate. This resulted in 45.3% removal of  $\text{NH}_3\text{-N}$  which was seen in the predicted model, and fit well with the laboratory results (45%). The adsorption isotherm data fit the Langmuir isotherm.

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### 1. Introduction

Palm oil mill waste water is a natural effluent from palm oil industry (Hassan et al. 1996). Palm oil mill effluent (POME) is rich in natural carbon with a nitrogen content around 0.2 g/L, ammonia nitrogen 0.5 g/L and biochemical oxygen demand (BOD) higher than 20 g/L (Ma et al. 2001). In 1993, world palm oil production was 13.7 million tons. Malaysia alone created 7.4 million tons (Yusof 1994). Presently, there are over 270 palm oil mills in Malaysia. The average factory, produces about double the amount of POME compared to the amount of crude palm oil produced (Ma 1982; Qush 1991). Currently, approximately 85% of POME treatment is based on anaerobic and facultative ponding systems used by Malaysian palm oil mills (Rahim and Raj 1982; Wong 1980; Chan and Chooi 1982), which are known for long hydraulic retention time (HRT), which is regularly over of 20 d, and requires big plots of land or digesters (Chin et al. 1996). Currently POME is predominantly treated anaerobically in lagoons that release bio-gas into the atmosphere (Madaki and Seng 2013). In addition to adding to the greenhouse effect, a carbonaceous matter is created, which could be utilized for the profitable product. Unfortunately, the quality of after-treatment wastewater did not meet the discharge requirement fixed by the Malaysian Department of Environment, therefore further treatment is needed before it can be used (Zhang et al. 2008). Nutrient compounds such as nitrate ( $\text{NO}_3^-$ ), nitrite ( $\text{NO}_2^-$ ) and ammonia nitrogen ( $\text{NH}_3\text{-N}$ ), that are regularly found numerous kinds of wastewater and water, can find their way to rivers, drinking water reservoirs and lakes. Nitrogen is a vital nutrient to all living creatures. It is a fundamental building block of plants as well, it can be found in

animal proteins. Changes in microbes releases ammonia and if the concentration of  $\text{NH}_3\text{-N}$  exceeds 0.3–0.5 mg/L it can reduce the dissolved oxygen aquatic life needs in order to promote the growth of algae (AWWA 1990). Treatment of wastewater  $\text{NH}_3\text{-N}$  is necessary to alleviate environmental problems such as polluting, eutrophication and decomposition (Rozic et al. 2000). The removal of  $\text{NH}_3\text{-N}$  can be done chemically, physically, biologically or by using a combination of these methods. Air stripping, ion exchange, membrane filtration, adsorption, chemical precipitation, denitrification, biological nitrification, reverse osmosis and breakpoint chlorination are all available technologies used to carry out these processes (Metcalf and Eddy 2004). Standard wastewater treatment methods, are plagued by maintenance problems, operational issues and are costly to build. Easy maintenance, constant and dependable physiochemical treatment is more desirable than biological systems. Aguilar et al. (2002) studied physio-chemical removal of  $\text{NH}_3\text{-N}$  using activated silica, powdered activated carbon and precipitated calcium carbonate. They discovered that ammonia removal was lowered by 3–17%. Ion exchange often utilizes natural resins, which are extremely discerning but costly, though, less-expensive alternate natural and waste materials can be used instead. Numerous researchers have studied the efficacy of using different inexpensive materials for ammonia removal such as clay and zeolite (Sarioglu, 2005; Demir et al. 2002; Celik et al. 2001 and Rozic et al. 2000); limestone (Aziz et al. 2004a); organic and waste materials such as waste cement, discarded paper and concrete (Ahsan et al. 2001), activated sepiolite and sepiolite (Ozturk and Bektas 2004; Balci and Dincel 2002). Roughing filtration technology is a filtration process that utilizes a medium filter that has low filtration

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rates. It is chiefly used as pretreatment to lessen solid matter prior to slow filtration (Wegelin 1988; Boller 1993). This paper discusses laboratory investigations on the use of various sizes of limestone filter media through varied filtration rates for the removal of NH<sub>3</sub>-N using a roughing filter. Water samples tested in the experiment were taken from the wastewater of a palm oil mill in NibongTebal, Pulau Pinang.

**2. Materials and methods**

**2.1. Wastewater analysis**

POME and palm oil mill effluent which were obtained from the United Palm Oil Mill Sdn. Bhd, Nibong Tebal, Pulau Pinang were selected as the case study of the present research. The pounding POME treatment system has been employed to treat wastewater. This study involves two main data collection methods including field measurement and laboratory experiment. Field measurement included tests for pH and temperature, while laboratory experiments involved tests for Chemical Oxygen Demand (COD), Biological Oxygen demand (BOD<sub>5</sub>), Suspended Solids (SS), Colour, turbidity, and Ammonia Nitrogen. All of the tests were conducted in accordance with the Standard Methods for the Examination of Water and Wastewater (APHA 2005). The typical characteristics of raw POME are illustrated in Table 1.

**Table 1.** Palm oil mill effluent characteristics from a polishing pond.

Parameter	Unit	Min	Max	Average	*Standard
pH	-	7.89	8.77	8.33	5.0-9.0
Turbidity	NTU	200	650	425	-
COD	mg/l	2200	3300	2750	100
BOD <sub>5</sub>	mg/l	120	210	165	50
BOD/COD	-	0.054	0.063	0.058	-
Colour	PtCo	3000	5000	4000	-
SS	mg/l	400	730	652	400
NH <sub>3</sub> -N	mg/l	190	300	245	150
Temperature	C°	35	37	36	-

\* Department of Environmental Standards

**2.2. Materials**

Natural limestone media, provided from Perak, Malaysia, was used in this study. Prior to the experiment, the limestone was crushed into three media size: small size (4 mm), medium size (12mm) and large size (20mm). They were washed with water, and then dried. The composition of limestone used in this study was tested in the XRD Laboratory, School of Material and Mineral Resources Engineering, Engineering Campus, Universiti Sains Malaysia, as presented in Table 2.

**Table 2.** Composition of limestone.

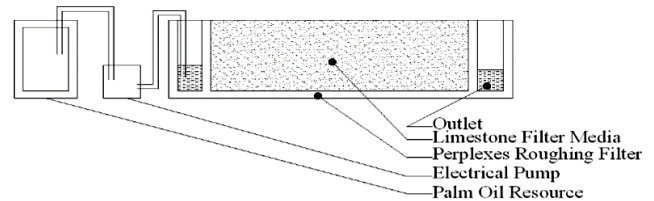
Component	Percentage (%)
MgO	1.722
SiO <sub>2</sub>	0.5212
CaO	56.5931
Fe <sub>2</sub> O <sub>3</sub>	0.0707
SrO	0.0376
NiO	0.0199
K <sub>2</sub> O	0.0243
CuO	0.0112
LOI	41

**2.3. Column study**

In this study, the horizontal roughing filter was used for the treatment. The laboratory filter made of a special Perspex material with 750 mm width, 200 mm length and 90 mm height, was filled with limestone in various sizes of 4 mm, 12 mm, 20 mm. The experiment was carried out at the Environmental Laboratory Two, School of Civil Engineering, Universiti Sains Malaysia. Filtration process was run with 3 different flow rates which were 20 ml/min, 60 ml/min and 100 ml/min (Table 3). Inlet and outlet samples were taken for each flow rate before and after the retention time for seven days consecutively. The schematic diagram of laboratory-scale roughing filter is shown in Fig. 1 Removal percentage, percentage=(c<sub>1</sub>-c<sub>2</sub>)/c<sub>1</sub>×100. Where C<sub>1</sub> and C<sub>2</sub> indicated untreated water and treated water concentration respectively.

**Table 3.** Operational parameters of the filter are shown.

Operational parameters	Column
Column heights	90 mm
Column lengthen	200 mm
Column material	Perspex
Width	750 mm
Volume of filter	13500 ml
Area of hols	3817 mm <sup>2</sup>
Adsorbent	Limestone
Particle size range	4 mm, 12 mm, 20 mm
Mode of flow	Horizontal
Flow rate	20 ml/min, 60 ml/min, 100 ml/min
Type of pump	Masterflex LS
Retention time	317 min, 260min, 106 min, 64 min, 88min



**Fig. 1.** Schematic of laboratory-scale roughing filter.

**2.4. Equilibrium studies**

In this study, adsorption isotherms were used to portray the media performance, and the relationship between the effects of dosage and settling times prior to the main experiment. Settling could examine the effect of particle sedimentation after shaking (Hussain et al. 2011). Different masses of media were used in 8 conical flasks from 25 g to 105 g, and the settling rate of the POME was observed for every 1 hour in 6 conical flasks for 6 hours, and shook at 350 rpm for 2 hours. Data for Freundlich and Langmuir isotherms generation were obtained by varying the amount of adsorbent dosage and different settling time. The isotherm constants and least squares correlation coefficients (R<sup>2</sup>) of both models were compared together. The amount of adsorption at equilibrium, q<sub>e</sub> (mg/g), was calculated by using the equation as shown below.

$$q_e = \frac{(c_0 - c_e)V}{M} \tag{1}$$

where C<sub>e</sub> and C<sub>0</sub> (mg/L) are the liquid-phase concentrations of equilibrium conditions and NH<sub>3</sub>-N at initial, respectively. V (L) is the volume of the POME and M (g) is the mass of limestone used.

**2.5. Experimental design and analysis**

Experimental design of the process for optimum removal of NH<sub>3</sub>-N from palm oil mill waste was carried out using RSM. The RSM is a collection of mathematical and measurable systems that are valuable for the advancement of synthetic responses and mechanical procedures, and are regularly utilized for experimental designs (Bas and Boyac 2007). In this study, RSM was used to evaluate the connection between independent variables and response (ammoniacal nitrogen removal, %), as well as to enhance the applicable circumstances of variables in order to forecast the best value of responses. Central Composite Design (CCD), which is the most commonly used type of RSM, was used to define the effects of operational variables on ammoniacal nitrogen removal efficiencies. According to Guven et al. (2008), CCD is a useful design that is appropriate for sequential experimentation because it enables a realistic amount of information to be used to examine lack of fit when a suitable number of experimental values exist. RSM and CCD were generated from the Design Expert 10.02 software program. The two significant independent variables considered in this study were flow rate (A) and size of limestone (B) as shown in Table 4. Based on some initial experiments, each independent variable was different over three levels between -1 and +1 at the determined ranges. Thirteen experiments were improved with six duplications to assess the pure error. The applicable model is the quadratic model characterized by Eq. (2).

$$Y = \beta_0 + \sum_{i=1}^k \beta_i X_i + \sum_{i=1}^k \beta_{ii} X_i^2 + \sum_{i < j}^k \beta_{ij} X_i X_j + \dots + e \quad (2)$$

where, Y is the response;  $\beta_0$  is a constant coefficient;  $\beta_i$ ,  $\beta_{ij}$  and  $\beta_{ii}$  are the interaction coefficients of quadratic terms, respectively; k is the number of studied factors;  $X_i$  and  $X_j$  are the variables and  $e_i$  is the error. The value of correlation coefficient ( $R^2$ ) represents the quality of the fit of the quadratic model. The key indicators that show the significance and fitness of the model used include the Adequate Precision, F-value model (Fisher variation ratio) and probability value (Prob>F) (Arslan 2009; Meyrs and Montgomery 2002). Immediate consideration of multiple responses contains the initial creation of a suitable response surface model. Then, after identifying a set of operational conditions at the maximizes the targeted or minimum maintains response, in the ranges (Bas and Boyac 2007; Meyrs and Montgomery 2002).

### 3. Results and discussion

A total of 13 runs of the CCD experimental design and response are shown in Table 4. The observed percent removal efficiencies of  $NH_3-N$  were varied between 4 and 45%.

**Table 4:** Experimental design for treatment of POME using roughing filter limestone

Std	Run	Factor 1 Flow rate (ml/min)	Factor 2 Limestone (mm)	Response $NH_3-N$ (mg/l)
11	1	60	12	30
8	2	60	20	13
12	3	60	12	34
6	4	100	12	22
5	5	20	12	38
10	6	60	12	33
9	7	60	12	32
2	8	100	4	37
1	9	20	4	45
4	10	100	20	4
7	11	60	4	39
3	12	20	20	11
13	13	60	12	33

#### 3.1. Analysis of variance (ANOVA)

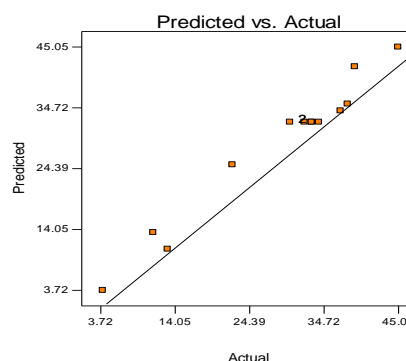
Table 5 illustrates the analysis of variance (ANOVA) of the variable for the predicted response surface quadratic model for ammonia nitrogen removal efficiency. According to Table 5, a low probability value ( $F < 0.0001$ ) and the F-value of 47.28; showed that the model was significant for  $NH_3-N$  removal. The model is not significant when values of  $P > F$  are greater than 0.1000 while values of  $P > F$  that are less than 0.0500 signify that the model terms are significant (Körbahti and Tanyolaç 2008). The "Adequate Precision" ratio of the model was 22.25 (Adequate Precision >4), which was an adequate signal for the model (Ölmez 2009). The Lack of Fit (LOF) for response was insignificant relative to pure error. An insignificant (LOF) is preferable because to create a model that fits the experimental data is the primary objective (Hosseini 2012). The value of correlation coefficient ( $R^2=0.97$ ) obtained in the present study for  $NH_3-N$  removal was higher than 0.80, indicated that only 3.78% of the total dissimilarity might not be explained by the empirical model. Joglekar and May (1987), mentioned that for a good fit of the model the correlation coefficient should be at a minimum of 0.80. High  $R^2$  value demonstrated a decent agreement between the observed results and calculated within the range of the experiment. The coefficient of variance (CV) is determined as a ratio of the standard error of assessment to the average of the observed response explained by the reproducibility of the model. CV more than 10%, shows model is considered reproducible (Aziz et al. 2011). In this study, A, B,  $B^2$ , were significant model terms. Insignificant model terms, which have limited influence such as  $A^2$  and AB, were kept out from the study to recuperate the model. The final regression model in terms of their coded factors is explicit by the following second-order equation below.

$$NH_3-N \text{ removal, \%} = 35.41 - 5.22A - 9.23B - 2.09A^2 - 3.14B^2 + 0.18 AB \quad (3)$$

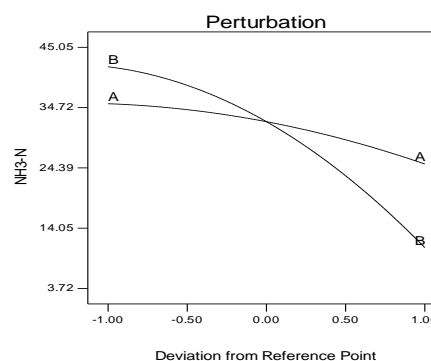
An empirical relationship between  $NH_3-N$  removal efficiency and process variables in terms of actual factors can show by the following second-order equation below.

$$NH_3-N \text{ removal, \%} = 45.48491 + 0.017924A + 0.29795B - 1.30388E-003A^2 - 0.095097B^2 + 7.81250E-004AB \quad (4)$$

Ultimately, it is important to verify that the selected model provides adequate conjecture of the real system. The model adequacy is evaluated by applying the diagnostic plots provided by the Design Expert 10.0.2 software, such as predicted versus actual value plots and normal probability plots of the residuals. As presented in Fig. 2, the predicted values of  $NH_3-N$  removal efficiency attained from actual experimental data and the model were in good agreement (Myers et al. 2016). Perturbation plot (Fig. 3) exhibited the comparative effects of two independent variables on  $NH_3-N$  removal efficiency. In Fig. 3, a sharp curvature in flow rate (A) and size of limestone (B) showed that the response of  $NH_3-N$  removal efficiency was slightly sensitive to these two process variables



**Fig. 2.** Design Expert plot; predicted versus actual.



**Fig. 3.** Perturbation plot for  $NH_3-N$  removal values plot for  $NH_3-N$  removal.

**Table 5.** ANOVA for analysis of variance and adequacy of the quadratic model.

Source	Sum of Squares	Degree of freedom	Mean square	F-value	Prob>F
Model	1766.19	5	353.38	47.28	0.0001
A	152.62	1	152.62	20.42	0.0027
B	698.36	1	698.38	93.43	0.0001
$A^2$	12.02	1	12.02	1.61	0.2453
$B^2$	102.31	1	102.31	13.69	0.0077
AB	0.25	1	0.25	0.033	0.8601
Residual	52.32	7	7.47		
Lack of Fit	43.12	3	14.37	6.25	0.0544
Pure error	9.20	4	2.30		

SD = 2.73,  $R^2 = 0.97$ ,  $R^2_{adj} = 0.95$ , Adeq Precision=22.25, CV = 9.58

#### 2.2. Ammoniacal nitrogen removal efficiency

The 3D surface responses of the quadratic model and contour plots were determined by utilized to assess the relationships between response and independent variables and design 10.02 software. (Fig. 4), illustrates that the  $NH_3-N$  removal increased with the decrease of

limestone size and flow rate. The maximum and minimum of NH<sub>3</sub>-N removal efficiencies obtained by the roughing filter were 45% (limestone size = 4 mm, flow rate = 20 ml/min) and 4% (Limestone size= 20 mm, flow rate = 100 ml/min), respectively. Faster flow rate causes lesser time required for the particle to travel along the settling

distance and stick onto the media layer or be absorbed (Muhammad et al. 1996; Nwonta and Ochieng 2009). The results showed that when the filter media was small and the flow rate was low, the NH<sub>3</sub>-N removal was more effective. These findings are in agreement with research reported by Maung (2006).

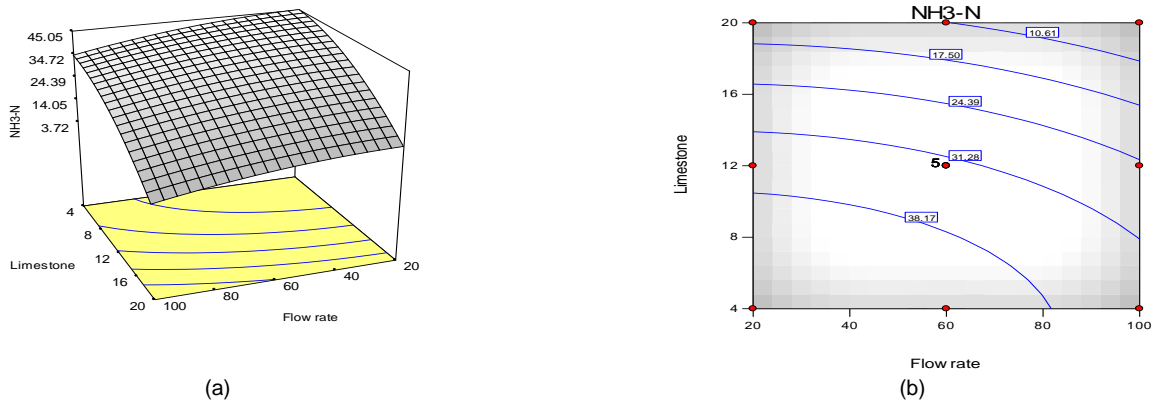


Fig. 4. Response surface (a) and contour plots (b) for NH<sub>3</sub>-N removal efficiency as a flow rate (ml/min) and size of limestone (mm).

3.3. Optimization of operational conditions

For determining the optimum value of ammonia nitrogen removal efficiency using the Design Expert 10.02 software. According to the software optimization step, the desired goal the response (NH<sub>3</sub>-N removal efficiency) was defined as “maximum” while for each operational condition (limestone size mm and flow rate ml/min) was chosen “within the range” to achieve the high performance. The program brings together the individual desirability into a single number, and then searches to maximize this function. Consequently, the optimal operating conditions and respective percent of removal efficiencies were established, and the results are presented in Table 6. As shown in Table 6, 45.3% NH<sub>3</sub>-N removal was predicted according to the model under optimized operational conditions (limestone size mm and flow rate ml/min). The desirability function value was found to be 1.0 for these optimum conditions. The optimum results were then confirmed by an additional experiment. Forty-five percent removal of NH<sub>3</sub>-N was obtained from the laboratory experiment which fits well with the predicted response value.

Table 6. The responses at an optimum condition for maximum removal.

Parameters	Flow rate (ml/min)	Limestone (mm)	Removal	Desirability
NH <sub>3</sub> _N	20.40	4.01	45.03	1.000

3.4. Adsorption isotherms

Equilibrium isotherms in this study were examined using the Langmuir and Freundlich isotherms. Langmuir and Freundlich's isotherms are the isotherm models used most of often for describing adsorption characteristics of the adsorbents used in water and wastewater treatments (Isa et al. 2007; Chabani et al. 2007). The Langmuir isotherm theory assumes monolayer coverage of adsorbate over a homogeneous adsorbent surface (Langmuir 1918). The linear form of Langmuir isotherm equations is given as:

$$\frac{1}{x/m} = \frac{1}{QbC_e} + \frac{1}{Q} \tag{5}$$

where, b (L/mg) and Q (mg/g) are the Langmuir constants related to rate of adsorption and adsorption capacity, separately, C<sub>e</sub> (mg/L) is defined as the equilibrium concentration of the adsorbate and x/m (q<sub>e</sub>) is the quantity of adsorbate per unit of adsorbent (mg/g). A straight line was obtained when 1/(q<sub>e</sub>) was plotted against 1/C<sub>e</sub>. While b was deduced from the intercept Q was calculated from the slope (Fig. 5 (a) and it shows that equilibrium data were fit with the Langmuir model. The R<sup>2</sup> value and constants are listed in Table 7. The features of the

Langmuir isotherm can be explicit in terms of equilibrium parameter RL (Weber and Chakravorti 1974), which is shown by:

$$R_l = \frac{1}{1 + bC_e} \tag{6}$$

where, C<sub>0</sub> (mg/L) is the initial nitrogen ammonia concentration and b is the Langmuir constant. The value of RL exhibit the type of isotherm to be unfavorable (RL>1), linear (RL=1), and favorable (RL<1) (Isa et al. 2007). As shown in Table 7, the RL value for adsorption of NH<sub>3</sub>-N onto limestone was 0.154, indicating that the adsorption process was favorable. The Freundlich model is an empirical equation, which assumes that the adsorption process takes place on heterogeneous surfaces (Freundlich, 1906). The Freundlich isotherm equations is given as:

$$\log q_e = \log K + \frac{1}{n} \log C_e \tag{7}$$

where, q<sub>e</sub> (mg/g) is the amount of ammonia nitrogen adsorbed per unit mass of adsorbent; C<sub>e</sub> (mg/L) is defined as the equilibrium concentration of the adsorbate; n and K are Freundlich constants with exhibit the favorability of the adsorption process; K (mg/g (L/mg)<sup>1/n</sup>) shows the adsorption capacity of the adsorbent. The value of 1/n<1 demonstrates that the adsorption capacity rises and the adsorption mechanism is favorable. The value of 1/n>1 indicates that the adsorption process is weak and the adsorption mechanism is unfavorable (Hussain et al. 2007; Aziz et al. 2008; Aziz et al. 2011). The linear correlation-regression (R<sup>2</sup>) and values of K for Freundlich isotherm are given in Table 8. It can be seen from Table 7 that the Langmuir isotherm fit the data better than the Freundlich. This is also confirmed that the higher value of R<sup>2</sup> for Langmuir (0.888) compared to Freundlich (0.838), exhibited that the adsorption of NH<sub>3</sub>-N occur as monolayer adsorption on a homogenous surface in adsorption attraction. Accordingly, the results show, the Langmuir isotherm described the adsorption isotherm data and the adsorption capacity obtained was 0.231× 10<sup>-3</sup> mg/g. This result is thought to be low when compared to others who also used limestone to remove NH<sub>3</sub>-N from wastewater.

Table 7. Langmuir isotherm parameters for NH<sub>3</sub>-N adsorption.

Media	B(L/mg)	Q (mg/g)	R <sup>2</sup>	R <sub>L</sub>
Limestone	0.023	4.658×10 <sup>-5</sup>	0.888	0.154

Table 8. Freundlich isotherm parameters for NH<sub>3</sub>-N adsorption.

Media	K <sub>F</sub>	1/n	R <sup>2</sup>
Limestone	0.231× 10 <sup>-3</sup>	-0.256	0.838

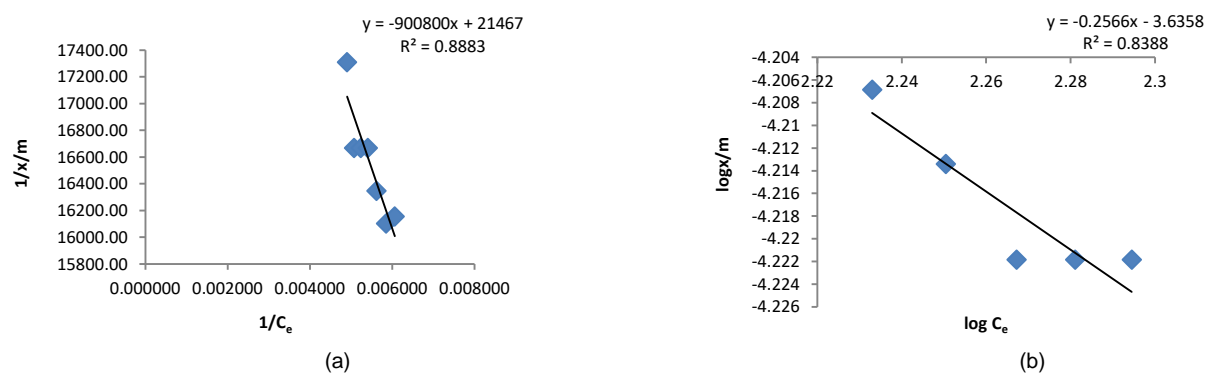


Fig. 5. (a) Langmuir isotherm and (b) Freundlich isotherm for NH<sub>3</sub>-N adsorption.

#### 4. Conclusions

In the present study, optimization of ammoniacal nitrogen removal from palm oil mill effluent using horizontal roughing filter and limestone as a filter media was investigated. Optimization of the treatment process was concentrated on the influence of operating variables and interaction among all the parameters, such as flow rate and size of limestone using CCD in RSM. The multiple correlation coefficients of determination  $R^2$  was 0.97, showing that the actual data

fitted well with the predicted data. The adsorption data fitted well with the Langmuir isotherm at  $R^2=0.888$ . The optimum results attained from the model indicated that 45% of NH<sub>3</sub>-N removal was achieved at limestone size of 4 mm, and flow rate of 20 ml/min, while and 4% removal was obtained at limestone size of 20 mm, and flowrate of 100 ml/min. Faster flow rate requires lesser time for the particle to travel along the settling distance and stick onto the media layer or be absorbed. The results showed that when the filter media was small and the flow rate was low, the NH<sub>3</sub>-N removal was more efficient.

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