Journal of Environmental Science and Technology

ISSN 1994-7887

Journal of Environmental Science and Technology 9 (1): 111-120, 2016
ISSN 1994-7887 / DOI: 10.3923/jest.2016.111.120

Removal Efficiency of Ammoniacal Nitrogen from Palm Oil Mill Effluent (POME) by Varying Soil Properties

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ABSTRACT

Palm Oil Mill Effluent (POME) contains large amounts of organic matters and nutrients. Instead of discharging into oxidation ponds, POME can be used as an alternative option to replace inorganic fertilizers which have been known to increase the hardness and acidity of the soil over time. This study investigated the effect of different soil properties on the ammoniacal nitrogen removal efficiencies in POME. The ammoniacal nitrogen removal efficiencies from three mediums over 7 days of retention times were studied. Medium I contained major amount of sand, whereas medium II contained major amounts of clay and lastly medium III contained large amounts of silt and clay. The results showed that medium II produced the highest removal efficiency of ammoniacal nitrogen in POME, with the average removal efficiency of 77±5.1%. The average values of removal efficiencies obtained from mediums I and III were 61±6.2 and 58±11.3%, respectively. In addition, the removal efficiency of ammoniacal nitrogen increased slowly with the retention time. Medium II recorded the highest removal rate ($k = 0.0897$ day^{-1}) compared to mediums I ($k = 0.0435$ day^{-1}) and III ($k = 0.0492$ day^{-1}). The mechanism of removal ammoniacal nitrogen from the medium occurred *via* absorption by the soil particle.

Key words: Clay, sand, silt, removal of ammonical nitrogen, first order kinetic model

INTRODUCTION

Malaysia is one of the biggest palm oil exporters in the world. Based on the recent data published by the Malaysian Palm Oil Board (MPOB., 2014), the total amount of crude palm oil production in 2014 was about 19.67 million t, in which generated a total of 63.62 billion Ringgit Malaysia (~USD 17.74 billion) in export revenue in the same year. However, the rapid growth of palm oil related industries has also led to significant pollution of air, water and soil (Wu *et al*., 2007; Ahmad *et al*., 2009).

Palm Oil Mill Effluent (POME) is one of the major wastes discharged from the mill, which contains rich amount of organic matters and nutrients (Ahmad *et al*., 2009). Ahmad *et al*. (2005) reported that the production of 1 t crude palm oil (COD) generates about 2.5-3.5 m^3 of POME. Typically, POME consists of 60,000-40,000 mg L^{-1} of total suspended solids, 6000 mg L^{-1} of oil and grease, 50,000 mg L^{-1} of chemical oxygen demand (COD) 30,000-25,000 mg L^{-1} of biochemical oxygen demand (BOD), 750 mg L^{-1} of total nitrogen and 220-120 mg L^{-1} of ammoniacal nitrogen (DOE, 1999; Ahmad *et al*., 2005; Ujang *et al*., 2010; Chin, 2013). These water quality characteristics account to the elevated levels of organic substances in POME which necessitate efforts to treat

POME. One of the most widely used technology to treat the POME is anaerobic process due to the ease of operation and low cost. However, this technology is rather slow, low in efficiency and demands large piece of land to accommodate different stages of oxidation ponds (Rupani *et al*., 2010).

In addition to the large amount of wastewater generated from palm oil mill, oil palm plantation has also occupied large piece of forest land. Up to December 2014, the amount of lands used for the plantation of this crop in Malaysia was 5.39 million ha (MPOB, 2014). Since oil palms uptake large amount of nutrients such as ammoniacal nitrogen (NH_3-N), phosphates (PO_4^{3-}) and minerals from soil, regular or continuous fertilizing becomes crucial to maintain the availability of nutrients in the soil. The usage of inorganic fertilizers such as urea increases the hardness and acidity of the soil (Opala *et al*., 2012; Ogbodo, 2013) which eventually cannot be used for other types of crops. In fact, some studies have found that the reuse of POME in agriculture can promote soil amendments and improvement of crops (Oviasogie and Omoruyi, 2007; Iwara *et al*., 2011; Okpamen *et al*., 2014). In a study conducted by Shamshuddin *et al*. (1992), the reuse of POME to acidic soils can actually increase the pH value and cation exchange capacity (CEC) of soil. Despite number of studies have been conducted on the reuse of POME for plantation purpose; these studies mainly focused on the improvement of soil properties leaving a knowledge gap about the pollutants in POME which may potentially pollute the ground water.

Therefore, the objective of this study was to investigate the removal efficiency of NH_3-N from POME using three different types of soil mediums. Soil texture, organic matter and moisture content were also determined as these parameters directly affect the absorption and infiltration of nutrients in the soil. From the results of this study, we are able to know which type of soil texture gave the best removal efficiency of NH_3-N through absorption process in the soil.

MATERIALS AND METHODS

Sampling of POME: The POME samples were sampled from Bau Palm Oil Mill Sdn Bhd, Sarawak, Malaysia. Three samplings were performed between October and December 2014. Two liters of POME were collected from the drain that channels POME from the mill to the cooling ponds using a polyethylene bottle. Upon collection, samples were refrigerated at 4°C prior to further analysis.

Soil sample: Three types of mediums were used in this study. All the soils were sampled manually from Universiti Malaysia Sarawak campus area whereas the sand sample was supplied by Sand Co. Sdn Bhd located in Batu kawah, Kuching, Sarawak, Malaysia. The soils and sand were kept in plastic container without further treatment until leaching tests were conducted.

Soil analyses: Soil analyses were carried out to determine the soil texture, organic matter and moisture content of the soil sample. All soil analyses were performed according to the United States Department of Agriculture soil standard methods (USDA., 1996).

The soil sample was air-dried for 3 days in the laboratory. The air-dried soil samples were subsequently grounded into powder using mortar and pestle. The fine powder was filtered using a 2 mm sieve and 0.25 mm sieve and kept in air tight container until further analysis.

Particle size analysis: The principle in pipette method for soil texture determination was based on mechanical dispersions of soil and sedimentation of soil based on Stoke's Law as shown in Eq. 1:

$$T = \frac{1.8\times10^{-7}nh}{\{d^2\ g(\rho_s-\rho)\}} \quad (1)$$

Where:

t = Sedimentation time
d = Diameter of particle (m)
ρ_s = Density of particle (Mg m^{-3})
ρ = density of water (Mg m^{-3})
h = Depth of water (m)
n = Viscosity of medium (mPa sec)
g = Gravimetric acceleration (m sec^{-2})

Equation 1 describes that particles precipitate through a dispersion medium (water) under gravimetric accretion. The particle size was estimated based on the rate at which particles sink through a fluid suspension. The standard method is summarized as follows. Soil sample (10.0 g) and 50 mL of 6% H_2O_2 was mixed together and left overnight. The soil organic matter was decomposed by heating at 290°C. Then, the pH was adjusted to 3.5 with 0.1 M HCl or 0.1 M NaOH and left overnight to allow the precipitation of clay particle. The supernatant formed was discarded by decantation and added with deionized water (300 mL). The pH was again adjusted to 10.5 using 0.1 M NaOH and the solution was transferred to a 1 L graduated cylinder. Particle size with heavier density falls faster than small size and less dense particle. Thus, sand settled the fastest within 1 min leaving clay and silt in the suspension. After 1 day, the suspensions were then shaken for 1 min and settled according to the settling time (clay fraction: 6 h 56 min, silt+clay fraction: 4 min 10 sec). Based on the settling time, the 10 mL of water suspension for both fractions was pipetted at 10 cm depth of water and transferred to weighing bottles. The samples were then oven-dried at 105°C for overnight. Percentage of silt, clay and sand were determined by the difference on the different fractions obtained at specific time intervals. All soil texture analyses were performed in triplicates. Calculation of clay, silt and sand percentages were based in Eq. 2-4:

$$\text{Clay (\%)} = (x_2\text{-}x_1)\times100\times10 \quad (2)$$

$$\text{Silt (\%)} = [(y_2\text{-}y_1)\text{-}(x_2\text{-}x_1)]\times100\times10 \quad (3)$$

$$\text{Sand (\%)} = 100\text{-}(\text{clay\%}+\text{silt\%}) \quad (4)$$

Where:

x_1 = Weight of weighing bottle
x_2 = Weight of clay fraction
y_1 = Weight of weighing bottle
y_2 = Weight of silt and clay fraction

Determination of Soil Organic Matter (SOM) and Moisture Content (MO): The SOM and MO content were determined based on loss on ignition method (USDA., 1996). Firstly, all the crucibles were labelled and oven-dried for 24 h at 105°C. Then, the crucibles were allowed cooled to room temperature and weighted. These processes were repeated twice to obtain constant readings. Soil sample (3 g) was placed into the crucible and heated in an oven for 24 h at 105°C.

The weight of the oven-dried sample was measured and recorded. The oven-dried sample was then heated in the furnace for 8 h at 500°C. Lastly, the weight of the furnace-dried samples were measured and recorded. The MO was measured using Eq. 5 and 6, whereas, SOM was obtained via Eq. 7:

$$\text{MO (\%)} = \frac{\text{Initial weight of soil before oven} - \text{Weight of soil after oven}}{\text{Intial weight of soil before oven}} \times 100 \quad (5)$$

$$\text{Mineral content} = \frac{\text{Weight of soil after furnace (g)}}{\text{Weight of soil after oven (g)}} \times 100 \quad (6)$$

$$\text{SOM (\%)} = 100\text{-mineral content} \quad (7)$$

Pre-treatment of soil: A mixture of soil and sand sample (100 g) was soaked in 500 mL of distilled water with different retention times of 1, 2, 3, 4, 5, 6 and 7 days. The leachate was separated from the soil and sand sample was analyzed for NH_3-N content, while the soil and sand sample was air-dried for column treatment.

Experimental design: A single column was packed with their respective mediums with the length of 20 cm (Fig. 1). For each medium, similar retention times of 1-7 days were tested. The NH_3-N content before and after passing through the column was analyzed.

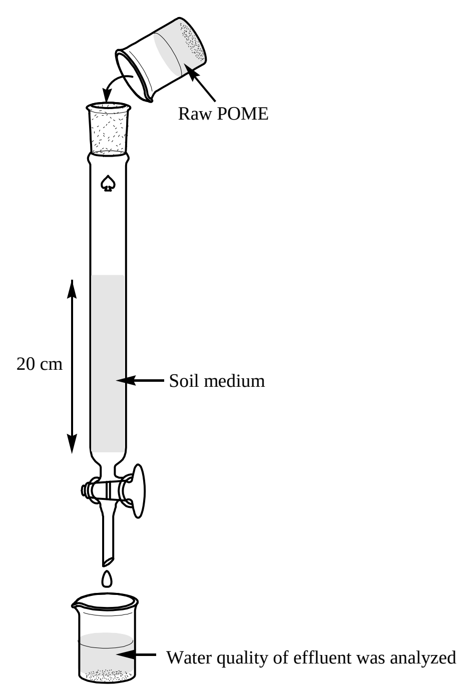

Fig. 1: Single column set up for POME treatment using soil as the medium

NH_3-N analysis: The content of NH_3-N in the leachates, raw POME and effluent were analyzed according to HACH method (HACH 8038). A spectrophotometer (HACH DR2800) was used to determine the NH_3-N concentration.

Analytical method and data processing: To evaluate the removal efficiency data obtained from the column study, all the data obtained was analyzed using SPSS statistics program, one-way analysis of variance (ANOVA) and Microsoft Excel 2007 for data processing and presentation. The dependent variable measured was NH_3-N while the independent variable was the retention time. The differences detected in statistical analysis were considered highly significant at or below 0.05 probability level.

RESULTS AND DISCUSSION

Soil analyses: The results of soil particle size, SOM and soil moisture MO are tabulated in Table 1.

Soil of mediums I, II and III are classified as sandy clay loam soil, sandy loam and clay loam soil, respectively. The percentage of clay in soil increased in the order medium I<medium III<medium II, while, the sand content of all mediums is arranged in the order of medium I>medium II>medium III. When the percentage of sand increased, the amount of grit also increased. On the contrary, the plasticity (capacity to mold) increases as the clay content increases.

The SOM and MO which also contributes to the liquid infiltration in soil of each medium were also determined in conjunction. It was found that soil of mediums I and II demonstrated minimal difference from each other, where the SOM was 8.41 and 8.16%, respectively, while the MO content was 14.11 and 14.25%, respectively. In comparison to mediums I and II, medium III contained the lowest values of SOM and MO, 7.69 and 11.38%, respectively. An increase in organic matter contents in the soil led to a greater pore space which can be linked to readily infiltration and higher retention of water in soil (Roth, 1985). As a result of this, the readily infiltration capacity improves the soil water holding capacity. FAO (2015) also indicated that organic matters contribute to the stability of soil aggregates and pores by bonding or adhesion properties of organic materials. Therefore, it can be concluded that mediums I and II have relatively higher soil porosity than that of medium III.

Leachate results: Prior to column treatment of POME, blank analyses were performed to determine the concentration of NH_3-N that can be leached out from the mediums in different retention times (from day 1-7). The results obtained from the blank analyses were used as the baseline reading when POME was added into each medium. By subtracting the blank value, the actual concentration of the effluents for each medium can be calculated.

The average amounts of NH_3-N leached from mediums I, II and III in different days are depicted in Fig. 2. For mediums I and III, the highest concentration of NH_3-N leached was 0.843 and 1.05 mg L^{-1} on the day 2, respectively, meanwhile for medium II, the highest concentration of NH_3-N leached was recorded at day 4 with 0.17 mg L^{-1}. Composite medium II

Table 1: Results of soil texture composition, SOM and MO of each soil medium

Medium	Clay (%)	Silt (%)	Sand (%)	SOM (%)	MO (%)	Color	Soil class
I	25	14	62	8.41	14.11	Red	Sandy clay loam
II	38	18	44	8.16	14.25	Orange	Sandy clay
III	29	30	41	7.69	11.38	Brown	Clay loam

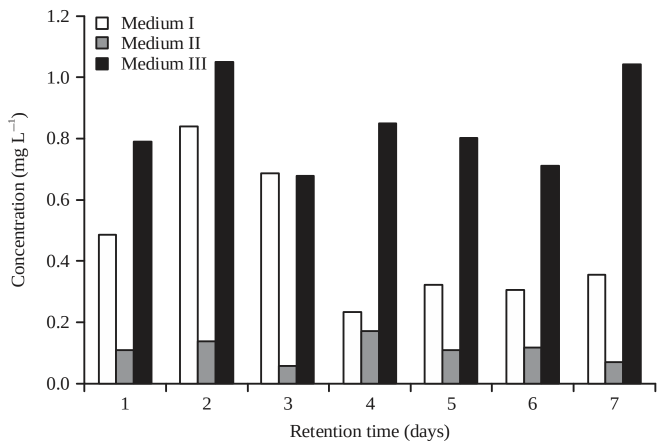

Fig. 2: Average concentration of NH_3-N from different mediums at different retention times

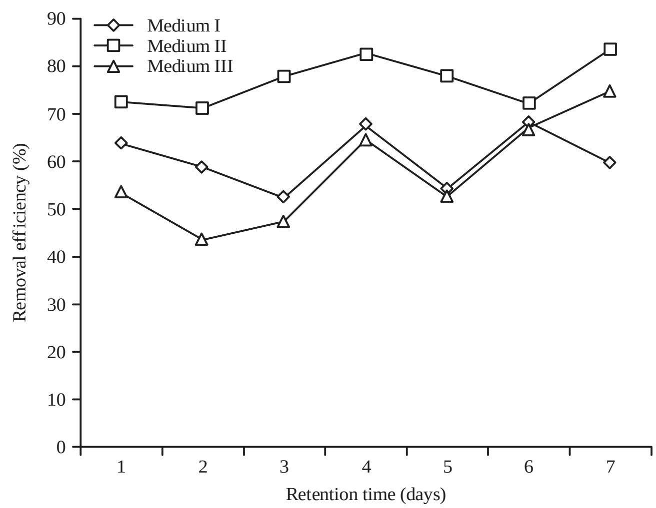

Fig. 3: Removal efficiencies of NH_3-N for mediums I, II and III from day 1-7

which contains the highest amount of clay has the greatest ability to hold water and nutrients. This is due to the fact that clay contains flat sheets of silicon dioxide (SiO_2) alternating with sheets of alumina (Al_2O_3) molecules. The tetravalent cations of silicon (Si^{4+}) in clay are often replaced by aluminum ion (Al^{3+}) and results a net negative charge on the surface of clay particles. With this negative charges present at the surface, clay has ability to hold K^+, Na^+ and NH_4^+. In addition to these cations, clay also absorbs other ions such as Ca^{2+}, Mg^{2+}, H^+, SO_4^{2-}, Cl^-, PO_4^{3-} and NO_3^-, which made clay as excellent adsorbent materials (Cadena *et al*., 1990; Srinivasan, 2011). As a result, the least NH_3-N amount was leached out from medium II. The higher reachability of NH_3-N from mediums I and III could be due to the sandy soil which has less porosity and lower water and nutrient retention abilities.

Removal efficiency of NH_3-N using different mediums: The removal efficiency of NH_3-N before and after POME passing through the column was investigated to a function of retention times from day 1-7. Figure 3 shows the removal efficiencies of NH_3-N against retention times.

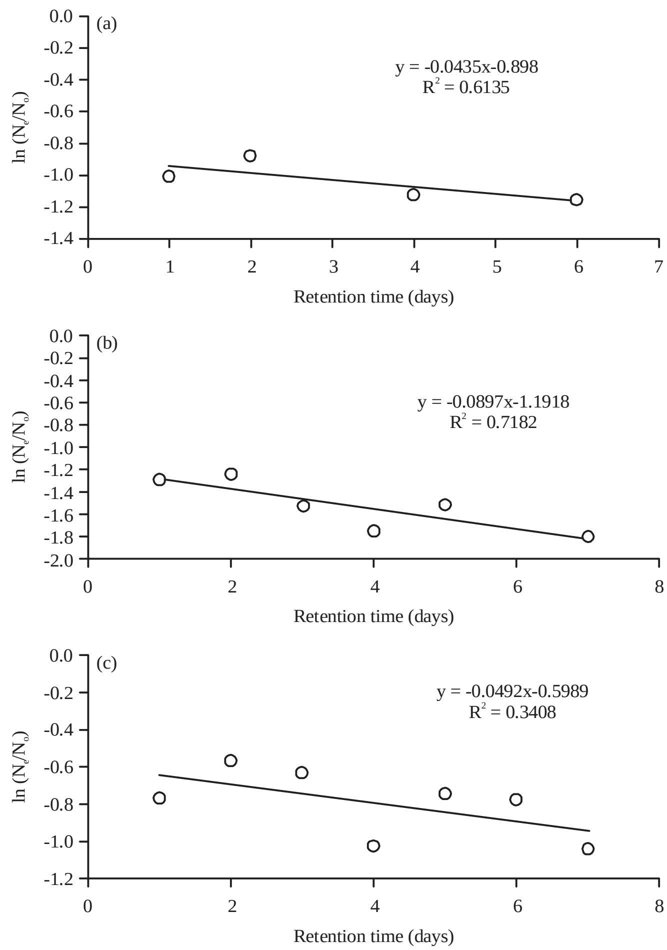

Fig. 4(a-c): Plots of ln (N_e/N_o) versus retention time of (a) Medium I, (b) Medium II and (c) Medium III

The removal efficiencies of NH_3-N using medium I (sandy clay loam) ranged from 52-68%, whereas for medium II (sandy clay), the removal efficiency varied from 71-84% and lastly for medium III (clay loam), the removal efficiency ranged from 44 and 75%. In line to the results obtained from the leaching study, medium II has the highest removal efficiency because most of the NH_3-N in POME was absorbed when it passed through the column. This is due to the fact that medium II has the highest clay amount in its component which can hold NH_4^+ (Srinivasan, 2011). In general, clay has finer particles, higher porosity, surface area, SOM and MO compared to sand and silt. Ball (2001) stated that soil with higher SOM tend to have a higher water holding capacity due to its affinity for water, in addition, the surface area is large, the absorption ability increases because of increase spaces for nutrients adsorption (Hossain, 1999).

Based on Fig. 3, we found that the removal efficiency for each medium gradually increased with the increase of retention time. The first order kinetic model was used to fit this set of data. In the first order kinetic model analysis, the NH_3-N concentration in effluent (N_e) was over with the initial concentration of NH_3-N (N_o) before passing the column. The results can be seen in Fig. 4a-c. The rate constant, k, was obtained from the slope of the graph ln (N_e/N_o) versus retention time (days). In fact, mathematical relationship in between k value and the removal efficiency of NH_3-N has been

Table 2: Mean removal efficiencies (%) and standard deviations (SD) of NH3-N in mediums I, II and III from different retention times

Retention time (days)	1	2	3	4	5	6	7	Mean	SD
Medium 1	63.75	58.74	52.31	67.60	54.17	68.28	59.55	60.63	6.22
Medium 2	72.41	71.19	77.76	82.60	78.07	72.03	83.56	76.80	5.09
Medium 3	53.70	43.62	47.44	64.54	52.72	66.90	74.82	57.68	11.32

Table 3: Results of ANOVA test for all mediums

					95% confidence interval	
Medium (I)	Medium (J)	Mean difference (I and J)	SE	p-value	Lower Bound	Upper bound
I	II	-16.17429*	4.28495	0.004	-27.1102	-5.2384
	III	2.95143	4.28495	0.773	-7.9845	13.8873
II	I	16.17429*	4.28495	0.004	5.2384	27.1102
	III	19.12571*	4.28495	0.001	8.1898	30.0616
III	I	-2.95143	4.28495	0.773	-13.8873	7.9845
	II	-19.12571*	4.28495	0.001	-30.0616	-8.1898

*Mean difference is significant at the 0.05 level

widely used in constructed wetland wastewater treatment systems (Reed, 1985; Lee *et al*., 1999; Lim *et al*., 2003). The rate constant indicates how fast of the medium to remove NH_3-N in POME, the greater the k value, the faster the removal.

All mediums produced negative slope indicating the removal efficiency increased with the retention times. The k values in all the plots were in the range of 0.0435-0.0897 day^{-1} with the poor coefficient, R^2 values (<0.900). Despite with the poor R^2 value, medium II yielded the best linear regression R^2 value to the first order kinetic model with the highest k value 0.0897 $days^{-1}$. The k values of mediums I and III were found to be about half of that of k value of medium II, which reveals that the absorption rate of mediums I and III were two magnitudes slower than medium II.

Mean comparisons between mediums: The mean values of removal efficiencies of NH_3-N for all the mediums along the 7 days treatment, were calculated (Table 2) and analyzed using one-way analysis of variance (ANOVA) (Table 3).

The mean removal efficiency for medium I was 61% with SD of 6%. Medium II showed the highest average removal efficiency value, 77% and SD of 5%. Meanwhile, the mean removal efficiency value of medium III was 58% with SD of 11%.

Mean comparisons were conducted to determine which treatments were responsible for the significant p-values. Multiple comparisons were conducted to find which means differ significantly using ANOVA Test. The results from ANOVA test showed that there was significant difference between the mean of mediums I and II as well as between mediums II and III because the p-values were 0.004 and 0.001, respectively. Meanwhile, there was no significant difference in term of the removal efficiency between mediums I and III as the p-value was 0.773.

CONCLUSION

This study has reported the removal efficiency of NH_3-N in POME using three different soil mediums. Based on the results obtained from the removal efficiency, first order kinetic study and ANOVA test, medium II, which contains high amount of clay, was concluded to produce the highest removal efficiency of NH_3-N and the mechanism of NH_3-N removal was through absorption because clay generally has high porosity to hold water. The average removal efficiency of NH_3-N using medium II was 77% compared to sandy loam (medium I and III) with 61 and 58%, respectively.

The k value of medium II in the first order kinetic model for NH_3-N removal was 0.0897 day^{-1}. From the result of first order kinetic model, we can conclude that the removal efficiency of NH_3-N increases with a longer retention time. The results obtained from this study have demonstrated the feasibility of using soil for the removal of NH_3-N from POME. This can be further extended to the soil planted with oil palms.

ACKNOWLEDGMENTS

The authors would like to thank to Mr. Krishan Singh, Mill Manager of Bau Palm Oil Mill Sdn Bhd for his long term supports and cooperation. The authors also would like to thank to financial support from Research Acculturation Collaborative Grant Scheme (RACE) [RACE/b(01)/1245/2015(01)] given by Malaysian Ministry of Education.

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