

Palm oil mill effluent digestion in an up-flow anaerobic sludge fixed film bioreactor

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ABSTRACT: The effect of organic loading rate (OLR) provided by hydraulic retention time (HRT) and influent chemical oxygen demand (COD_{in}) on the performance of an up-flow anaerobic sludge fixed film (UASFF) bioreactor treating palm oil mill effluent (POME) was studied. Anaerobic digestion of POME was modeled and analyzed with two variables i.e. HRT and COD_{in} . Experiments were conducted based on a general factorial design and analyzed using response surface methodology (RSM). The region of exploration for digestion of POME was taken as the area enclosed by HRT (1 to 6 days) and COD_{in} (5260 to 34725 mg/L) boundaries. A simultaneous increase of the variables determined a decrease of COD removal efficiency, SRT and SRF and an increase of COD removal rate, VFA/Alk., CO_2 fraction in biogas, methane production rate. The best COD removal rate for POME treatment in an anaerobic hybrid reactor has obtained at an OLR of 17.6 g COD/l.d while it was at 26.21 g COD/l.d (Corresponds to COD_{in} of 26210 mg COD/l and HRT of 1 day) in the present study. Minimum and maximum SRT values obtained were 16 and 1904 days at OLR of 34.73 and 0.88 g COD/l.d, respectively. The present study provides valuable information about interrelations of quality and process parameters at different values of the operating variables.

Key words: Anaerobic digestion, UASFF reactor, POME, HRT, COD_{in} , RSM

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INTRODUCTION

Palm oil mill effluent (POME) is a mixture of wastewaters generated from different stages of production process, sterilizer condensates, hydro-cyclone waste, sludge separator and plant washing. It is hot (80-90 °C), acidic (pH 3.8-4.5) and contains very high concentration of organic matter ($COD=40000-50000$ mg/L, $BOD=20000-25000$ mg/L; Najafpour, *et al.*, 2006). The palm mills in Malaysia faced the challenge of balancing environmental protection, their economic viability, and sustainable development after the department of environment enforced the regulation for the discharge of effluent from the CPO industry, under the Environmental Quality (prescribed premises) order and regulations 1997. There is an urgent need to find a way to preserve the environment

which maintaining the economy in good conditions (Department of Environment Malaysia, 1999).

Biological treatment processes, in an effort to minimize cost, utilize microbial communities of varying degrees of diversity that interact in a multitude of ways to mediate a myriad of biological reactions, (Wise, 1987). Anaerobic digestion has been widely accepted as an interesting alternative for wastewater treatment and simultaneous fuel gas production. Its successful and economic employment arises from the development of new reactor designs (Jans and Man, 1988). It presents a number of significant advantages when compared with conventional aerobic wastewater systems (Droste, 1997; Metcalf & Eddy, 2003).

Due to the inherently lower efficiencies and loading rates of anaerobic filter and the occasional instability of the UASB, a hybrid of these two systems are being proposed an unpacked lower section and a packed upper section (Speece, 1988). Employing two technological approaches in up-flow anaerobic sludge fixed film (UASFF) reactor, microbial granules development and biofilm attachment on an inert media, allow independency of sludge retention of the hydraulic retention time (HRT), (Najafpour, *et al.*, 2006).

Owing to high anaerobic digestibility of POME, a wide range of anaerobic approaches have been examined. Among them, UASFF as a hybrid reactor has been the most efficient process due to its high ability to retain biomass even under overload (Najafpour, *et al.*, 2006). Effects of organic loading rate on the performance of the different biological reactors have been investigated for POME treatment (Ma and Ong, 1988; Borja and Banks, 1994a; Borja and Banks, 1994b; Borja, *et al.*, 1996; Fakhurul-Razi and Noor, 1999; Faisal and Unno, 2001; Norulaini *et al.*, 2001; Najafpor, *et al.*, 2005; Najafpour, *et al.*, 2006; Zinatizadeh, *et al.*, 2006). The studies have been carried out with the aim of process investigations in different anaerobic systems for POME treatment and changes in effluent quality parameters were discussed in a certain operating condition.

Response surface methodology (RSM) is a collection of mathematical and statistical techniques useful for analyzing the effects of several independent variables on the response (Box and Draper, 1987). RSM has an important application in the process design and optimization as well as the improvement of existing design. This methodology is more practical compared to the approaches mentioned above as it arises from experimental methodology which include interactive effects among the variables and, eventually, it depicts the overall effects of the parameters on the process (Baş and Boyaci, 2006).

In the present study, in addition of process analysis, a general factorial design was employed to describe and model variation trend in eight important responses (TCOD removal and removal rate, Volatile fatty acids to alkalinity ratio, CO₂ fraction in biogas, solid retention time (SRT), sludge retention factor (SRF), methane production rate per volume of the reactor and feed) as

function of two independent variables, HRT and influent COD concentration.

MATERIALS & METHODS

A laboratory-scale, UASFF reactor was used in this study (Najafpour *et al.*, 2006). The glass bioreactor column was fabricated with an internal diameter of 6.5 cm and a liquid height of 112 cm. Total volume of the reactor was 4980 mL (including the section containing the gas-solid separator), and the working volume (total liquid volume excluding volume of the pall rings in fixed bed section) was 3650 mL. The column consisted of three sections; bottom, middle and top. The bottom part of the column, with a height of 80 cm was operated as a UASB reactor, the middle part of the column with height of 25 cm was operated as a fixed film reactor and the top part of the bioreactor served as a gas-solid separator. The middle section of the column was packed with 90 Pall rings with diameter and height each equal to 16 mm. The void age of the packed-bed reactor was 91.25 % and the specific surface area of the packing material was 341 m²/m³. An inverted funnel shaped gas separator was used to conduct the biogas to a gas collection tank. The UASFF reactor was operated under meso-philic conditions (38 ± 1 °C) and temperature was maintained by circulating hot water through the bioreactor jacket. In order to distribute the feed uniformly in the reactor, an influent liquid distributor was mounted at the base of the column. The inoculum for seeding was an equal proportion mixture of sludge taken from a drainage channel bed of Perai Industrial Zone (Butterworth, Malaysia), digested sludge from a food cannery factory and animal manure. Details regarding the start up procedure can be found elsewhere (Najafpour, *et al.*, 2006).

The bioreactor was fed with POME pre-settled for 1 h. The characteristics of POME are summarized in Table 1.

Table 1. Characteristics of raw POME

Parameter	Amount
BOD ₅ (mg/L)	23000-26000
COD (mg/L)	42500-55700
Soluble COD (mg/L)	22000-24000
TVFAs (mg acetic acid/l)	2500-2700
SS (mg/L)	16500-19500
Oil and grease (mg/L)	4900-5700
Total N (mg/L)	500-700
pH	3.8-4.4

In order to describe the interactive effects of HRT and COD_m on the responses, 36 continuous experiments were conducted as HRT varied from 1 to 6 days at 6 levels (1, 1.5, 3, 4, 5 and 6), and as influent COD concentration varied from 5260 to 34725 mg/L at 6 levels (5260, 10575, 14485, 21310, 26210, 34725 mg/L). HRT and influent COD concentration were chosen as two independent factors in the experiment design. TCOD removal and TCOD removal rate, Volatile fatty acids to alkalinity ratio, CO_2 fraction in biogas, solid retention time (SRT), sludge retention factor (SRF), methane production rate per volume of the reactor and feed were dependent output responses.

The experiment design is shown in Table 2. Regression analysis was performed to estimate the response function as below:

$$Y = \beta_0 + \beta_i X_i + \beta_j X_j + \beta_{ii} X_i^2 + \beta_{jj} X_j^2 + \beta_{ij} X_i X_j + \dots \quad (1)$$

Where, i and j are the linear and quadratic coefficients, respectively, β is the regression coefficient. Model terms were selected or rejected based on the P value with 95% confidence level. The results were completely analyzed using analysis of variance (ANOVA) by Design Expert software. Three dimensional plots and their respective contour plots were obtained based on the effect of the levels of the two factors. From these three-dimensional plots, the simultaneous interaction of the two factors on the responses was studied.

The following parameters were analyzed according to standard methods, (APHA, 1999): pH, alkalinity, TSS, VSS, BOD and COD. Total Kjeldahl nitrogen (TKN) was determined by colorimetric method using a DR 2000 spectrophotometer (Hach Co. Loveland, Co). Gas chromatographs equipped with thermal conductivity detector (TCD) and flame ionization detector (FID) were used for the determination of biogas and volatile fatty acid compositions, respectively, (Najafpour et al., 2006). The dry weight of the attached biofilm per unit wetted surface area of pall rings (X) was determined by drying a Pall ring at 80 °C for 24 h before and after biofilm attachment.

RESULTS & DISCUSSION

After a short and successful start up period, the reactor was operated at different HRTs and

influent COD concentrations. In this study, the up-flow velocity was maintained relatively constant at 0.44 ± 0.02 m/h while the influent COD concentration was increased from 5260 to 34725 mg/L (at six levels). The HRT was stepwise decreased from 6 to 1 day for each influent COD concentration, So that, the UASFF bioreactor was subjected to 36 different conditions. High performance of the UASFF reactor at steady state conditions with HRT and influent COD concentrations in the range under studied was successfully demonstrated. In order to analyze and model the interactive effects of the two variables (COD_m and HRT) on the responses, Design-Expert software (Version 6) was used. In this program, general factorial design was selected. It allows the user to have factors that each has a different number of levels. The responses from the resulting 36 runs are shown in Table 2.

As various responses were investigated in this study, different degree polynomial models were used for data fitting. In order to quantify the curvature effects, the data from the experimental results were fitted to higher degree polynomial equations i.e. two factor interaction (2FI), quadratic and so on. In the Design Expert software, the response data were analyzed by default. Some raw data might not be fitted and transformations which apply a mathematical function to all the response data might be needed to meet the assumptions that made the ANOVA valid. Data transformations were needed for the TVFA and SRT responses as errors (residuals) were a function of the magnitude of the response (predicted values). Therefore, \log_{10} function was applied for these responses, (Draper and Smith, 1998; Chapra and Canale, 2003; Ahmad, et al., 2005). The model terms in the equations are after elimination of insignificant variables and their interactions. The interaction term, i.e. AB, was significant for all equations except the one defining TCOD removal rate. Based on the statistical analysis, the models were highly significant with very low probability values (from 0.0759 to < 0.0001). It was shown that the model terms of independent variables were significant at the 95 % confidence level. The square of correlation coefficient for each response was computed as the coefficient of determination (R^2). It showed high significant regression at 95 % confidence level. Adequate precision is a measure of the range in predicted response relative to its associated error or, in other words, a signal to noise ratio. Its desired

Table 2. Experimental conditions and results of general factor design

Run	Variable		Response							
	Factor 1 A:HRT	Factor 2 A:COD _{in}	TCOD removal	TCOD removal rate	VFA/Alk. (eq. acetic acid/eq. CaCO ₃)	CO ₂ fraction in biogas	SRT	Retention factor	Methane production rate	Methane production rate
	(d)	(mg/L)	(%)	(g/l.d)		(%)	(day)		(l CH ₄ / lreactor.d)	(l CH ₄ / lfeed.d)
1	1	5260	90.97	4.8	0.023	20.8	119	119	1.7	1.7
2	1.5	5260	93.16	3.3	0.014	17.7	204	136	1.1	1.7
3	3	5260	95.63	1.7	0.01	16.0	714	238	0.5	1.6
4	4	5260	96.77	1.3	0.011	15.4	1269	317	0.4	1.5
5	5	5260	96.96	1.0	0.008	14.9	1587	317	0.3	1.5
6	6	5260	97.24	0.9	0.008	14.7	1904	317	0.3	1.6
7	1	10575	89.60	9.5	0.025	26.5	39	39	3.2	3.2
8	1.5	10575	94.91	6.7	0.015	24.1	101	67	2.2	3.3
9	3	10575	96.35	3.4	0.01	17.9	363	121	1.1	3.4
10	4	10575	97.07	2.6	0.01	17.0	807	202	0.9	3.5
11	5	10575	97.64	2.1	0.009	16.1	1210	242	0.7	3.3
12	6	10575	97.68	1.7	0.007	15.9	1452	242	0.6	3.3
13	1	14485	87.40	12.7	0.028	30.2	26.5	27	4.3	4.3
14	1.5	14485	92.23	8.9	0.018	27.4	88	59	3.1	4.6
15	3	14485	97.10	4.7	0.005	19.9	363	121	1.6	4.8
16	4	14485	97.86	3.5	0.007	18.5	797	199	1.2	4.9
17	5	14485	98.00	2.8	0.007	15.1	1412	282	0.9	4.7
18	6	14485	98.14	2.4	0.007	17.9	1694	282	0.8	4.8
19	1	21310	86.72	18.5	0.044	35.9	22.7	23	5.6	5.6
20	1.5	21310	91.04	12.9	0.026	31.0	65	43	4.5	6.7
21	3	21310	97.51	6.9	0.006	22.2	231	77	2.3	6.9
22	4	21310	98.15	5.2	0.007	20.2	544	136	1.7	6.9
23	5	21310	98.33	4.2	0.006	19.0	1179	236	1.4	6.9
24	6	21310	98.40	3.5	0.006	17.7	1516	253	1.2	6.9
25	1	26210	84.43	22.1	0.103	42.1	19	19	6.6	6.6
26	1.5	26210	90.18	15.8	0.032	35.8	39.5	26	5.4	8.1
27	3	26210	97.81	8.5	0.007	25.8	237	79	2.9	8.8
28	4	26210	98.19	6.4	0.008	22.2	383	96	2.2	8.8
29	5	26210	98.44	5.2	0.008	20.4	909	182	1.7	8.6
30	6	26210	98.59	4.3	0.007	20.0	991	165	1.5	9.1
31	1	34725	80.56	28.0	0.288	48.7	16	16	8.0	8.0
32	1.5	34725	89.58	20.7	0.045	38.0	38.7	26	7.2	10.8
33	3	34725	97.90	11.3	0.008	28.1	217	72	4.0	11.9
34	4	34725	98.39	8.5	0.007	25.1	334	84	2.9	11.5
35	5	34725	98.57	6.8	0.007	22.8	672	134	2.3	11.5
36	6	34725	98.62	5.7	0.006	23.2	806	134	2.1	12.3

It was shown that the model terms of independent variables were significant at the 95% confidence level. The square of correlation coefficient for each response was computed as the coefficient of determination (R²). It showed high significant regression at 95 % confidence level. Adequate precision is a measure of the range in predicted response relative to its associated error or, in other words, a signal to noise ratio. Its desired value is 4 or more, (Mason *et al.*, 2003). The value was found desirable for all models. Simultaneously, low values of the coefficient of variation (1.54-9.55 %) indicated good precision and reliability of the experiments as suggested by Khuri and Cornell, (1996); Kuehl, (2000) and Ahmad, *et al.*, (2005).

Two reduced quadratic models were selected to describe the response surface of TCOD removal and TCOD removal rate within the range of the factors. The regression equations (built with codified factors) are as follow:

$$TCOD\ removal = 94.72 + 9.04A - 1.61B - 4.94A^2 + 2.57AB \quad (2)$$

$$\log_{10}(TCOD\ removal\ rate) = 0.92 - 0.45A + 0.39B + 0.13A^2 - 0.18B^2 \quad (3)$$

Figure 1 shows simultaneous effects of the two factors on TCOD removal efficiency obtained from Eq. (2). It showed a significant decreasing trend in TCOD removal efficiency with the decrease in HRT at a constant influent COD. Whereas slight decrease in TCOD removal

efficiency was observed when influent COD was increased under constant HRT and lower than 4 days. The lowest efficiency in TCOD removal was predicted to be 84.4 % at the highest OLR (corresponds to HRT of 1 day and COD_{in} of 34725 mg/L) while the experimental value has been 80.6 %. In this condition, the reactor instability was temporarily observed after 4 days at OLR of 34.7 g COD/L.d. But the buffer supplied by adding NaHCO₃ in the feed and recycling effluent alkalinity into influent prevented pH changes a lot and pH was controlled at 6.92. The main reason for relatively poor efficiency at OLR of 34.7 g

obtained at an OLR of 17.6 g COD/L.d (Borja, *et al.*, 1996) while it was at 26.21 g COD/L.d (Corresponds to COD_{in} of 26210 mg COD/L and HRT of 1 day) in the present study. It might be attributed to larger amount of biomass in the form of granule due to bigger volume of UASB portion relative to total volume in a hybrid reactor.

The ratio of VFA/Alk. is an important indicator of the acid-base equilibrium and process stability (Sanchez, *et al.*, 2005; Fannin, 1987). When this ratio is less than 0.3-0.4, the process is considered to be operating favorably without the risk of acidification. The measurement of quantity and composition of the biogas produced, in terms of methane and carbon dioxide content, is of fundamental importance to evaluate the stability of the process (Stafford *et al.*, 1980). When the process is stable the amount and composition of biogas are stable too. A decrease in biogas production contemporary to an increase in CO₂ content can indicate an inhibition of the methanogenesis of the system. In fact, VFA/Alk. ratio and biogas composition are strictly linked one to each other.

The following two regression equations were obtained for the variation of VFA to alkalinity ratio (p) and CO₂ fraction in biogas.

$$\log_{10}(VFA/Alk.) = -1.95 - 0.73A + 0.077B + 0.42A^2 + 0.11B^2 - 0.52AB + 0.33A^2B \quad (4)$$

$$CO_2 \text{ fraction in biogas} = 24.96 - 12.25A + 8.94B + 5.54A^2 - 4.68AB \quad (5)$$

The ratio of maximum to minimum for p was 55. Hence a logarithmic function with base 10 was required to fit the data. Fig. 3 illustrates the effects of the variables on VFA/Alk.. From the Fig. 3, the maximum value of the ratio is predicted to be 0.18 at highest OLR (corresponds to HRT of 1 and COD_{in} of 34725 mg/L) whereas the actual value was 0.28 at this condition. The VFA/Alk. ratio remained lower than the suggested value throughout experiments.

Figure 4 shows the variation of the CO₂ fraction in the biogas as a function of the two factors studied. The CO₂ fraction increased with simultaneous decrease and increase in HRT and COD_{in}, respectively. The maximum CO₂ fraction in biogas was modeled to be 46.05 % whereas the actual is 48.7 %.

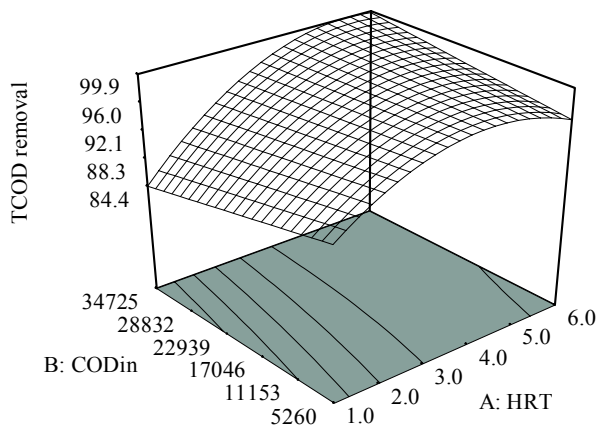


Fig. 1. Response surface plot for TCOD removal

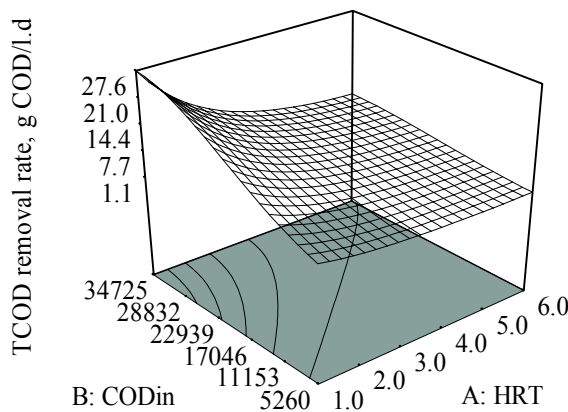


Fig. 2. Response surface plot for TCOD removal rate

The regression was conducted after transformation of raw data to a function of log base 10. Fig. 2 illustrates the effect of the factors on the TCOD removal rate in original scale. As can be seen, the TCOD removal rate increased with the increase in influent COD and decrease in HRT. The best COD removal rate for POME treatment in an anaerobic hybrid reactor has

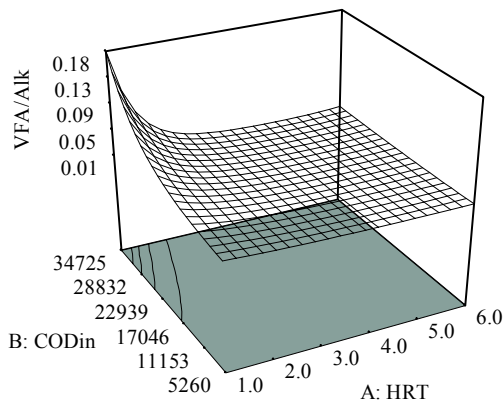


Fig. 3. Response surface plot for VFA/Alk

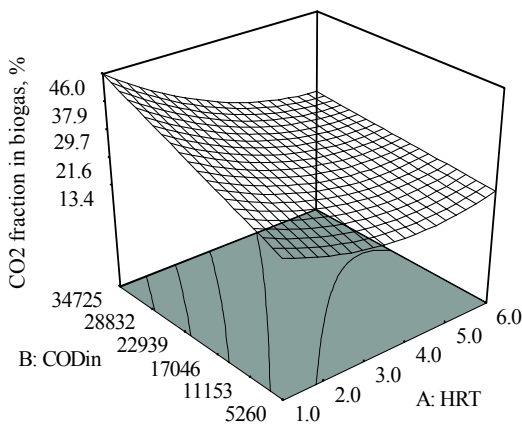


Fig. 4. Response surface plot for CO₂ percentage in biogas

It was also reflected by an increase in the effluent VFA concentration (from 158 to 843.2 mg acetic acid/l) and decrease in the effluent pH (from 7.63 to 6.92). This resulted from partial upsetting of the balance between acid formation and methane production in the anaerobic process due to high organic load at low HRT. The effluent pH remained within the optimal working range for anaerobic digestion (6.9 -7.9) throughout the experiment. SRT, as a process control parameter, was also determined by measuring VSS in the reactor and in the effluent at various concentrations of influent COD. The high SRT values denote effective role of the packed bed portion on process stability due to biomass retention which allows the system to cope with changes in OLR. Minimum and maximum SRT values obtained were 16 and 1904 days at OLR of 34.73 and 0.88 g COD/l.d, respectively. It was found that at the shortest HRT (1 day), the sludge age to HRT ratio was 16 which is in the range of safety factor (3-20) for the minimum SRT for successful

operation of anaerobic biological reactors (Lawrence and McCarty, 1969).

HRT as an operating factor affecting SRT by following relationship:

$$SRT = \frac{X_r \cdot HRT}{X_e} \tag{6}$$

Where X_r is the concentration of sludge in the reactor (g VSS/l); X_e is the concentration of VSS in the effluent of the reactor (g VSS/l). The SRT to HRT ratio is defined the sludge retention factor (SRF). The increase in retention factor involves increasing in HRT. Large values of retention factor providing longer SRT which favoring methanogenesis and improving process performance. The best option to achieve high SRT while maintaining HRT at low levels is biomass immobilization which is applied in the UASFF reactor in the form of granular sludge and biofilm attached on the packing.

In order to model interactive effects of the variables (HRT and COD_m) on the process control responses, SRT and SRF, two following quadratic models were obtained.

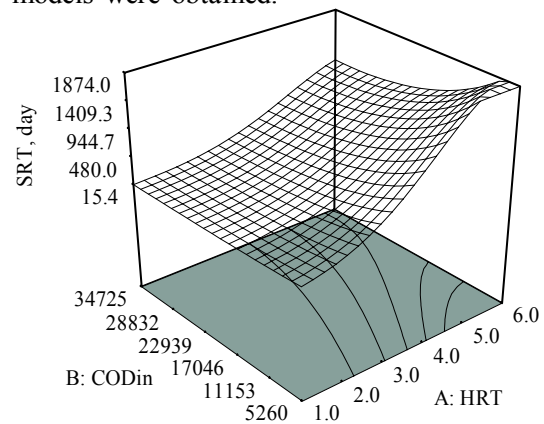


Fig. 5. Response surface plot for SRT

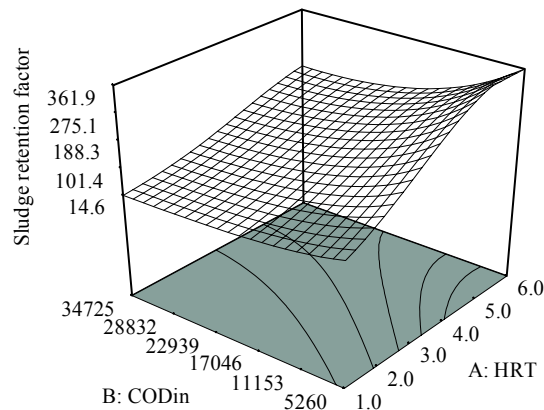


Fig. 6. Response surface plot for sludge retention time

$$\text{Log}_{10}(\text{SRT}) = 2.24 + 1.09A - 0.31B - 0.35A^2 + 0.098B^2 + 0.1AB \quad (7)$$

$$\text{Log}_{10}(\text{SRF}) = 1.85 + 0.59A - 0.31B - 0.2A^2 + 0.098B^2 + 0.1AB \quad (8)$$

Since maximum to minimum ratio for SRT and SRF were 119 and 19.8, respectively, a logarithmic function with base 10 was applied to fit the data. The same variation trend for SRT and SRF was observed as shown in Fig. 5 and 6, indicating inverse proportion between OLR and the responses. In this study, the up-flow velocity was maintained constant (0.44 m/h) (controlled by recycle ratio), therefore, increase in the concentration of VSS in the effluent was attributed to an increase in gas production rate due to increase in HRT and COD_{in}.

The methane production is a function of OLR (changing HRT or/and COD_{in}). The two following regression equations were selected to describe changes in methane production rate as function of the variables.

$$\text{Methane production rate per unit of reactor volume} = 2.9 - 2.88A + 2.29B + 1.19A^2 - 1.24AB \quad (9)$$

$$\text{Methane production rate per unit of feed} = 6.31 + 0.96A + 4.39B - 0.52A^2 + 0.8AB \quad (10)$$

CONCLUSIONS

The UASFF bioreactor was a successful biological treatment process to achieve a high COD removal efficiency in a short period of time. The response surface methodology results demonstrated the effects of the operating variables as well as their interactive effects on the responses. A simultaneous increase of the variables determined a decrease of COD removal efficiency, SRT and SRF and an increase of COD removal rate, VFA/Alk., CO₂ fraction in biogas, methane production rate. The best COD removal rate for POME treatment in an anaerobic hybrid reactor has obtained at an OLR of 17.6 g COD/l.d while it was at 26.21 g COD/l.d (Corresponds to COD_{in} of 26210 mg COD/l and HRT of 1 day) in the present study. Minimum and maximum SRT values obtained were 16 and 1904 days at OLR of 34.73 and 0.88 g COD/l.d, respectively. Experimental findings were in close agreement with the model prediction.

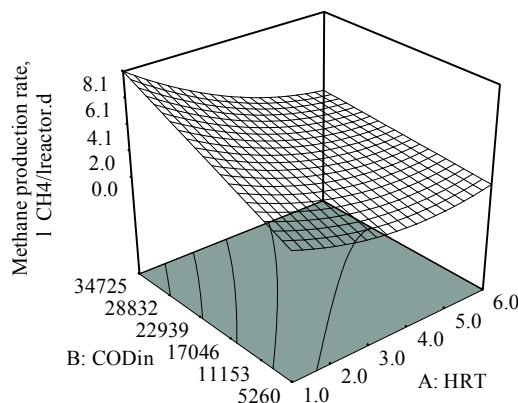


Fig. 7. Response surface plot for methane production rate per unit of reactor volume

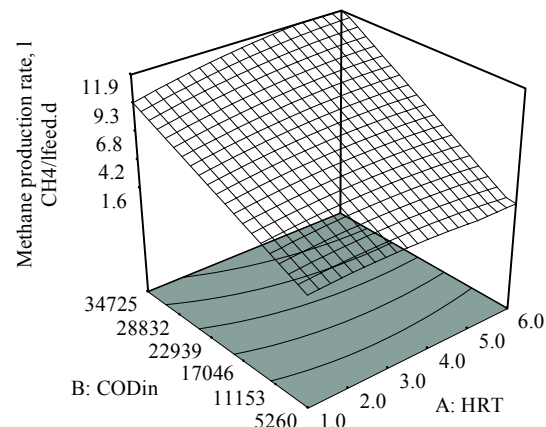


Fig. 8. Response surface plot for methane production rate per unit of feed

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