PRETREATED PALM OIL MILL EFFLUENT (POME) DIGESTION IN AN UP-FLOW ANAEROBIC SLUDGE FIXED FILM BIOREACTOR: A COMPARATIVE STUDY

A. A. L. Zinatizadeh

Water and Environment Division, Water and Power Industry Institute for Applied and Scientific Higher Education Kermanshah, Iran, aliazinatiz@yahoo.com

A. R. Mohamed, M. D. Mashitah and A. Z. Abdullah

School of Chemical Engineering, Engineering Campus, Universiti Sains Malaysia Malaysia, chrahman@eng.usm.my – chzuhairi@eng.usm.my – chmashitah@eng.usm.my

G. D. Najafpour*

Department of Chemical Engineering, Engineering Faculty, University of Mazandaran Babol, Iran, najafpour@nit.ac.ir - najafpour8@yahoo.com

*Corresponding Author

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Abstract An up-flow anaerobic sludge fixed film (UASFF) bioreactor was used to treat the pretreated palm oil mill effluent (POME). In physical pretreatment, POME was pre-settled for 2 h and the supernatant was fed into the reactor. In chemical pretreatment, optimum dosages of cationic and anionic polymers were used. Experiments of pretreated POME digestion were conducted based on a central composite face-centered design (CCFD) with two independent operating variables, feed flow rate (Q_F) and up-flow velocity (V_{up}). The operating variables were varied to cover a wide range of organic loading rate (OLR) from 3.8 to 29 g COD /l.d. A stable TCOD removal efficiency of 83.5 % was achieved at the highest Q_F (3.31 l/d, corresponding to OLR of 26 g COD/l.d) for pre-settled POME whereas only 62.2 % TCOD removal was achieved with chemically pretreated POME at Q_F of 7.63 l/d (corresponding to OLR of 29 g COD/l.d) and that too was coupled with process instability. At comparable OLRs i.e. 16.95 g COD/l.d (Q_F = 2.16 l/d) for pre-settled POME and 16.42 g COD/l.d (Q_F = 4.32 l/d) for chemically pretreated POME, the VFA concentrations for the two cases were also similar.

Key Words Pome, Pretreatment, UASFF Reactor, Central Composite Face-Centered Design (CCFD)

چکیده راکتور تلفیقی از بستر لجن بی‌هوازی و بستر آکنده با جریان رو به‌بالا به منظور تصفیه ثانویه فاضلاب پیش‌تصفیه‌شده کارخانه روغن نخل(POME) آزمایش شد. در پیش تصفیه فیزیکی پس از دو ساعت ته‌نشینی مایع رویی برای تصفیه بیولوژیکی به عنوان خوراک به UASFF پمپ شد. در پیش تصفیه شیمیایی، مقادیر بهینه پلیمرهای کاتیونی و آنیونی استفاده شدند. آزمایشهای هضم بی‌هوازی فاضلاب پیش‌تصفیه‌شده براساس برنامه آماری مختلط مرکزی با دو متغیر عملیاتی غیروابسته مشتمل بر شدت جریان ورودی (Q_F) و سرعت جریان رو به بالا (V_{up}) اجرا شد. تغییرات فاکتورهای عملیاتی به گونه‌ای طراحی شد که محدوده گسترده‌ای از شدت بار آلی (OLR) را از مقدار ۳/۸ تا ۲۹ گرم COD/l.d بپوشاند. مقدار ۸۳/۵٪ بازدهی حذف TCOD در بالاترین مقدار Q_F (۳/۳۱ l/d، معادل با مقدار ۲٦ گرم COD/l.d از OLR) برای فاضلاب پیش‌ته‌نشین شده بدست آمد. این در حالی است که تنها ٦۲/۲٪ حذف TCOD همراه با ناپایداری فرآیند برای فاضلاب پیش تصفیه شده شیمیائی درQ_F ۷/٦۳ l/d (معادل با مقدار ۲۹ گرمCOD/l.d از OLR) حاصل شد. در شدت های قابل مقایسه از بار آلی (به ترتیب مقادیر ۱٦/۹۵ و ۱٦/٤۲ گرم COD/l.d برای فاضلاب پیش تصفیه شده فیزیکی و شیمیایی)، غلظت های تقریباً مشابهی از VFA در خروجی راکتور برای هر دو مورد بدست آمد. در این شرایط، مقادیر Q_F و V_{up} به ترتیب ۱/۰۱ l/d و ۰/۲ m/h بودند.

1. INTRODUCTION

Palm oil is one of main agricultural products in Malaysia as it contributes 49.5 % of the total world production [1]. On an average standard palm oil mills, for each tonne of fresh fruit bunch (FFB)

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processed, generates about 1 tonne of liquid waste with a pollution load of biochemical oxygen demand of (BOD) 37.5 kg, a chemical oxygen demand the (COD) 75 kg, suspended solids (SS) 27 kg and oil and grease 8 kg [2]. There are currently about 265 active palm oil mills in Malaysia with a combined annual CPO production capacity of about 13 million tonnes [3]. This amounts to a population equivalent of around 80 million in terms of COD. Thus, there is an urgent need to find an efficient and practical approach to preserve the environment while maintaining the economy.

Considering the highly organic character of palm oil mill effluents (POME), the anaerobic process is the most suitable approach for treatment [4]. The common practice of treating POME is by using ponding and/or open digestion tank systems which have particular disadvantages such as: long hydraulic retention times of 45-60 days [5], bad odour, difficulty in maintaining the liquor distribution to ensure smooth performance over huge areas and difficulty in collecting biogas which could have detrimental effects on the environment [6-7].

High-rate anaerobic reactors, that can retain biomass, have a high treatment capacity and hence low site area requirement [8]. POME COD removal efficiencies in excess of 85% have been reported for high rate reactors such as the anaerobic baffled reactor (ABR) [9], the single up-flow anaerobic sludge blanket (UASB) reactor [10], two stage UASB system [11] and membrane anaerobic system (MAS) [12]. In summary, the high rate anaerobic reactors mentioned above are successfully able to treat POME at short HRT.

The UASB reactor exhibits positive features, such as high organic loadings, short hydraulic retention time (HRT) and a low energy demand, especially for POME treatment [10,13]. Suspended and colloidal components of POME in the form of fat, protein, and cellulose have an adverse impact on UASB reactor performance and can cause deterioration of microbial activities and wash out of the active biomass [10,14]. The use of internal packing as an alternative for retaining biomass in the UASB reactor is a suitable solution for the mentioned problems [2-15]. Process instability was observed when a UASB reactor (at HRT of 4 d) and an anaerobic hybrid reactor (AHR) (at HRT of 3.5 d) were operated with high influent COD concentrations of 42500 and 65000 mg/l, respectively [10-15]. Consequently, complete digestion of raw POME without pretreatment demands high HRT, which is not easily achieved due to the high volume of POME produced by the mills. Various pretreatment approaches have been examined for the separation of suspended solids, oil and grease from POME. These include: Chemical coagulation and flocculation [16-18], air flotation simple and skimming [19-20], ultra-filtration [21-22], evaporation [23], centrifugation [20].

The present research is a comparative study of anaerobic digestion of POME which has been physically (primary sedimentation unit) and chemically (chemical coagulation and flocculation pretreated). Results obtained from the high rate digestion of pretreated POME were compared using the response surface methodology (RSM) with respect to the simultaneous effects of two independent operating variables, feed flow rate (Q_F) and up-flow velocity (V_{up}).

2. MATERIAL AND METHODS

2.1. Wastewater Preparation Raw POME was collected from a local palm oil mill in Nibong Tebal, Penang, Malaysia. In the first stage, raw POME was pre-settled using an ordinary sedimentation tank. In the second part of this study, raw POME was chemically pretreated to remove suspended solids and residual oil (using a cationic and anionic polymers). The samples were then stored in a cold room at 4 ^oC. PMOE stored under such conditions has no observable effects on its composition. The characteristics of the raw and pretreated POME are summarized in Table 1.

2.2. Bioreactor and Start Up A laboratory-scale, up-flow anaerobic sludge fixed film (UASFF) reactor was used in this study. 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, and the working volume was 3650 ml. The column consisted of three sections; bottom, middle and top. The bottom part of the column, with a height

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TABLE 1. Characteristics of the Raw, Pre-Settled and Pretreated POME[a].

Parameter	Raw POME	Pre-settled POME	Chemically Pretreated POME
BOD_5 (mg/l)	22700	20100	9750
COD (mg/l)	44300	28640	13880
Soluble COD (mg/l)	17140	17140	13880
TVFA (mg acetic acid/l)	2510	2510	2760
SS (mg/l)	19780	5760	< 20
Oil and grease (mg/l)	4850	1 630	Negligible
Total N (mg/l)	780	660	480
pH	4.05	4.05	4.2

[a]Values are average of three measurements. The differences between the measurements for each were less than 1%.

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of 80 cm was operated as a UASB reactor, the middle part of the column with a 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 a diameter and height equal to 16 mm. The voidage of the packed-bed reactor was 91.25 % and the specific surface area of the packing material was 341 m^2/m^3. An inverted funnel shaped gas separator was used to conduct the biogas to a gas collection tank. The UASFF reactor was operated under mesophilic conditions (38 ± 1ºC) and the 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 [2].

2.3. Bioreactor Operation and Experimental Design

The UASFF bioreactor was separately operated with pre-settled and chemically pretreated POME and experiments were designed by Design Expert software (Stat-Ease Inc., version 6.0.6) with two variables, feed flow rate and up-flow velocity. In an earlier study [2], feed flow rate (Q_F) and up-flow velocity (V_{up}) were found to be the most critically independent operating variables which affected the performance of the reactor. The region of exploration for POME treatment was decided as the area enclosed by Q_F (1.01, 3.31 l/d) and V_{up} (0.2, 3 m/h) boundaries for pre-settled POME and Q_F (1.01, 7.63 l/d) and V_{up} (0.2, 3 m/h) for chemically pretreated POME. This would cover an OLR range of 7.9 to 26.0 and 3.8 to 29.0 g COD/l.d for pre-settled and chemically pretreated POME respectively. A steady state was assumed after five turnovers.

In order to carry out a comprehensive analysis of the anaerobic process, 4 dependent parameters were either directly measured or calculated as response. These parameters were total COD (TCOD) removal, effluent total volatile fatty acid (TVFA), effluent bicarbonate alkalinity (BA) and methane yield (Y_M).

Data analysis was carried out using the response surface methodology (RSM) under general factorial design. The results were completely analyzed using analysis of variance (ANOVA) which was automatically performed by Design Expert Software (ver. 6.0.6).Three dimensional (3D) 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 RSM used in the present study was a Central Composite Face-

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centered Design (CCFD) involving two different factors, Q_F and V_{up}. The experimental conditions and results are shown in Table 2.

2.4. Analytical Methods The following parameters were analyzed according to Standard Methods [24]: pH, alkalinity, TSS, VSS, BOD and COD. Total Kjeldahl nitrogen (TKN) was determined by a colorimetric method using a DR 2000 spectrophotometer (Hach Co. Loveland, Co). Gas chromatographs equipped with a thermal conductivity detector (TCD) and a flame ionization detector (FID) were used for the determination of biogas and volatile fatty acid compositions, respectively [2].

3. RESULTS AND DISCUSSION

Raw POME contains a high concentration of suspended solids (Table 1) which requires long

TABLE 2. Experimental Conditions and Results of Central Composite Design.

		Variables		Response					
Run	Type of pretreatment	Factor1 A:Feed flow rate (l/d)	Factor2 B: Up - flow velocity (m/hr)	Total COD removal (%)	Eff. TVFA (mg acetic acid/l)	BA (mg $CaCO_3$/l)	Methane Percentage in biogas %	Methane production Rate, l CH_4/d	Methane Yield (l CH_4 /g COD_{rem}.d)
1	Pre-settled POME	1.01	0.2	97.3	132.4	2006	61.41	9.7	0.344
2		1.01	1.60	93.9	104.2	2026	84.4	7.8	0.298
3		1.01	3.00	93.1	80.54	2087	73.83	6.5	0.258
4		2.16	0.20	90.7	243.1	1957	60.33	15.4	0.295
5		2.16	1.60	91.3	35.17	2225	72.33	16.0	0.305
6		2.16	1.60	91.5	44.2	2208	69.98	14.8	0.303
7		2.16	1.60	91.4	36.1	2234	71.24	14.9	0.315
8		2.16	1.60	90.3	58.8	2219	71.8	15.4	0.305
9		2.16	1.60	92.7	38.3	2162	72.12	14.2	0.29
10		2.16	3.00	95.5	41.9	2210	63.33	13.0	0.25
11		3.31	0.2	82.7	573.75	1723	51.33	15.3	0.196
12		3.31	1.60	91.8	144.5	2157	74.08	25.7	0.292
13		3.31	3.00	94.2	137.8	2162	67.89	25.5	0.285
1	Chemically pretreated POME (coagulation and flocculation)	1.01	0.2	96.7	64.86	1394	62.58	4.5	0.335
2		1.01	1.60	94.3	23.1	1504	82.61	3.6	0.273
3		1.01	3.00	93.24	22.1	1664	70.78	2.9	0.226
4		4.32	0.20	84.3	146.1	1776	51	13.2	0.261
5		4.32	1.60	85.2	33.15	1896	61.75	14.8	0.29
6		4.32	1.60	84.5	37.49	1833	61.81	14.5	0.285
7		4.32	1.60	86	34.7	1915	63.8	14.2	0.275
8		4.32	1.60	87.6	33.99	1896	62.19	13.2	0.27
9		4.32	1.60	84.5	36.07	1854	62.07	12.9	0.255
10		4.32	3.00	86.5	37.92	1893	54.98	11.8	0.228
11		7.63	0.2	62.2	1613.2	915	30.96	6.3	0.109
12		7.63	1.60	80.2	289.68	1474	62.2	20.7	0.244
13		7.63	3.00	83.8	251.02	1312	67.7	19.9	0.224

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retention time for satisfactory digestion. A fraction of TSS which is not digestible is gradually accumulated in the reactor by attaching to it the sludge granules in the UASFF reactor it causes reduction in process efficiency. From a practical point of view and according to various studies [2,10,11,15], the oil-bearing suspended solids need to be removed (partially or completely) before anaerobic treatment in order to have a reliable, stable and efficiently high rate anaerobic process.

3.1. POME Digestion

3.1.1. TCOD Removal The effect of the variables on TCOD removal efficiencies are shown in Figure 1a and b as contour plots for pre-settled and chemically pretreated POME, respectively. Since total suspended solids (TSS) of the pre-settled POME contained 5760 mg/l, a fraction of the OLR is in suspended solids whereas in the chemically pretreated POME, the entire OLR is solubel. In an overall comparison, the trend of changes in TCOD removal efficiency was quite similar for both conditions. The TCOD removal (%) decreased with an increase in Q_F while the rate of TCOD removal (g COD/l.d) was increased (Table 2), due to an increase in the diffusion rate of substrate at higher substrate concentration [26-27].

A stable TCOD removal efficiency of 83.5 % was achieved at the highest Q_F (3.31 l/d, corresponding to OLR of 26 g COD/l.d) for pre-settled POME whereas only 62.2 % TCOD removal was achieved with chemically pretreated POME at Q_F of 7.63 l/d (corresponding to OLR of 29 g COD/l.d) and that also was coupled with process instability. It was found that at the same OLR (center points, V_{up} from o.2 to 3 m/h), despite 33% of OLR in the pre-settled POME being suspended solids, which needs to be hydrolyzed first and greater COD removal efficiency (90-94 %) was achieved compared to chemically pretreated POME the COD removal efficiency was in the range of (82-88 %). This may be attributed to possible inhibitory effects of the polymers which were applied for chemical pretreatment.

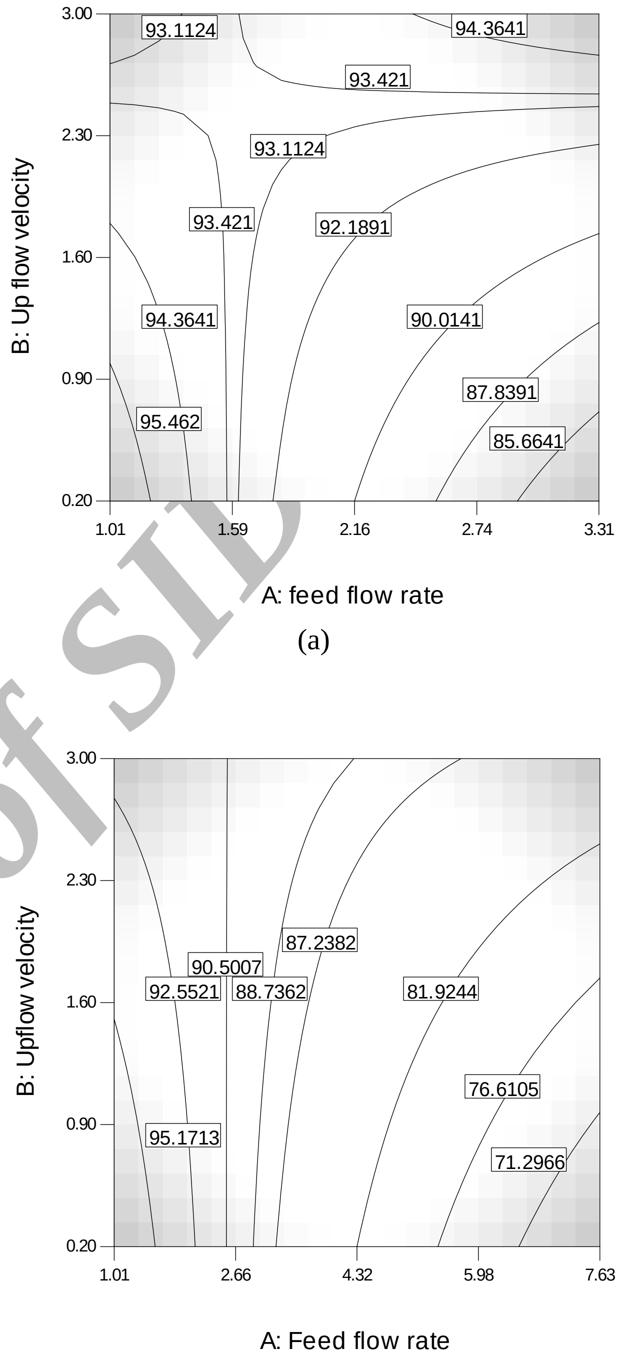

Figure 1. DESIGN-EXPERT plot. Contour plot of TCOD removal efficiency representing the effect of the feed flow rate and up-flow velocity (a) Pre-settled POME (b) Chemically pretreated POME.

3.1.2. Effluent TVFA The VFA concentration is a key indicator of system performance. Figure 2a and b depict the effects of the variables on the effluent VFA for the pre-settled and the chemically pretreated POME, respectively. The highest concentrations of VFA, as intermediates, was

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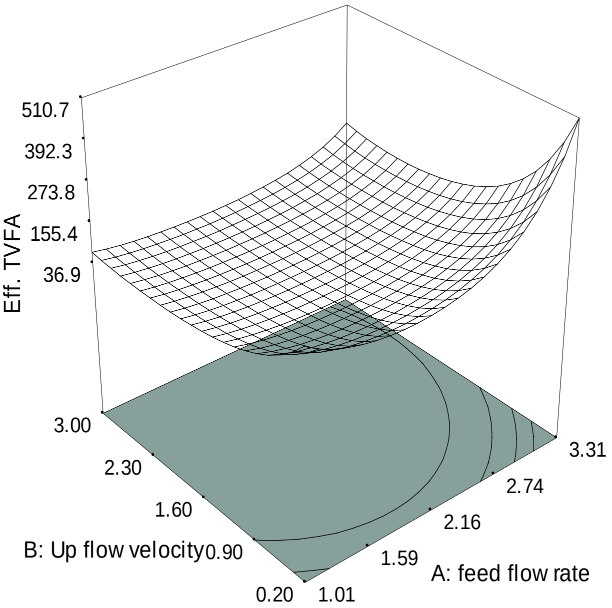
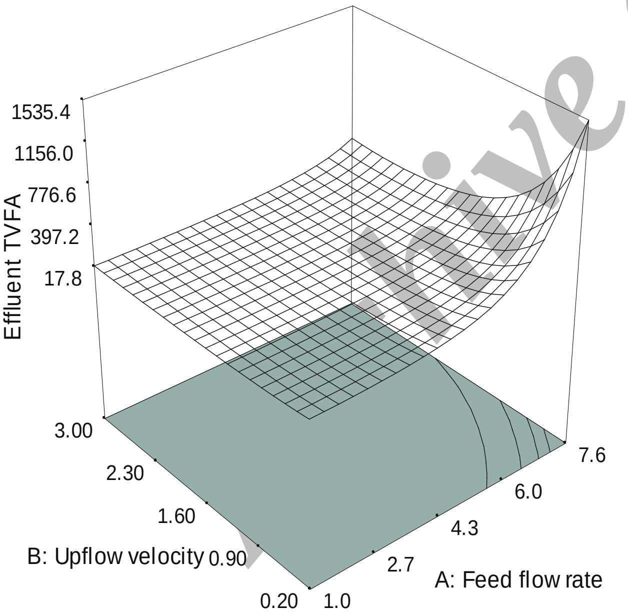


(a)



(b)

Figure 2. DESIGN - EXPERT plot. 3D graph of effluent TVFA representing the effect of the feed flow rate and up-flow velocity (a) Pre-settled POME (b) Chemically pretreated POME.

found during overloading conditions when they were 553 mg/l for the pre-settled POME at OLR of 26 g COD/l.d and 1613 mg/l for the chemically pretreated POME at OLR of 29 g COD/l.d. In both experiments, the role of V_{up} in the system recovery at a high Q_F was very significant due to its effects on recycled alkalinity and contact between substrate and biomass [28]. From the 3D graph, at comparable OLRs i.e. 16.95 g COD/l.d (Q_F = 2.16 l/d) for pre-settled POME and 16.42 g COD/l.d (Q_F = 4.32 l/d) for chemically pretreated POME, the VFA concentration for the two cases were also similar. It showed that there was a balance between acetogenesis and methanogenesis in the systems at this OLR. In the chemically pretreated POME digestion, process upset occurred as methanogenic bacteria could not metabolize the VFA as fast as they were produced; resulting in a possible reduction in pH. In the chemically pretreated POME digestion, process upset occurred when methanogenic bacteria could not metabolize the VFA as fast as they were produced; resulting in a possible reduction in pH. This condition (Q_F = 7.63 l/d, V_{up} = 0.2 m/h) had a strong influence on the biogas quality, increasing the CO_2 percentage (75.22 %). Similar behavior was observed in a secondary UASB reactor treating piggery waste at an HRT of 1 day and an influent COD of 10189 mg/l [29].

3.1.3. Effluent BA Figure 3a and b show the interactive effects of Q_F and V_{up} on bicarbonate alkalinity (BA). It was understood that the BA was produced through POME digestion reactions as no chemical was added to cneate alkalinity. Effluent BA values for pre-settled POME were greater than the values for chemically pretreated POME within the tested range of the two variables.

From Figure 3a, the maximum level for BA was predicted to be the region where Q_F and V_F were relatively high (the values larger than center point) while it was obtained from the middle part of the design space in Figure 4b. At the highest OLR and the lowest V_{up} (corresponding to Q_F of 3.31 l/d in Figure 3a and 7.63 l/d in Figure 4b both at V_{up} 0.2 m/h), BA was obtained as 1720 and 960 mg $CaCO_3$/l for the pre-settled and the chemically pretreated POME, respectively. In this condition (V_{up} = 0.2 m/h), the BA concentration was not high enough to avoid process upset for chemically

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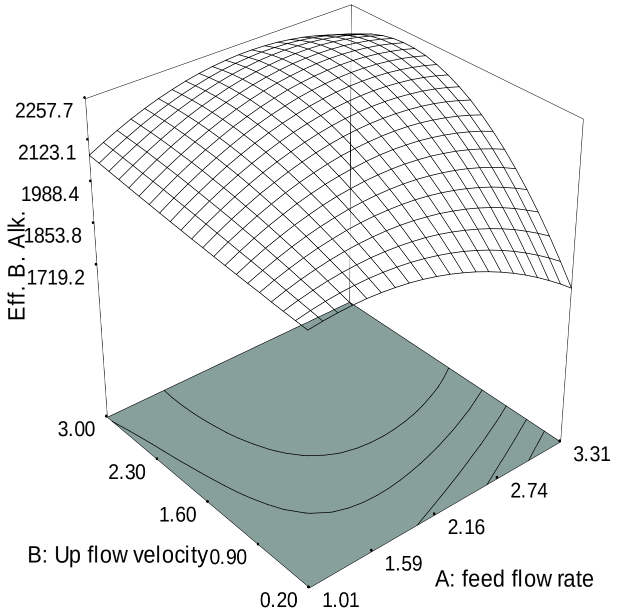
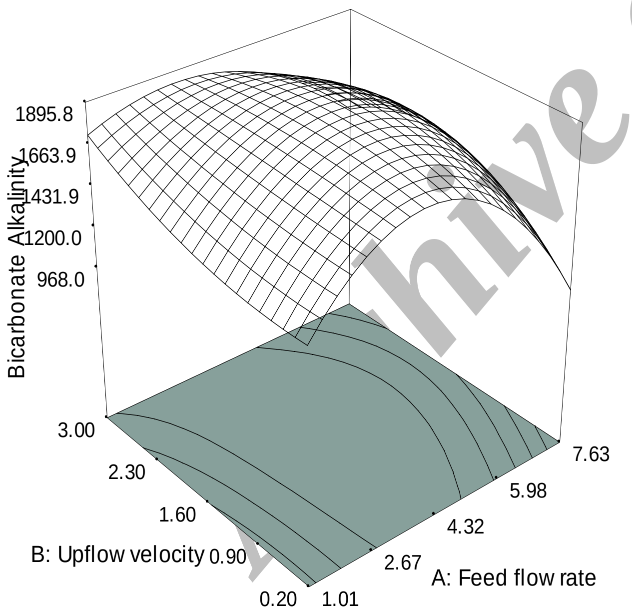


(a)



(b)

Figure 3. DESIGN - EXPERT plot. 3D graph of effluent BA representing the effect of the feed flow rate and up-flow velocity (a) Pre - settled POME (b) Chemically pretreated POME.

pretreated POME while the process remained stable for pre-settled POME. The effect of V_{up} on system recovery was more significant at a high feed flow rate due to more alkalinity recycling.

3.1.4. Methane Yield Figure 4a and b represent the simultaneous effects of the variables on the methane yield as contour plots. It shows that a simultaneous decrease in the variables yielded an increase in the response for both pre-settled and chemically pretreated POME. It was found that the yield values for the pre-settled POME (Figure 4a) were greater than the values for the chemically pretreated one. The highest level of the yield was 0.34 and 0.33 for pre-settled and chemically pretreated POME, in that some order where Q_F and V_{up} were 1.01 "l/d" and 0.2 m/h respectively. It was also found that a minimum retention time longer than 1.5 and 2.2 d, respectively, for the pre-settled and chemically pretreated POME digestion was needed to achieve high methane yield.

4. CONCLUSION

Response surface methodology was a potent tool to compare the results obtained from the anaerobic treatment of the two different types of pretreated POME with different characteristics. At an OLR of about 16.5 g COD/l.d (center point), despite 33% of OLR in the pre-settled POME being suspended solids, greater COD removal efficiency was achieved compared to chemically pretreated POME. Effluent BA values for pre-settled POME were greater than the values for chemically pretreated POME within the tested range of the two variables. The highest level of the yield was 0.34 and 0.33 for pre-settled and chemically pretreated POME, in that some order where Q_F and V_{up} were 1.01"l/d" and 0.2 m/h respectively.

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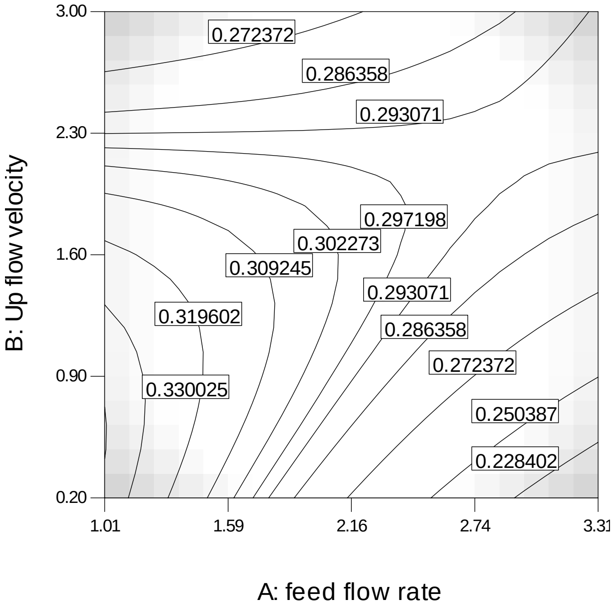
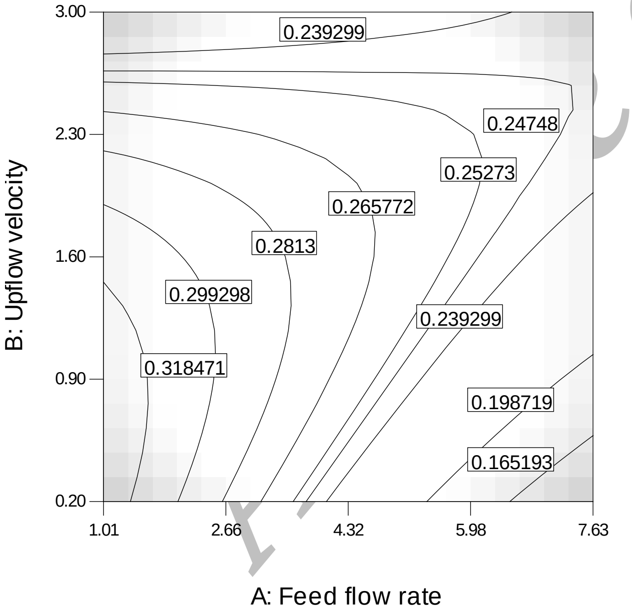


(a)



(b)

Figure 4. DESIGN-EXPERT plot. Contour plot of methane yield representing the effect of the feed flow rate and up-flow velocity (a) Pre-settled POME (b) Chemically pretreated POME.

the industry personnel for their full cooperation.

6. ABBREVIATION AND NOMENCLATURE

ABR	Anaerobic bafled rector
AHR	Anaerobic hybrid rector
ANOVA	Analysis of variance
BA	Bicarbonate alkalinity
BOD	Biochemical oxygen demand
COD	Chemical oxygen demand
CCFD	Centerl composite face-centered design
FFB	Fresh fruit bunch
FID	Flame inoiztion detector
HRT	Hydraulic retention time
MAS	Memberane anaerobic system
OLR	Organic loading rete
POME	Palm oil mill effluent
Q_F	Feed flow rete
RSM	Response surface method
SS	Supended solids
TCD	Thermal conductivity detector
TCOD	Total chemical oxygen demand
TKN	Total kjeldahl nitrogen
TSS	Total suspended solids
TVFA	Total volatile fatty asid
UASB	Up-flow anaerobic sludge blanket
UASFF	Up-flow anaerobic sludge fixed film
VFA	Volatile fatty asid
V_{up}	Up-flow velocity
Y_M	Yield of methane production

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