ORIGINAL RESEARCH ARTICLE

Influence of pH on the anaerobic fluidised bed reactor performance for palm oil mill effluent treatment

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ABSTRACT

The effect of pH on the performance of a pilot-scale anaerobic fluidised bed reactor (AnFBR) was studied using palm oil mill effluent (POME) as the substrate. The performance of the 2000-litre reactor at different operating conditions, such as organic loading rates and retention times was studied. This acidic agro-industrial wastewater (pH 4.0–5.0) was neutralised by adding slacked lime. It was observed that, within 12 hr of hydraulic retention time (HRT), the AnFBR removes as high as 85% of the substrate chemical oxygen demand (COD) at a loading rate of 4 kg/m³ day. High pre-treatment cost is needed to neutralise the bulk volume of wastewater that was generated from the palm oil industries. Thus, an attempt was made to study the performance of the AnFBR under pH shock load. The influent pH was increased to 9.2 and then dropped around 5.0 to intensify the effect of the pH shock load. At shock load, the reactor performance for COD removal dropped by about 25% lower than the optimum condition. The maximum and minimum COD removal rates during the short period of continuous shock load were 60% and 56.5%, respectively. The average effluent pH remained steady at around 6.1. From the analysis, it was revealed that the anaerobic fluidised bed had the buffering ability and was capable of treating POME with moderate removal efficiency at an influent pH of 5.0.

Keywords: anaerobic fluidised bed reactor; palm oil mill effluent; pH shock loading; reactor performance

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1. Introduction

POME is one of the most highly concentrated organic agroindustrial effluents in the world. It is characterized as having an obnoxious odour and primarily consists of about 96% water, 5% total solids, and 0.7% oil and grease^[1]. It requires an additional cost of buffer to treat POME using the conventional biological treatment technique because of its low pH in the range of 4.0–5.0. Influent pH or hydrogen ion concentration is a measure of the acidic nature of the substrate. In an anaerobic environment, acedogens produce volatile fatty acids (VFAs), and the methanogens convert the VFAs into methane (CH₄) and carbon dioxide (CO₂). The activated sludge reactor performance and stability depend mainly on the performance of these groups of microorganisms. Acetogenic bacteria have a better tolerance against low pH values, and volatile acid production may continue relatively undisturbed, despite conversion to methane being thereby inhibited. However, methanogenic bacteria have more strict requirements for pH than acidogens, with an optimum pH in the range of 6.8–8.0^[2]. Therefore, a pH below 6.2 drops the rate of methane production drastically. The conversion rate of VFA and methane production was highly dependent on the reactor pH^[3,4]. The effects of pH variations on the AnFBR and other anaerobic reactors' performance have been explored in several studies^[5–7]. The available literature on anaerobic digestion revealed that the injection of low pH influent causes souring of the reactor substrate inhibiting the optimum performance of the system. As such acidic wastewater became the source of problems for the underperformance of the common biological treatment facilities, and thus lime or caustic is used to raise influent pH. In Malaysia, burnt palm fruit bunch is commonly used as an alternative to the chemical buffering agent.

Despite a high level of biochemical oxygen demand (BOD), COD, total suspended solids (TSS), volatile suspended solids (VSS), sufficient nutrient content in this acidic and organic wastewater helps POME to be suitable for anaerobic treatment. A considerable amount of literature has been established on the biological treatability of POME under different environments^[8–11]. Previous studies have not dealt with the performance of the anaerobic fluidised bed reactor (AnFBR) at pH shock loads. Shock loading is a phenomenon whereby there is a sudden and drastic increase of digester constituents such as organic, temperature, pH, or toxicant. Nevertheless, AnFBR is reported to have the ability to handle organic shock loadings without any considerable reduction in COD removal rate^[12]. On the other hand, the effect of temperature on the anaerobic reactor performance was documented^[13,14]. Similarly, the influence of pH on anaerobic biodigester has been reported extensively in the literature^[15–18]. As POME is acidic in nature with average pH values ranging between 4.0 and 4.5, it was appropriate to consider pH change as a controlling parameter for a shock-loading study. Thus, the main objective of this research was to study the influence of pH shock loading on the performance of the AnFBR-treating acidic palm oil mill effluent.

2. Materials and methods

The acidic substrate (POME) consists of about 90%–95% water, 4.5%–9% solids (approximately half in solution and the rest in suspension), and 0.5%–1% residual oil and grease (O&G). The main nutrient contributor to this effluent is 10% protein, 12% fibre, 20% fatty materials, 11% ash, and 47% nitrogen-free extract^[19]. A typical characteristic of palm oil mill effluent is shown in **Table 1**.

Parameters	Minimum	Maximum
BOD (mg/L)	20,000	40,000
COD (mg/L)	45,000	70,000
TSS (mg/L)	20,000	35,000
VSS (mg/L)	15,000	30,000
O&G (mg/L)	5000	8000
TN (mg/L)	7000	10,000
TP (mg/L)	150	300
pН	4.0	4.5

Table 1. Typical characteristics of palm oil mill effluent (POME).

An AnFBR with a capacity of 2000 litres and an aspect ratio (height/diameter) of 5.0 was used in this study, which was possible due to the availability of materials and facilities in the laboratory. A wide range of aspect ratios was studied, ranging from $2.1^{[20]}$ to as high as 64 grounded mined sand with a uniformity coefficient (C_u) of 1.6 was used as the filter media for the pilot plant shown in **Figure 1**. The minimum fluidisation velocity for the filter media was determined, using the Ergun equation^[21] as 12.74 m/hr. Furumai et al.^[22] studied a fluidised bed reactor with an up-flow velocity of 12.5 m/hr. The recycle ratio of 45 for 12 hr HRT gave an up-flow velocity of 16.1 m/hr, which is higher than the minimum fluidisation velocity required

for the media.



Figure 1. Schematic diagram of the AnFBR pilot plan.

The media was acclimatised with active sludge taken from an anaerobic pond treating the same substrate. The raw POME was diluted to provide a uniform organic load ranging between 1.8 and 2.0 kg COD/m³.day during the start-up period. As BOD does not represent the total organic content in the substrate^[23], COD was considered as the deterministic parameter for the steady states. An ideal steady state is not practicable for pilot plants or full-scale reactors^[24]. Thus, despite the complexity of microbial population, irregularities, and fluctuation in operating conditions, it was assumed that the system had attained a steady state when the difference among COD removal rates for three consecutive tests was within 5%. Samples were analysed daily during the normal operation of the reactor. As a standard protocol the experiments and laboratory tests were conducted in duplicate, and the average values were taken as the results. The COD, total suspended solids (TSS), volatile suspended solids (VSS) and other parameters were measured following the standard methods^[25] and alkalinity was determined by HACH method 10244 procedure^[26].

3. Results and discussions

Before pH shock loading studies, five steady-state conditions were initially observed to evaluate the overall performance of the reactor. The reactor had been operating at a hydraulic retention time of 12 hr which gave the optimum COD removal efficiency and a loading rate of about 4.0 kg COD/m³.day. The COD and TSS removal efficiency at this condition was 85% and 89%, respectively. To intensify the effect of shock loading, the pH of the reactor feed was increased steadily up to 9.2 and then suddenly reduced to about a mean value of 5.1. The influent and effluent samples were analysed twice a day for pH, alkalinity, COD, and TSS. These parameters were considered as the controlling parameters during the shock load study. The overall performance was evaluated based on the mean values shown in **Table 2**.

A sudden drop in effluent pH, as shown in **Figure 2a,b**, was observed within the first 10 hr of shock load application. After the shock load was introduced, the effluent pH dropped to a minimum value of 5.9 within 5 to 10 hr. From the second day of the experiment, the effluent pH started to rise and continued until it reached an equilibrium pH of 6.0 during the rest of the days. It was observed that the effluent pH was always higher than that of the influent, though the values were less than the required minimum pH for anaerobic digestion of $6.5^{[27]}$. The study of semi-continuous UASB by Chew^[28] experienced an effluent pH value above 7.0, while the influent pH was around 4.5, which indicates better performance. Another full-scale study by Collivignarelli

et al.^[29] confirmed that AnFBRs are capable of raising effluent pH, even when the influent pH is as low as 4.0. Thus, when the AnFBR is stable, it can exhibit better buffering properties against low feed pH. Unlike anaerobic suspended growth processes, the results demonstrate that the AnFBR is capable of absorbing POME at a low pH value.

Table 2. Reactor performance at pir snock toad.													
Operating	Sample	рН		Alkal	inity	COD		RR	TSS		RR		
Condition	ID	Inf.	Eff.	Inf.	Eff.	Inf.	Eff.	(%)	Inf.	Eff.	(%)		
Optimum	Normal	7.6	7.5	562	1106	2000	300	85.0	959	105	89.0		
High pH	Normal	9.2	7.1	528	876	2400	600	75.0	980	315	67.7		
pH shock	1 st sample	5.4	6.7	396	524	2400	800	66.7	956	240	74.9		
After 1st day	2 nd sample	5.0	5.9	364	468	2100	900	57.1	972	268	72.4		
pH shock	1 st sample	4.8	6.0	360	384	2300	1000	56.5	941	261	72.3		
After 2nd day	2 nd sample	5.2	6.2	404	416	2000	800	60.0	922	239	74.1		
pH shock	1 st sample	5.1	6.0	388	420	2400	1000	58.3	983	280	71.5		
After 3rd day	2 nd sample	5.1	6.1	368	408	2500	1000	60.0	1018	304	70.1		
mean values of	parameters	5.1	6.1	380	437	2300	900	59.8	965	265	72.6		

Note: Inf. = Influent, Eff. = Effluent and RR = Removal rate.



Figure 1. Variation of (a) pH; and (b) COD concentration during the shock load study.

The optimum anaerobic digestion is reported to be achieved at a mixed liquor alkalinity range of 2000–4000 mg/L as CaCO₃. However, in this study, alkalinity at the optimum performance was obtained around 1106 mg/L as CaCO₃. However, when the AnFBR was operated before applying the shock load at a high influent pH of 9.2 and an influent concentration of 2000 mg/L, the maximum effluent alkalinity was found to be 876 mg/L (as CaCO₃). During the initial 5 hr of the shock load introduction, the effluent alkalinity was reduced to 524 mg/L (**Table 2**), indicating that much of the alkalinity acts as a buffer to increase the reactor pH. The minimum alkalinity measured during this shock load study was 384 mg/L because much of the alkalinity was used to balance or offset the pH change. Similarly, the influent COD varies between 2000 mg/L and 2500 mg/L, as shown in **Figure 3b**, giving an average VLR of about 4.6 kg COD/m³.day. The COD removal rate decreased by about 9% when the pH shock load was applied (**Figure 3b**). However, comparing the COD removal with that of the optimum performance clearly shows about 25% reduction was accounted for as the result of the pH shock load introduction. Likewise, the maximum effluent COD removal efficiency remained constant from the 2nd day onward.



Figure 2. Variation of (a) TSS concentration; and (b) removal rate in the shock load.

However, the TSS removal efficiency increased from 67.7% to 74.9% when the pH shock load was added to the reactor. After that, little change in effluent TSS concentration and removal rates was observed, as shown in **Figure 3a,b**. These might be due to the possibility of fast hydrolysis of the organic particulate occurring at the pH of 6.0, which is reported to be optimum for the hydrolysis of carbohydrates^[30]. On the other hand, lower pH must have increased the solubility of particulate, at least to some extent, which resulted in a better TSS removal rate during the shock load. However, compared to the optimum TSS removal rate performance of the reactor during the shock load was reduced from 89% to the average value of 72.6%.

Therefore, from the performance analysis of AnFBR at pH shock load in treating POME, it can be generally recommended that an anaerobic fluidised bed reactor can absorb an average pH shock of 5.0. That means stable AnFBR can withstand pH shock at a mean volumetric loading rate (VLR) of 4.6 kg COD/m³.day. As such, the raw POME can be fed into a matured and stable AnFBR without neutralising the influent pH, and the reactor can be expected to perform with moderate efficiency and the admissible souring of the mixed liquor.

4. Conclusion

A pilot-scale anaerobic fluidised bed reactor was studied to determine its ability to withstand a pH shock load for the palm oil mill effluent (POME) treatment. The reactor performance was evaluated using a neutralised POME for five steady states at different loading rates. The AnFBR exhibited an optimum removal efficiency of 85% at a neutralised feeding rate of 4.0 kg COD/m³.day. Decreased feed pH from 9.2 to about 5.1 resulted in reduced effluent pH and alkalinity of about 6.1 and 437 mg/L (as CaCO₃), respectively. The average COD and TSS removal rate during the pH shock load was 59.8% and 72.6%, respectively. Hence, it can be concluded that the raw POME can be fed into a matured and stable AnFBR without neutralising the influent pH, and the reactor can be expected to perform with moderate efficiency.

Author contributions

Conceptualization, AAM; methodology, AAM and AI; formal analysis, AAM; investigation, AAM and MM; resources, AI; data curation, AAM and AI; writing—original draft preparation, AAM; writing—review and editing, AI and MM; supervision, AI; project administration, AAM and AI; funding acquisition, AI. All authors have read and agreed to the published version of the manuscript.

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Conflict of interest

The authors declare no conflict of interest.

References

- Lee ZS, Chin SY, Lim JW, et al. Treatment technologies of palm oil mill effluent (POME) and olive mill wastewater (OMW): A brief review. Environmental Technology and Innovation 2019; 15: 100377. doi: 10.1016/j.eti.2019.100377
- 2. Sitorus B, Sukandar, Panjaitan SD. Biogas recovery from anaerobic digestion process of mixed fruit-vegetable wastes. Energy Procedia 2013; 32: 176–182. doi: 10.1016/j.egypro.2013.05.023
- Eryildiz B, Lukitawesa, Taherzadeh, MJ. Effect of pH, substrate loading, oxygen, and methanogens inhibitors on volatile fatty acid (VFA) production from citrus waste by anaerobic digestion. Bioresource Technology 2020; 302: 122800. doi: 10.1016/j.biortech.2020.122800
- 4. Zhang W, Li L, Xing W, et al. Dynamic behaviors of batch anaerobic systems of food waste for methane production under different organic loads, substrate to inoculum ratios and initial pH. Journal of Bioscience and Bioengineering 2019; 128(6): 733–743. doi: 10.1016/j.jbiosc.2019.05.013
- 5. Alzate ME, Muñoz R, Rogalla F, et al. Influence of substrate to inoculum ratio, biomass concentration and pretreatment. Bioresource Technology; 2012; 123: 488–494. doi: 10.1016/j.biortech.2012.06.113
- Capson-Tojo G, Trably E, Rouez M, et al. Dry anaerobic digestion of food waste and cardboard at different substrate loads, solid contents and co-digestion proportions. Bioresource Technology 2017; 233: 166–175. doi: 10.1016/j.biortech.2017.02.126
- Kawaiv M, Nagao N, Tajima N, et al. The effect of the labile organic fraction in food waste and the substrate/inoculum ratio on anaerobic digestion for a reliable methane yield. Bioresource Technology 2014; 157: 174–180. doi: 10.1016/j.biortech.2014.01.018
- Borja R, Banks CJ, Sánchez E. Anaerobic treatment of palm oil mill effluent in a two-stage up-flow anaerobic sludge blanket (UASB) system. Journal of Biotechnology 1996; 45(2): 125–135. doi: 10.1016/0168-1656(95)00154-9
- 9. Halim KA, Yong EL. Integrating two-stage up-flow anaerobic sludge blanket with a single-stage aerobic packedbed reactor for raw palm oil mill effluent treatment. Malaysian Journal of Sustainable Agriculture (MJSA) 2018; 2(1): 15–18. doi: 10.26480/mjsa.01.2018.15.18
- 10. Kim SH, Choi SM, Ju HJ, et al. Mesophilic co-digestion of palm oil mill effluent and empty fruit bunches. Environmental Technology (United Kingdom) 2013; 34(13–16): 2163–2170. doi: 10.1080/09593330.2013.826253
- 11. Vijayaraghavan K, Ahmad D, Aziz MEBA. Aerobic treatment of palm oil mill effluent. Journal of Environmental Management 2007; 82(1): 24–31. doi: 10.1016/j.jenvman.2005.11.016
- 12. Oliva E, Jacquart JC, Prevot C. Treatment of waste water at the El Aguila brewery (Madrid, Spain). Methanization in fluidized bed reactors. Water Science and Technology 1990; 22(1–2): 483–490. doi: 10.2166/wst.1990.0172
- Giuliano A, Zanetti L, Micolucci F, Cavinato C. Thermophilic two-phase anaerobic digestion of source-sorted organic fraction of municipal solid waste for bio-hythane production: Effect of recirculation sludge on process stability and microbiology over a long-term pilot-scale experience. Water Science and Technology 2014; 69(11): 2200–2209. doi: 10.2166/wst.2014.137
- 14. Oles J, Dichtl N, Niehoff HH. Full scale experience of two stage thermophilic/mesophilic sludge digestion. Water Science and Technology 1997; 36(6–7): 449–456. doi: 10.1016/S0273-1223(97)00554-4
- Devlin DC, Esteves SRR, Dinsdale RM, Guwy AJ. The effect of acid pretreatment on the anaerobic digestion and dewatering of waste activated sludge. Bioresource Technology 2011; 102(5): 4076–4082. doi: 10.1016/j.biortech.2010.12.043
- Guštin S, Marinšek-Logar R. Effect of pH, temperature and air flow rate on the continuous ammonia stripping of the anaerobic digestion effluent. Process Safety and Environmental Protection 2011; 89(1): 61–66. doi: 10.1016/j.psep.2010.11.001
- Wang K, Yin J, Shen D, Li N. Anaerobic digestion of food waste for volatile fatty acids (VFAs) production with different types of inoculum: Effect of pH. Bioresource Technology 2014; 161: 395–401. doi: 10.1016/j.biortech.2014.03.088
- 18. Zhai N, Zhang T, Yin D, et al. Effect of initial pH on anaerobic co-digestion of kitchen waste and cow manure. Waste Management 2015; 38: 126–131. doi: 10.1016/j.wasman.2014.12.027
- 19. Ma AN, Cheah SC, Chow M, et al. Current status of palm oil processing wastes management. Environmental Science 1993. Available online: https://www.semanticscholar.org/paper/Current-status-of-palm-oil-processing-wastes-Ma-Cheah/0988f27ee3500559ca98551e90f1c4c78c0fcd8b (accessed on 7 August 2023)
- 20. Sutton PM, Huss DA. Anaerobic fluidized bed biological treatment: pilot to full-scale demonstration (New Orleans, Louisiana). Water Pollution Control Federation 1984.
- 21. Idris A. Scale-up studies on anaerobic fluidized bed [PhD thesis]. University of Newcastle Upon-Tyne, United Kingdom; 1989.
- Furumai H, Kuba T, Imai T, Kusuda T. Transient responses of wastewater treatment and biomass development in a methanogenic fluidized bed. Water Science and Technology 1991; 23(7–9): 1327–1336. doi: 10.2166/wst.1991.0585

- 23. Orhon D, Taşh R, Sözen S. Experimental basis of activated sludge treatment for industrial wastewaters—The state of the art. Water Science and Technology 1999; 40(1): 1–11. doi: 10.1016/S0273-1223(99)00357-1
- 24. Hobson PN, Wheatley AD. Anaerobic Digestion: Modern Theory and Practice England. Elsevier Science Publishers; 1993.
- 25. APHA. Water Environment Federation, American Water Works Association. Standard Methods for the Examination of Water and Wastewater; 1999.
- 26. HACH. Alkalinity Method; 2014. Available online: https://uk.hach.com/quick.searchquick.search.jsa?keywords=Alkalinity+Method+10244 (accessed on 7 August 2023)
- 27. Fannin KF, Srivastava VJ, Mensinger JD, Chynoweth DP. Marine Biomass Program: Anaerobic Digester Process Development and Stability Study. Institute of Gas Technology; 1983.
- 28. Chew TY. The Use of Flocculants in Anaerobic Digestion of Palm Oil Mill Effluent [Master's thesis]. Universiti Pertanian Malaysia; 1994.
- Collivignarelli C, Urbini G, Fameti A, et al. Anaerobic-aerobic treatment of municipal wastewater with full-scale UASB and attached biofilm reactor. Warter Science and Technology 1990; 22(1–2): 475–482. doi: 10.2166/wst.1990.0171
- 30. Matsumoto A, Noike T. Effects of substrate composition and loading rate on methanogenic process in anaerobic fluidized bed systems. Water Science and Technology 1991; 23(7–9): 1311–1317. doi: 10.2166/wst.1991.0583