Anaerobic Treatment of Produced Water

F.C. Khong, M.H. Isa, S.R.M. Kutty, S.A. Farhan

***Abstract*—**An experimental study of anaerobic treatment was performed by hybrid upflow anaerobic sludge blanket (HUASB) reactor to treat produced water (PW) of an onshore crude oil terminal (COD: 1597 mg/L, NH_3-N: 14.7 mg/L, phenol: 13.8 mg/L, BOD_5: 862 mg/L, sodium: 6240 mg/L and chloride 9530 mg/L). The produced water with high salinity and other toxic substances will inhibit the methanogens performance if there is no adaptation on biomass before anaerobic digestion. COD removal from produced water was investigated at five different dilutions of produced water and tap water (TW) without any nutrient addition and pre-treatment. The dilution ratios were 1PW:4TW, 2PW:3TW, 3PW:2TW, 4PW:1TW and 5PW:0TW. The reactor was evaluated at mesophilic operating condition (35 ± 2 °C) at 5 days of HRT for 250 days continuous feed. The average COD removals for 1PW:4TW, 2PW:3TW, 3PW:2TW, 4PW:1TW and 5PW:0TW were found to be approximately 76.1%, 73.8%, 70.3%, 46.3% and 61.82% respectively, with final average effluent COD of 123.7 mg/L, 240 mg/L, 294 mg/L, 589 mg/L and 738 mg/L, respectively.

***Keywords*—**Anaerobic, fixed film, hybrid UASB, produced water, inhibitor

I. Introduction

PRODUCED WATER (PW), formation water or oilfield brine, is defined as any water that is present in a reservoir with hydrocarbon resource and brought to the surface with crude oil or natural gas during onshore or offshore operation [1]. PW properties and volume can vary throughout the lifetime of a reservoir. The amount and quality of PW generated is dependent upon the nature of formation and the recovery method. PW is the largest waste stream for hydrocarbon production. A recent report shows that daily single water production volumes significantly exceeds that of oil, to the extent that 211 million barrels of water are produced by the industry daily compared to 85 million barrels of oil in which the PW treatment market is expected to worth US$ 4.3 billion for the next five years [2]. Besides the large number of barrels of water, it contains naturally-occurring chemicals i.e. salinity, inorganic ions, metals, radioisotopes, organic acids, total organic carbons, phenols, petroleum hydrocarbons, volatile hydrocarbons, aliphatic hydrocarbons, polycyclic aromatic hydrocarbons and other organic components and production chemicals [3].

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Previously, shore-side treatment facilities i.e. onshore recovery by gravity separation of crude oil, gas or water are designated and extraction of water is permitted for territorial sea discharge [4]. However, in Malaysia, due to the increasing amount of PW, the fate and effect of discharging PW into water bodies has become a significant issue of environmental concern which is now required to comply with Environmental Quality (Industrial Effluent) Regulations 2009. Therefore, it is vital to deal with produced water efficiently and cost effectively to ensure fully optimized production and compliance with national and regional regulations.

Anaerobic treatment was chosen as it is an efficient, economical and environmentally-friendly method. Anaerobic treatment is technologically simple with low energy consumption. It is an established technology for the treatment of wastes and wastewater. The final product is biogas which is a mixture of methane (55 – 77 vol %) and carbon dioxide (25 – 45 vol %) that can be used for heating and upgrading natural gas quality or co-generation [5]. Fat, protein, and cellulose from POME wastewater which contain suspended and colloidal components can cause deterioration of microbial activities and wash out of active biomass. [6] - [7]. The use of packing media in the middle portion reduced channelling problem and loss of biomass due to flotation associated with poorly performing UASB. Additionally, the packing material caused the flocculated biomass to precipitate over the sludge blanket to serve as suitable and natural hydrophobic core for the development of granular sludge [8]- [9]. HUASB (a combination of UASB and Anaerobic filter) allows treatment of low strength wastes by maintaining long solid retention time (SRT) independent of the hydraulic retention time (HRT). This system will reduce the need for elevated temperatures [10].

However, a wide variety of inhibitory substances are the primary cause of anaerobic digester upset or failure since they are present in substantial concentration in wastes especially produced water which usually contains high suspended solids (SS) and is heavily polluted and difficult to degrade [11]. High salinity of produced water affects the metabolism of microorganisms due to plasmolysis in the presence of salt [12]. Sodium concentration in the reactor may increase because of the use of NaOH or Na_2CO_3 for pH control [13]. Therefore, before anaerobic digestion, adaptation of microorganisms by serial of dilution to inhibitory substances and incorporation methods to remove or counteract toxicants can significantly improve treatment efficiency [14].

II. Background

Traditionally, activated sludge process has been the common method for treating wastewater because it could maintain a total petroleum hydrocarbon (TPH) removal efficiency of 98 – 99% at a sludge retention time (SRT) of 20 days[15]-[16]. Sequencing batch reactor (SBR), aerated lagoon, waste stabilization pond, trickling filter, rotating biological contactor and filtration are also widely used to treat produced water [17]. Freire et al.[18] studied COD removal with different percentages of produced water and sewage. COD removal efficiencies varied from 30 - 50% in mixtures of 45% and 35% (v/v) of wastewater.

Fakhru'l-Razi et al.[19] improved biological treatment of effluent by coupling membrane to SBR; COD, total organic carbon (TOC) and oil and grease (O&G) removal efficiencies were 90.0%, 92% and 91.2%, respectively. Another study was conducted to compare the TOC removal by three different biological systems including SBR, trickling filter and chemostat reactor with acclimated microorganisms in 180 mg/L ofNaCl. Total TOC removal in SBR was 80% which was higher than trickling filter or chemostat. However, the study showed that continuous operation of SBR could lead to a loss of biomass [20].

Waste stabilization pond (WSP) with a deep separating baffle between the aerobic and anaerobic zones and three shallow baffles serving as oil stoppers were employed [21]. The COD and O&G removals were 85% and 82%, respectively and improved over time. An anoxic hydrolysis-aerobic treatment was conducted on a hydrolysis acidification/bio-contact oxidation system (HA/BCO) [12]. It was able to remove COD by 63.5%, ammonia nitrogen(NH_3-N) by 45%, TSS by 79.5% and TPH by 68%.

Even though aerobic treatment alone has high efficiencies in removing biodegradable organic compounds in contrast to anaerobic treatment, aerobic treatment is generally characterised by high operational cost (energy), while a very large fraction of the waste is converted to another type of waste (sludge) [22]. In addition, during anaerobic treatment, energy for on-site use is produced in the form of methane. It can be converted to mechanical or electrical energy for other purposes [23]. With these reasons, anaerobic treatment has become a favourable method.

Gallaghner[24]had conducted a research by using a small fixed film anaerobic reactor with a low-density of porous as packing material. Biodegradation of organic acids in produced water under anaerobic condition in the presence of naphthenic acids were investigated and showed that no biodegradation of naphthenic acids was observed in anaerobic condition. Other researchers [25] treated produced water by anaerobic process coupled with micro-electrolysis. COD and biodegradability were studied and showed that COD in anaerobic alone exhibited an abnormal change which it increased within 168 hours before decreased to 222 mg/L after 360 hours of treatment (average removal was 15%). Total COD removal when coupling with micro-electrolysis was 53.3%.

Rincon et al.[26] studied three categories of anaerobic biodegradation of water separated from extracted crude using up-flow anaerobic sludge blanket (UASB) with acclimatization of produced water. The COD removal efficiency for light, medium and heavy oils were 87%, 20% and 30%, respectively. They claimed that acclimatization of anaerobic sludge did not reduce COD in light, medium or heavy oils. G.D.Li et al. [27] used anaerobic baffled reactor (ABR) to treat heavy oil for produced water, with COD and oil removal of 65% and 88%, respectively.

However, anaerobic treatment may be hindered by the presence of sodium salt, which can cause inhibition and toxicity problems in the methanization process. The level of inhibition depends on the concentration of sodium ion. McCarty [28]reported that sodium is essential for methanogens in the range of 100 – 200 mg/L and will begin to inhibit moderately in the range of 3500 – 5500 mg/L and above 8000 mg/L to be strongly inhibitory to methanogens at mesophilic temperatures.

Panswad and Anan[29] studied high salt acclimatization and showed that the biomass was able to adapt themselves to high salt environment when there was no significant drop of mixed liquor suspended solids (MLSS). COD removal efficiency decreased as salt concentration went up. The salt effect was more severe on the non-acclimated system and anaerobic performance should still be satisfactory as long as the salinity concentration is less than 10 g/L [30].

Besides salinity, produced water has high concentration of ammonia, sulphide, heavy metals and many other toxic substances and it is highly recommended for methanogens to acclimatize themselves to those toxic substances. Dilution of produced water is recommended to adjust the characteristics of the final mixture according to a predefined strategy to evolve the applied organic loading rate and toxic substances in the digester to achieve biomass acclimatization [31] - [32].

The aim of this study was to evaluate the performance of HUASB reactor in removing COD from produced water at five different dilutions of produced water and tap water (TW):- 1PW:4TW, 2PW:3TW, 3PW:2TW, 4PW:1TW and 5PW:0TW.

III. Methodology

A. Water Sampling and Characteristics

The produced water was collected from a local onshore crude oil terminal in Malaysia. During on-shore recovery, the crude/gas/water mixture is processed by gravity separation at the well site and the water extract is discharged into surface pits or above ground storage facilities for evaporation or subsequent reinjection into the subsurface for permanent disposal or secondary recovery.

Samples of produced water were stored at 4°C after sampling. Characteristics of the produced water are shown in Table I.

TABLE I
PRODUCED WATER CHARACTERISTICS

Parameters	mg/L	Parameters	mg/L
Sodium	6240	COD/N/P ratio	350/3.2/0.4
Potassium	125	COD/BOD_5 ratio	1.9
Calcium	325	TSS	57.33
Magnesium	230	BOD_5	862
Barium	<0.50	COD	1597
Strontium	10	TOC	0.501

Iron	0.30	pH	7.93
Chloride	9530	NH_3-N	14.7
Sulphate	6250	TDS	19070
Bikarbonate	1810	Oil & Grease	30
Boron	17	Phenol	13.8
Aluminium	<1.0	Total Phosphorus	2.0
Silicon	27	Color (PtCo)	678.7
Phosphorus	<0.30	Lithium	1.6

B. Experimental Set-up

A schematic diagram of experimental setup is shown in Fig. 1. A 5-L Hybrid UASB containing a 3.5 L portion of UASB and 1.5 L of fixed film. Plastic media from *EasyPro* with 3 cm in diameter were used as fixed film media. There was no agitation or recirculation inside the reactor. The reactor was operated in mesophilic operating condition (35 ± 2 °C). A temperature controller was connected to a heating blanket to maintain equivalent temperature and the temperature was measured by a temperature probe. A peristaltic pump was used to continuously feed the reactor with produced water at 5 days of HRT for 250 days. A sampling port on the bottom of the reactor was used for sludge sampling. The top reactor was connected to a gas tank for gas collection by water displacement.

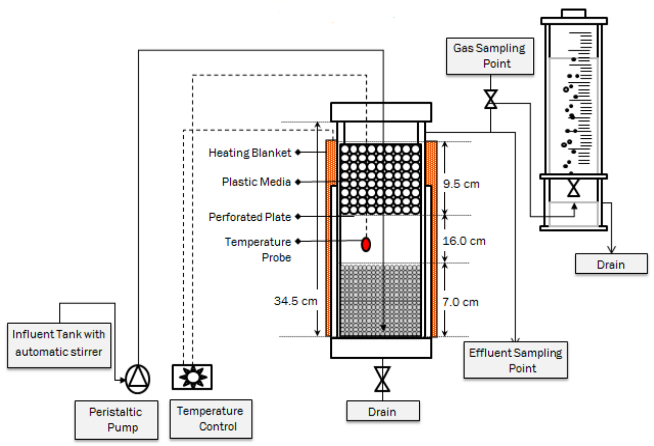

Fig. 1 Schematic diagram of experimental setup of HUASB

C. Organism, Medium and Operating Parameters

The reactor was seeded with the sludge of a mesophilic anaerobic digester from a local petrochemical company containing 18.0 g VSS with volatile fraction of 84.72%. The growth medium is recommended by the Oregon Collection of Methanogens for MSH medium [24] and Nutrient Requirement for UASB process [33]- [34].

The growth medium composed of 1.8 g/L acetate, 0.2 g/L propionate, 1.0 g/L butyrate, 0.2 g/L $CaCl_2$, 0.2 g/L $MgCl_4.6H_2O$, 50 mg/L $FeCl_2.4H_2O$, 1.0 g/L NH_4Cl, 0.5 g/L K_2HPO4, 0.25 g/L Na_2SO_4 and 1.0 g/L yeast. Sucrose was used as the carbon source. Trace elements of 0.5 mg/L $ZnCl_2$, 0.5 mg/L $CuCl_2$, 1.0 mg/L $NiCl_2.6H_2O$, 1.0 mg/L $MnSO_4.6H_2O$, 0.5 mg/L H_3BO_3, 0.5 mg/L $(NH_4)_3Mo_7.4H_2O$, 0.5 mg/L $CoCl_2.6H_2O$, 0.5 mg/L $AlCl_3$, 4.0 mg/L EDTA. $NaHCO_3$ was provided to improve the buffering capacity (2.5-3.5 g $CaCO_3$/L).

The sludge was fed with 7.0 - 8.0 g/L high strength medium growth for 40 days that is decreased gradually to 4.0 g/L for 10 days at HRT of seven days and 1.0 g/L for 31 days at HRT of five days.

Sample characteristic also show that sodium of PW was 6240 mg/L which can be categorized as moderately inhibitory to methanogens. Acclimatization of methanogens over prolonged periods of time could increase the tolerance and shorten the lag phase before methane production begins [31], minimize the toxicity built up and allow microorganisms sufficient time to adapt. Therefore, the reactors were seeded with five different dilutions of PW and tap water (TW) without any micro or macro nutrients added. The dilution ratios were 1PW:4TW, 2PW:3TW, 3PW:2TW, 4PW:1TW and 5PW:0TW (100% PW) with the HRT of five days.

D. Analysis

The reactor was monitored for basic water quality parameters viz. biochemical oxygen demand (BOD), pH, chemical oxygen demand (COD), alkalinity, total suspended solids (TSS) and volatile suspended solids (VSS). Laboratory analyses were conducted according to Standard Methods [35].

The methane content of biogas was measuredwith Agilent GC with a packed column of 20% TCEP PAWS80/100, nitrogen as a carrier gas (30 ml min^{-1}), column temperature of 150°C and injection port and detector (FID) temperatures at 200°C and 160°C respectively. Sample injection volume is 1.0 ml [36].

IV. Results And Discussion

A. Sludge Seeding

The sludge was seeded in the UASB reactor with three strengths of growth medium, 7.0 g/L and 4.0 mg/L COD with HRT of seven days, and 1.0 g/L COD for HRT of five days. The COD removal ranged from 80% to 96%.

The sludge contained 18.0 g VSS with 84.72% of volatile fraction at the beginning and increased to 19.7 g VSS with 89.94% of volatile fraction after 92 days of seeding with growth medium.

The influent, effluent COD and COD removal efficiency in UASB during cultivation is summarized in Fig. 2.

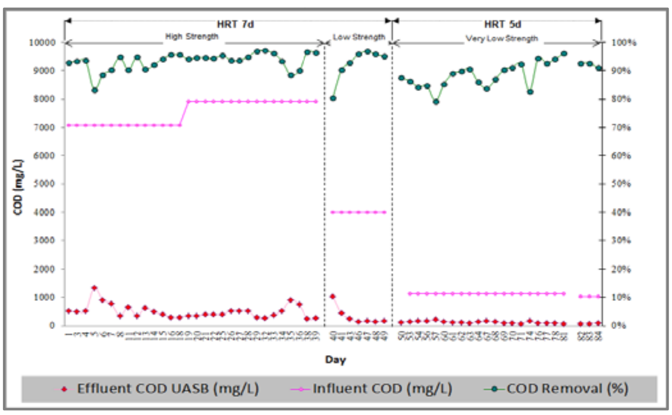

Fig. 2 COD removal rate and COD removal percent at different HRT and strength

B. Reactor Performance

1) Alkalinity and pH

Alkalinity of the reactor was maintained between 2.5 – 3.5g $CaCO_3$/L. The pH of influent was adjusted to 6.5 – 7.5. The pH of effluent ranged from 8.1 to 8.9, presumably as a result of alkalinity supplementation, but did not appear to have any adverse effect on reactor performance.

2) Methane Production

Percentage methane of biogas, throughout this study were in increasing trend where CH_4 from 35.98 mol% (3PW:2TW), 38.74 mol% (4PW:1TW) to 44.62 mol% (5PW:0TW). Inhibitory effect of produced water might be the reason of low rate of methane formation and methanogenic activity.

3) COD, TSS and VSS

When the reactors were seeded with different ratios of PW and TW: 1PW:4TW, 2PW:3TW, 3PW:2TW, 4PW:1TW and 5PW:0TW (100% PW), the COD removal efficiency began to decrease slowly as shown in (Table II) Fig. 3 and Fig. 4. The final VSS was 26.8 g and 81.7% of volatile fraction after 250 days of operation.

TABLE II
COD REMOVAL IN DIFFERENT FEED RATIO

	Ratio	Influent COD (mg/L)	COD Removal Efficiency (%)
1	1PW:4TW	550 - 570	70.92 – 84.64
2	2PW:3TW	700 - 800	66.33 – 73.10
3	3PW:2TW	925 - 1150	65.65 – 77.80
4	4PW:1TW	1010 - 1300	41.02 – 58.15
5	5PW:0TW	1400 - 1650	42.67 – 67.49

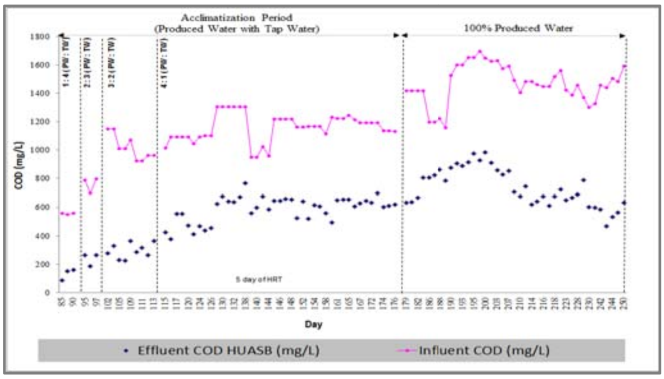

Fig. 3 COD removal rate in HUASB reactor

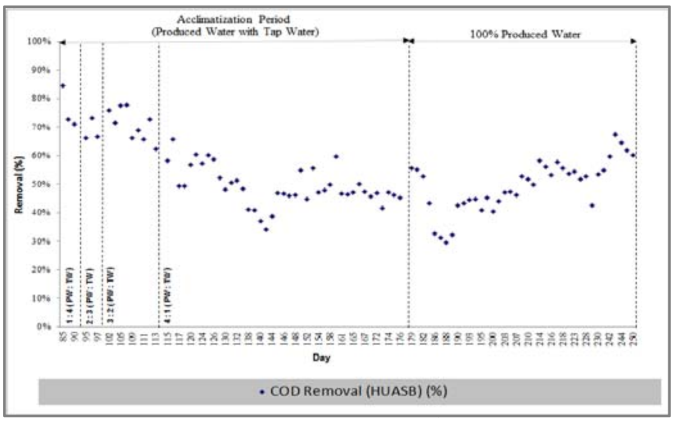

Fig. 4 COD removal percent in HUASB reactor

V. CONCLUSION

Biomass acclimatization by adjusting the characteristics of the final mixture according certain ratio over prolonged periods of time could increase the tolerance, shorten the lag phase before methane production begins, minimize the toxicity built up and allow microorganisms sufficient time to adapt. This study reveals that the treatability of produced water using hybrid UASB to remove COD at five different dilutions of produced water and tap water (TW). The dilution ratios were 1PW:4TW, 2PW:3TW, 3PW:2TW, 4PW:1TW and 5PW:0TW was successful when COD removal at ratio 5PW:0TW started to increase and possible to remove further if time permitted.

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