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Bioreactor Landfilling of Oil Sludge

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ABSTRACT

Biochemical or thermochemical treatment of industrial waste not only is environmentally sound, but also is a potential source for high heating value fuel, chemical and sensible heat. The petroleum industry is been generating disconcerting amount of solid waste in the form of oily sludge. This hazardous sludge is a mixture of petroleum hydrocarbons (PHCs), solid particles and soil, water and heavy metals. This work will focus on the anaerobic digestion of PHC waste in a well-controlled bioreactor for the generation of landfill/biogas gas (LFG). Several conditions has been attempted to maximize the LFG generation and the specific methane yield (SMY). This include the influence of temperature, addition of water, different reactor size and the co-digestion with waste water treatment sludge (WWTS). The best results were obtained in the larger 5.3 kg reactor, at 35 ^{o}C temperature and with the additional of 2.3 liters of water at 60%PHC to 40% WWTS. At this condition the yield was 55.5% CH_4 molar fraction, only traces of CO_2, and SMY of 0.21 (N L CH_4 kg-1).

Keywords: Bioreactor; Landfill-gas; Sludge; Anaerobic-digestion

I. INTRODUCTION

The oily sludge is described as remnants obtained from the water, oil, fat and solids, organic compounds and minerals predominantly by alkanes [1]. Different treatment methods, such as incineration, gasification, pyrolysis, and biodegradation [2] have been explored to stabilize oily sludge waste. The high inorganics contents and the low heating value of PHC waste inflate their thermal treatment energy penalty and rendering the process uneconomically..

The most common waste management solution is landfilling, but the limited urban space and negative public perception is phasing out this option [3]. Anaerobic digestion is an attractive waste treatment practice in which both pollution control and energy recovery can be achieved.

Anaerobic digestion involves four sequenced biochemical reactions that associated with different colonies of anaerobic microorganism leading to the formation of landfill gas and intermediate organic acids. These are hydrolysis, acidogenesis, acetogenesis and methanogenesis [4].

Anaerobic digestion involves the degradation and stabilization of organic materials under anaerobic conditions by microbial organisms and leads to the formation of biogas (a mixture of carbon dioxide and methane, a renewable energy source) and microbial biomass [5]. Compared to mesophilic digestion, thermophiles anaerobic digestion has additional benefits including a high degree of waste stabilization, more thorough destruction of viral and bacterial pathogens, and improved post-treatment sludge dewatering [6]. In anaerobic digestion, the acid forming and the methane forming microorganisms differ widely in terms of physiology, nutritional needs, growth kinetics, and sensitivity to environmental conditions [7].

In order to describe the performance and kinetics of the anaerobic digestion process for methane generation, several anaerobic digestion models have been developed in the last 30 years [8]. Moreover, operation temperature is also an important parameter in AD process [9]. A psychrophilic anaerobic digestion (PAD near 20 ^{o}C) offers several advantages, i.e. reduces odors, decreases the organic pollution load by more than 70% [11], produces high quality biogas, significantly diminishes pathogens survival [12], and improves the agronomic value of digestive [13]. A psychrophilic dry anaerobic digestion (PDAD) was performed by Saady and Masse [9] on cow feces. They stated that PDAD offers the energy advantage compared to mesophilic and thermophiles. In their results, they indicated that PDAD at 20℃ of cow feces and wheat straw is feasible at total solid (TS) of 21% [9]. Table 1 summarizes the reported SMY. It is mainly for the livestock manures and it ranges between 85 to 280 l/kg per 20% total solid.

Varel [10] reported the highest SMY of 240-280 L/kg. Agricultural and industrial wastes can be ideal candidates for anaerobic digestion if they contain high levels of easily biodegradable materials. Problems such as low methane yield and process instability are often encountered in anaerobic digestion, preventing this technique from being widely applied. In this work the PHC industrial waste will be subjected to anaerobic digestion under different conditions to assess its viability for anaerobic biodegradation process.

Table 1: Reported literature on the SMY using anaerobic digestion

Temperature	Feedstock	SMY (N L CH4 kg-1 VS)	Reference
35-65	Cattle manure	240-280	Varel (1980) [10]
20	Cow feces	184.5 ± 24	Saady and Masse (2014) [9]
65	Cattle manure	165	Ahring (2001) [11]
30	Dairy cattle manure	164	Shyam (2002) [13]
35	Dairy cattle feces	148 ± 41	Moller (2004) [14]
30	Dairy cattle manure	135	Somayaji Khanna (1994) [15]

II. MATERIAL AND METHODS

i) Material Characterization: Numerous samples of the PHC and WWTS sludge from BeAAT/TAKREER (Abu Dhabi refinery) and Tadweer (The Center of Waste Management Abu Dhabi) were obtained and subjected to homogenization by thorough mixing using soft hammering and manual stirring in an inverted bill-shaped container. This is followed by several sampling for: i) Thermo-Gravimetric Analysis (TGA) using Q600 thermo-scientific) targeting approximate analysis, and ii) CHNSO elemental analysis using thermoscinetific Flash2000 analyzer. The samplings preparation is described elsewhere by Shabbar et al. [10]. A constant nitrogen purge rate of 100 mL/min was kept and the temperature was set to stabilize at 50°C and remain isothermal for 5 minutes before starting the ramp up at 20°C/min. To remove all moisture, the temperature was kept constant at the 5th heating minute for 5 minutes before continuing the ramp up to 900 °C with 20 ^{0}C/min. The second ramp up release the volatiles from the sample. At 900 °C the purge gas was switched from nitrogen to oxygen to burn the remaining fixed carbons. After switching the gas, the sample was kept at 900 °C for 15 minutes before the end of the analysis. TGA analysis was repeated five times to obtain statistically acceptable values. The results of the TGA analysis are summarized in table 1.

Table 1: Proximate analysis of PHC and WWTS

Proximate	PHC	WWTS	Reactor Feed
Moisture %	3	50	21.8
Volatile %	11	30	18.6
Fixed-carbon %	27	5	18.2
Ash %	60	15	42

Ultimate analysis carried out using Flash CHNOS to measures the elemental fractions of the feedstock at two configurations. In the first configuration for CHNS measurements a complete sample combustion incurred at temperatures up to 1800°C using high purity oxygen at 250ml/min [16] that insures combustion and formation of a homogenized gas stream of CO_2, H_2O, NO_2, SO_2. The gases are then separated in the chromatographic column and quantified using the thermal conductivity detection (TCD) method. The second configuration is used for oxygen determination which is pyrolysis of the sample under 1060°C using helium as the carrier gas at 100ml/min [16]. Results of ultimate analysis of the PHC and WWTS are depicted in Table 2.

Table 2: Ultimate analysis of PHC and WWTS

Ultimate analysis	PHC	WWTS
Carbon [wt%]	16.53	4.1
Hydrogen [wt%]	1.29	0.34
Nitrogen [wt%]	0.08	0.98
O2 [wt%]	12.7	2.32
Sulphur [wt%]	0.6	0.65
Molecular formula	$CH_{0.965}O_{0.593}N_{0.004}S_{0.015}$	$CH_{0.091}O_{0.565}N_{0.2}S_{0.16}$

The above two analysis allows to infere the unit molecular formaula for the feedstock. This is based on sigle mole of carbon leading to $\mathbf{CH_{0.965}O_{0.593}N_{0.004}S_{0.015}}$ representation for PHC and $\mathbf{CH_{0.091}O_{0.565}N_{0.2}S_{0.16}}$ for the WWTS.

ii) Reactor design: Two sets of cylindrical jar-type bench scale reactors were designed and made of transparent glass and acrylic material. The two are at 10cm radius, but at 30cm and 90cm lengths. The reactors are accessible by multiple inlets and outlet ports using precision machined air tight and multiple port endcap made of acrylic. These reactors are designed to simulate the biodegradation activities that take place in

the scale up engineering landfill bioreactor. This jar-type reactors contains all the basic elements of the landfill bioreactors, including the separation anti-leakage membrane which equivalent to the jar wall, the leachate circulation system, gas collection well and the topping membrane represented by the sealed reactor cap and its accessible ports that extend to a mall aquarium type pump for leachate circulation. The four main accessible ports for these jar reactors are the gas output, moisture addition access, the leachate inlet, and leachate outlet which extends to the bottoming base-course layer. The leachate collection takes place at the very bottom of the jar which is separated from the toping feedstock by means of a stiff and porous scrapping sponge. Schematic of the reactor and their laboratory experimental set up is shown in Fig. 2a and 2b.

The gas port is connected to water displacement column for precise gas measurements. Following the gas measurements, several samples are directed and released into a Teflon toddler/DuPont bags which can be directly fed to the GC/MS and Gas-board analyzers for appropriate gas analysis. The composition of this gas is mainly CH_4, CO_2, O_2 and H_2S. Several reactors are used for multiple experimental setups to simulate and optimize the biodegradation of the PHC in anaerobic conditions. These are divided into three sets according to: i) the feedstock composition (fraction of PHC to that of WWTS), ii) temperature conditions (i.e. psychrophilic or mesophilic), and iii) with or without the addition of water.

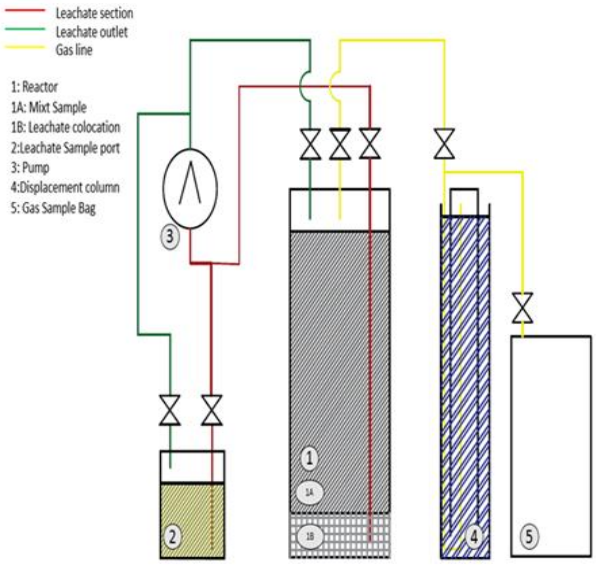

Fig. 1a: Schematic set up of the bench scale bioreactor

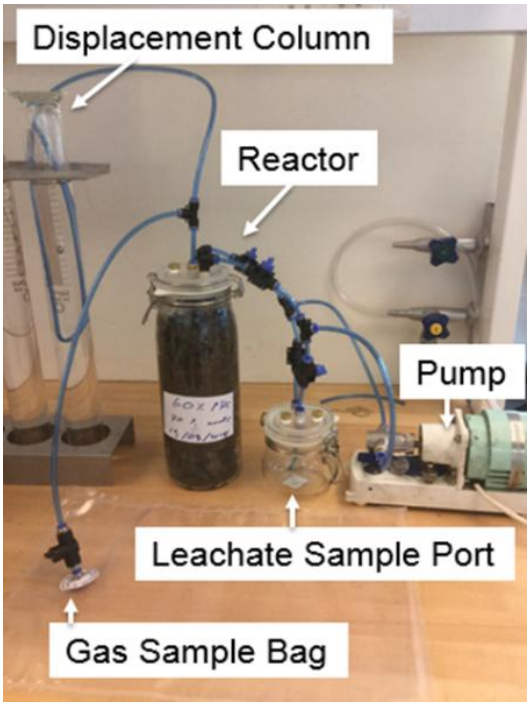

Fig. 2b: Experimental set up of the bench scale bioreactor showing the leachate and displacement column setup

III. LANDFILL GAS ESTIMATION

The estimated theoretical yield follows the biodegradation stoichiometric eq. (1). Thus, given the feedstock unit formula in the form of $C_aH_bO_cN_d$,that inferred from the proximate and ultimate analysis, provides the basis of theoretical estimation of the gas volume that could be produced from oily sludge.

$$C_aH_bO_cN_d + ((4a-d-2c-3d)/4)\ H_2O \rightarrow ((4a+d-2c-3d)/8)\ CH_4 + ((4a-d+2c-3d)/8)\ CO_2 + dNH_3 \quad (1)$$

The anticipated landfill gas values both in unit mass and volume are summarized in table 3. Conventionally, higher volume fraction of CH_4 is produced than CO_2 and potentially reaching twice the volume.

IV. EXPERIMENTAL SETUP

Three sets of experimental setups were carried out each with multiple reactors as summarized in table 4. The goal of multiple setups is to identify the best experiment for the anaerobic biodegradation to generate the maximum SMY. The three experiments are discussed below.

A. Experiment 1:

Four of the developed jar reactors were used in this experiment and denoted as EXP1. Each is loaded with 2.3 Kg feedstock reactors used at variable PHC contents (i.e. 100%, 70%, 60% and 50% , and denoted EX1R1, EX1R2, EX1R3, and EX1R4, respectively as indicated in Table 4. The temperature condition was set to psychrophilic condition at 20 ^oC and without modifying the moisture contents of the mixture (i.e. without adding any water) after being loaded in the reactor. The setups of the EX1R1-R4 reactors are depicted in fig. 3.

Also because the PHC is not as rich in nutrition compared to the WWTS, their co-digestion would compensate the bacterial malnutrition deficiency.

Table 3: Theoretical Landfill gas generation

Yield	PHC	WWTS	Mixture 60&40%
CH_4 Wt (kg)	11.57	8.8	10.46
CO_2 Wt (kg)	35.56	41.55	37.95
CH_4 Vol (m^3)	7.32	5.56	6.61
CO_2 Vol (m^3)	8.15	9.53	8.70
CH_4 (%)	47.29	36.87	43.12
CO_2 (%)	46.65	46.65	46.65
Gen. Amt. (L Kg^{-1})	13.4	49.02	27.65

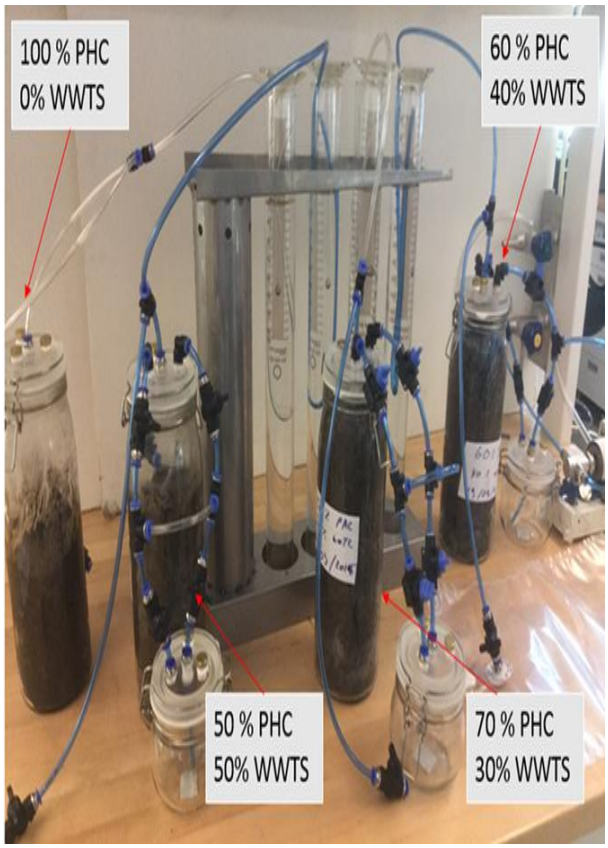

Fig. 3: Experimental reactors setup

Table 4: Experimental conditions

Experiment	Reactor No	Addition Water L	Ratio		total weight kg	Weight of PHC kg	Weight of WWTS kg	Temperature °C
			PHC %	WWTS %				
	EX1R1	0	100	0	2.3	2.3	0	20
	EX1R2	0	70	30	2.3	1.61	0.69	20
	EX1R3	0	60	40	2.3	1.38	0.92	20
No 1	EX1R4	0	50	50	2.3	1.15	1.15	20
	EX2R1	1	60	40	2.3	1.38	0.92	20
	EX2R2	0	60	40	2.3	1.38	0.92	20
	EX2R3	1	60	40	2.3	1.38	0.92	35
	EX2R4	0	60	40	2.3	1.38	0.92	35
	EX2R5	1	0	100	2.3	0	2.3	35
No 2	EX2R6	0	0	100	2.3	0	2.3	35
	EX3R1	2.3	60	40	5.3	3.2	2.1	35
No 3	EX3R2	0	60	40	5.3	3.2	2.1	35

B. **Experiment 2:**

This experimental setup is denoted as EX2R1 through EX2R4. In this set of experiments and following the observation on maximum SMY of the previous set, the mixture fraction was set at 60% weight of PHC to 40% weight of WWTS. The temperature and additional moisture are varied. Additionally, two control reactors at 100% WWTS with and without additional water denoted as EX2R5 and EX2R6 is also used which were both kept at 35 °C. Table 4 summarizes EXP2 conditions for each of the six reactors. Two different conditions are pursued psychrophilic at 20°C and mesophilic at 35°C. Each temperature condition has two reactors setups with and without adding extra water and are denoted, respectively EX2R1 (20°C and water), EX2R1 (20°C and no water), EX2R3 (35°C and water), and EX2R4 (35°C and no water). To keep the reactors at the stipulated mesophilic temperature condition and to avoid lower laboratory temperature, the sealed reactors R3 through R6, were immersed in controlled and well-stirred thermal water bath that set at 35°C. The schematics and the actual EXR1-R5 reactors setups are shown in Figs 4a and 4b .

Due to low water contents in the PHC and WWTS a fixed amount of water was added. Therefore, reactor EXP2R1, EXP2R3, and EXP2R5 were subsidized with an additional of one liter of distilled water. Reactors EXP2R1, EXP2R2 maintained at psychrophilic 20°C condition whereas rectors EXP2R3, EXP2R4, EX2R5 and

EXP2R6 were immersed in the 35°C water bath.

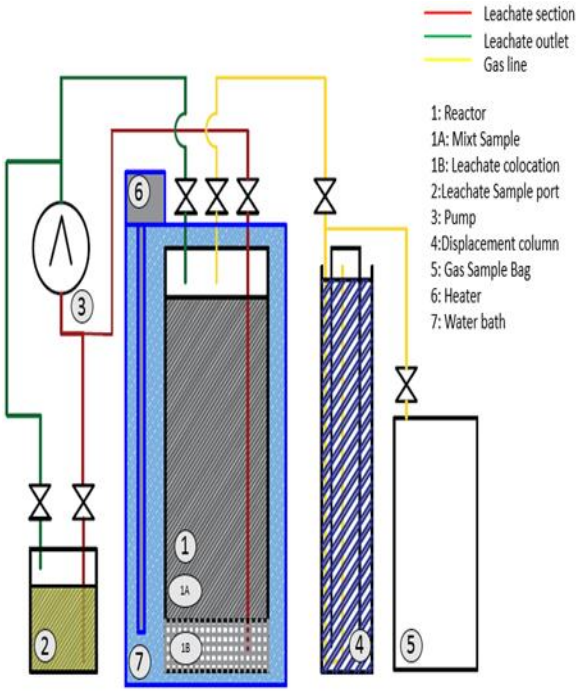

Fig. 4a: Schematic set up of the bench scale bioreactor of EXP2 under well-controlled temp conditions

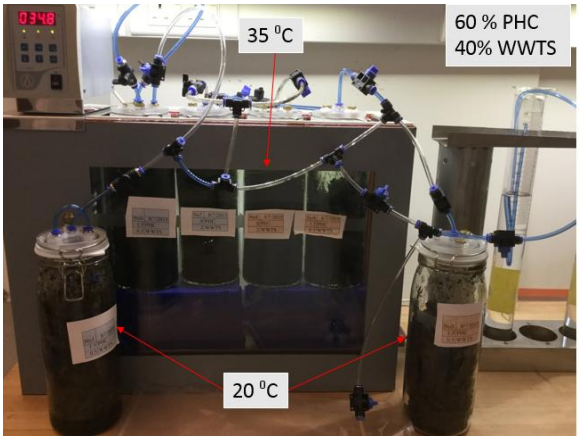

Fig. 4b: Experimental set up of the bench scale bioreactor of EX2 under well-controlled temp conditions

C. Experiment 3:

This experimental set up is consists of the two larger capacity reactors setup and denoted as EX3R1 and EX3R2. Each accommodates 5.3kg of total mixture feedstock and carried out at near mesophilic conditions at temperature of 35°C. It deployed the larger laboratory scale as depicted in Fig. 8 and placed in the building corridor that maintained between 34-36 °C. It also conducted at 60% weight of PHC to that of 40% weight of WWTS mixture in line with the maximum LFG generation proportion in EX1. The only variation between EX3R1 and EX3R2 is the additional of 2.3L of water in EX3R2 reactor. Therefore, EX3R2 was subsidized with 2.3 L of distilled. Both reactors were kept at mesophilic 35°C condition. The setup of the two reactors of EX3 is depicted in Fig. 5.

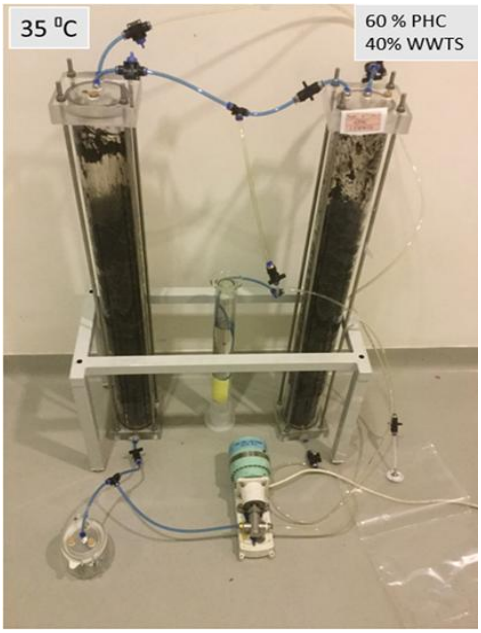

Fig. 5: EX3R1 and EX3R2 reactor setup with larger capacity at the mesophilic condition

V. RESULTS

A. Gas production

Experimental results of the LFG yield was continuously monitored and recorded on

daily basis. The weekly accumulation of LFG of EX1 for the four reactors R1-R4 is depicted in Fig. 6. The cultivation is kept for ten weeks where in the last two weeks the reactors observed unnoticeable production. As it can be seen in Fig 6, LFG generation marks the highest for the 60% PHC and 40% WWTS weight ratio reactor in EXP1R3 and reaching as high as 120ml. The PHC weight at 70% (EX1R2) and 50% (EX1R4) reactors led to a lower amount of LFG gas production reaching 61ml, and 32ml. It was observed also that almost zero LFG generation by the 100%PHW reactor in EX1R1 over the entire ten weeks period at this temperature condition and provided moisture.

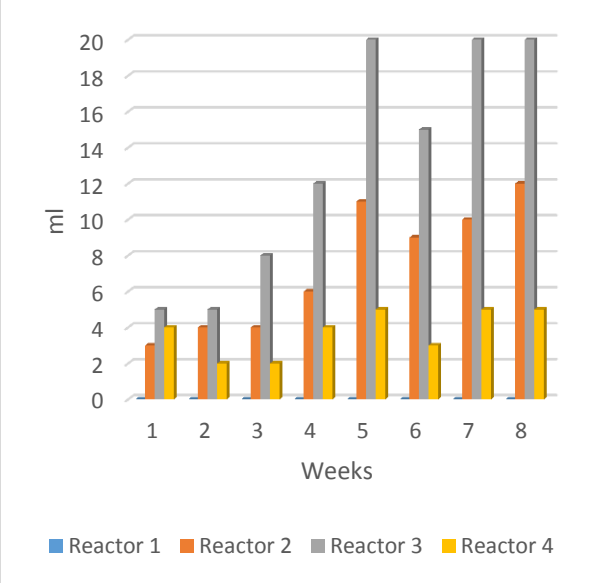

Fig. 6: Gas production EX1R1 through R4

In EX2R1 through R6 a longer degradation period was monitored lasting fifteen weeks. A quick decline in the LGF generation in the last three weeks was also evident. Figs. 7 and 8 show the accumulative weekly production of the LFG over twelve week's period.

Increasing the temperature to Mesophilic (35 ^{o}C) in reactors EX2R3 through EX2R6 appears to promote higher biodegradability than psychrophilic conditions (20 ^{o}C) in reactors EX2R1 and EX2R2. Furthermore, the addition of water in reactors EX2R1, EX2R3, and EX2R3 led to further increase in the LFG production. The reactor EX2R2 at Psychrophilic and without the addition of water showed the least LFG (165ml) generation as seen in Fig. 7.

In the control reactors at 100% WWTS (EX2R5 and R6) the LFG yield was over an order of magnitude higher that for the 60%PHC mixture reactors as depicted in Fig. 8. It totals 2500ml and 2740ml for with and without the additional water respectively. Its average weekly production is near 220-235 ml before one starts to observe a drastic decline in LFG yield beyond the 13th week. The additional of water seems to slightly undermine the LFG yield. This is because WWTS is inherently rich in moisture contents (~50%) compared to PHC waste that only contains ~3% by weight as was evaluated in proximate analysis (table 1).

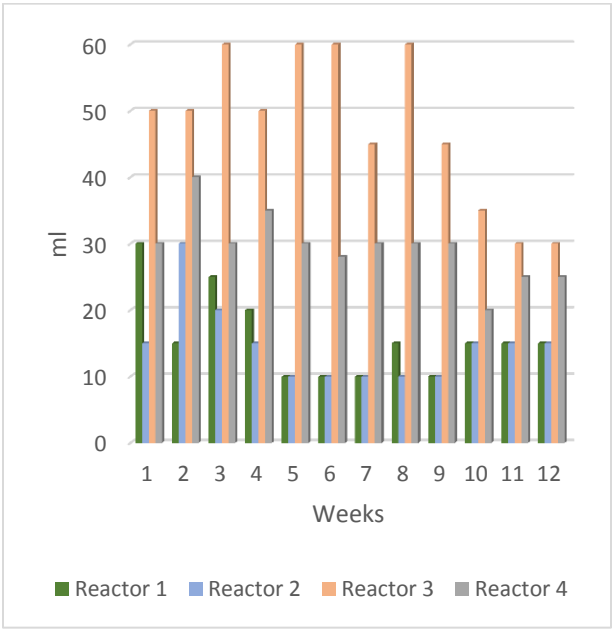

Fig. 7: Gas production EX2R1 through R4

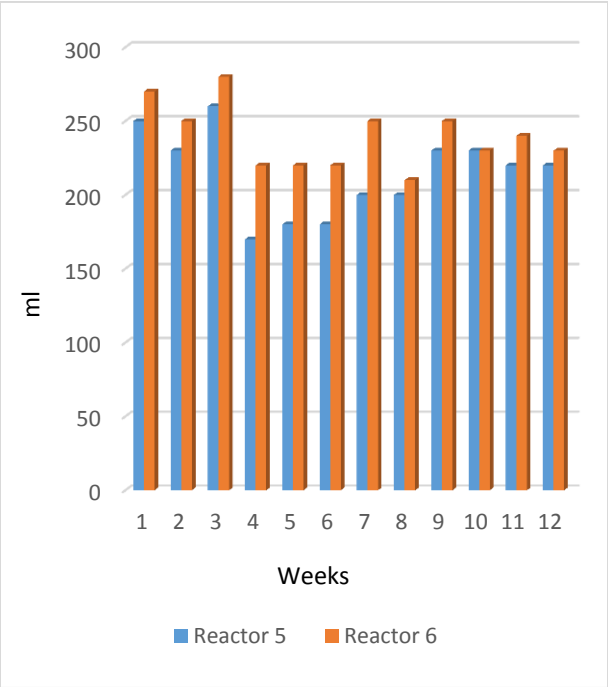

Fig. 8: Gas production in control experiments of EX2R5 and EX2R6

The accumulative weekly LFG production of EX3 for R1 and R2 reactors is depicted in Fig. 9. The incubation period also lasted over 15 weeks beyond which the LFG yield starts to show a steep decline. These reactors are at a larger capacity, but with similar conditions of EX2R3 and EX2R4 (60% PHC, 35 ^{o}C) and corresponding, respectively to additional and without water. The average weekly production of LFG is nearly 150-160ml (or 29ml/kg) which higher than what was observed in the smaller reactors of EX2R3 (47ml or 20ml/kg) and EX2R4 (28ml or 12ml/kg). This clearly suggesting higher capacity reactor propel more production of the LFG. This is in line with the observation of Saady et al. [17] who indicated that larger reactor provides more room for bacteria eutrophication and for the gas to be released creating more porosity and less compaction. The influence of the water addition however is not clear as it changes from week to week with more favorable contribution in the 2nd and 4th through the 7th week. This may be attributed to the low adsorption of the PHC to the water as after the 6th to 7th week the added water starts to settle at the bottom of the jar. It is expected however, that using the leachate circulating system will remedy this inconsistency in LFG production. The slight decrease with the water addition can be explained by the depletion of the biodegradable volatile that converted earlier.

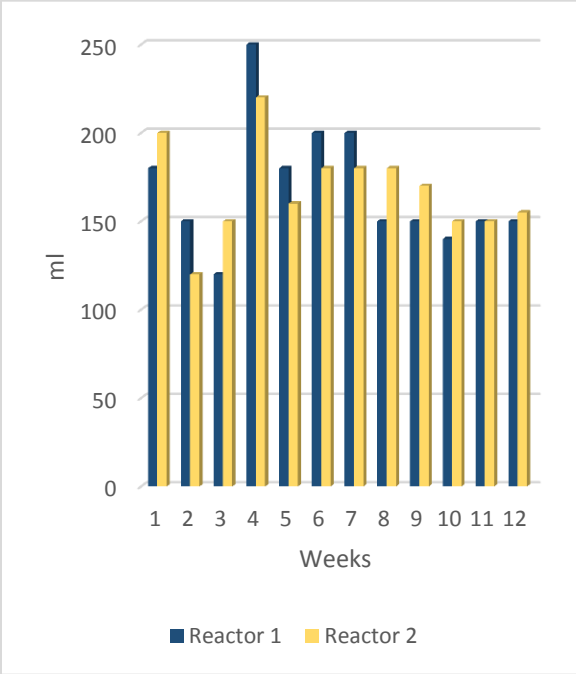

Fig. 9: Gas production EX3R1 and EX3R2

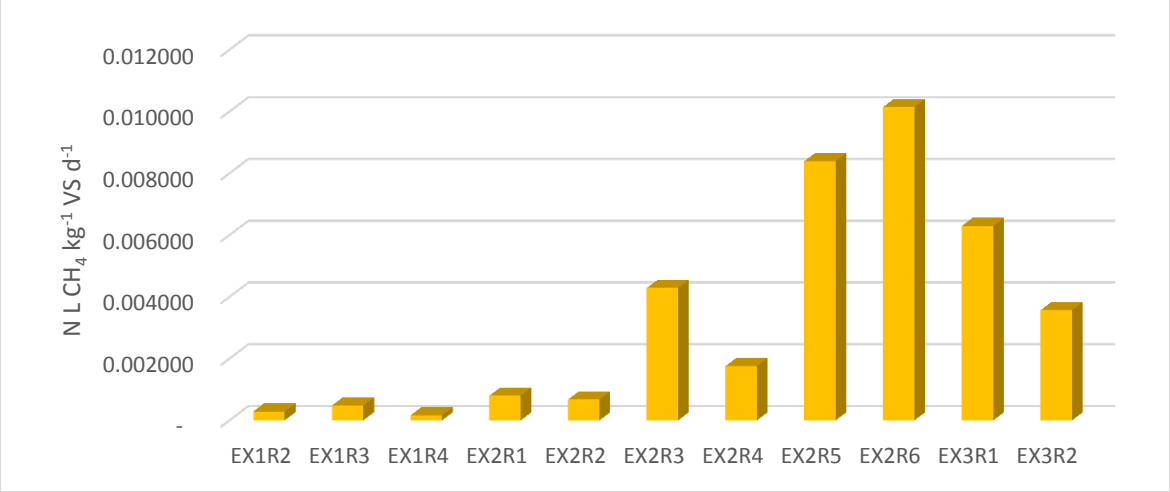

Fig. 10: Specific methane yield

B. Gas analyzer

The Gasboard-3100P was used to analyze the four main gases, i.e. CH_4, CO_2, O_2 and H_2S present in the LFG Teflon bags gas samples. These samples are obtained from the corresponding displacement column setup for each reactor. EX1 samples are retrieved in the 12th week whereas EX2 and EX3 are retrieved bi-weekly. The results are summarized in table 5. A part of EX1R1, all experiment produced CH_4 and at different molar fraction. The reactor in mesophilic (~35 ^{o}C) and with water addition is the resulted in the highest CH_4 molar fraction whereas the psychrophilic reactors conditions (~ 20 ^{o}C) led to the lowest methane production. In general good yield distribution vary between 50-60% CH4 and complemented by CO_2 molar fraction. This value, however, did not attained for the 60%PHW in all the tested variation. On the other hand the control reactors of EX2R5 and R6 inculpated at 100%WWTS reached 54% CH_4, and 25% CO_2 with water addition and 58% CH_4 and 39.6% CO_2 and without water addition. The larger reactor of EX3 resulted in substantial CH4 molar fraction of 55% and 32% for the additional water and without water reactors, respectively. However both reported traces of CO_2 molar fraction.

C. PHC Disintegration

To assess the biodegradation that took place inside of these reactors comparative TGA study for the pristine and converted feedstock of the reactor is analyzed. The latter samples are retrieved from the reactors following their incubation period. These results are summarized in table 6 and presented on dry basis implying water initially is dried out from the samples. The results are clearly show a decrease in the volatile fraction of 27-43%. This demonstrates the incurred biodegrading in the volatile fraction of PHC with zero biodegradability in the fixed carbon proportion. Pre-treatment of the feedstock to access more degradable volatile has been the subject of much research for biomass and other waste feedstock including Municipal solid waste. This can be achieved via steam, microwave, or ultrasound which all add another energy penalty to the conversion

process. In short, EX2R3 had 42.96 % volatiles disintegration in 24 weeks whereas EX2R2 had less volatiles disintegration in 24 weeks 27.2%. Fig. 11 plots the PHC disintegration curve in comparison of the pristine reference sample.

Table 5: Summary of gas analysis for the four main species

Experiment	Reactor	CH4	CO2	O2	H2S
1	No 3-4	21.46%	0.16%	0.06%	17 PPM
2	No 1	32.47%	0.02%	0.43%	1 ppm
	No 2	30.18%	0.03%	0.82%	1 ppm
	No 3	57.56%	0.09%	0.13%	89 ppm
	No 4	38.45%	0.04%	0.02%	14 ppm
	No 5	58.05%	39.6%	0.02%	8580 ppm
	No 6	58.05%	39.6%	0.02%	8580 ppm
3	No 1	55.47%	0.03%	0.03%	245 ppm
	No 2	31.51%	0.04%	0.25%	0 ppm

Table 6: Proximate analysis Experiment 2 after 24 weeks

	Reference	EX2R1	EX2R2	EX2R3	EX2R4
Volatiles	23.16	16.16	16.86	13.21	15.27
Volatiles disintegration	-	30.22 %	27.2 %	42.96 %	34.07 %
Fixed carbon	24.47	29.52	27.69	31.18	30.15
Ash	52.37	54.22	55.45	55.61	54.20

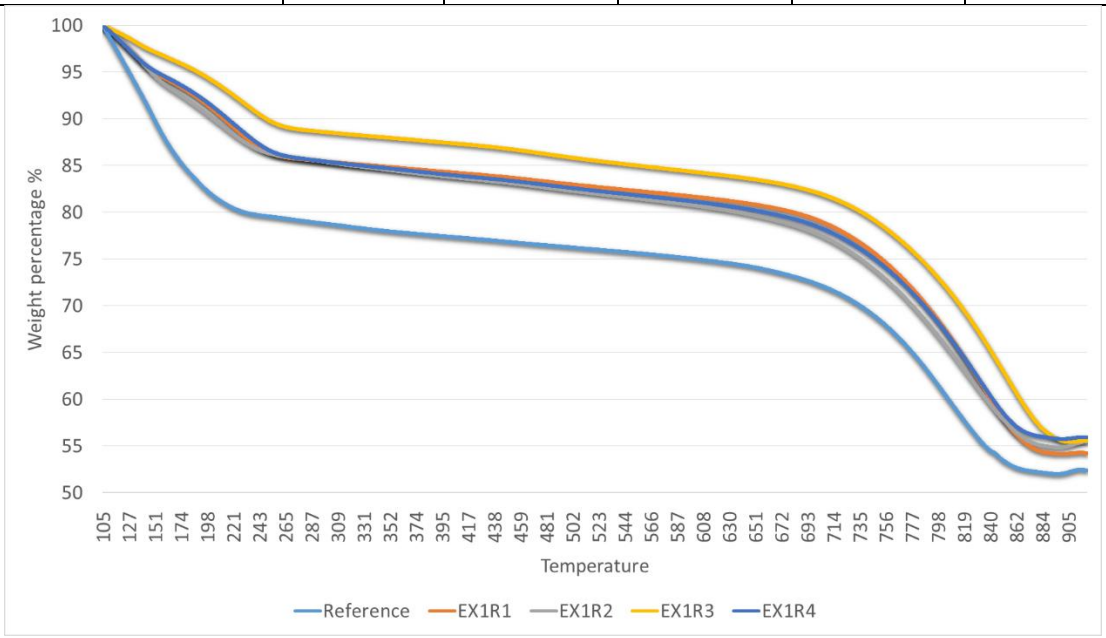

Fig. 2: TGA plot of the pristine PHC sample and the bio-degraded samples of EX2R1 through EX2R4

VI. CONCLUSION

Biodegradation of Petroleum Hydrocarbon Sluge that generated from oil and gas at substantial quantity is subject to anaerobic digestion in this analysis. Estimation of the landfill gas of PHC waste digestion is evaluated at different temperature, moisture condition, WWTS co-digestion proportion, and in both small and large reactors. While the control reactor at 100% WWTS (co-digestant) and 35 ^{o}C produced the desired CH_4 and CO_2 proportion of molar fraction, neither the 100% digestion of PHW nor the co-digestion of PHC and WWTS led to the desired CH_4 yield. The best results were obtained in the larger 5.3 kg reactor, at 35 ^{o}C temperature and with the additional of 2.3 liters of water at 60%PHC to 40% WWTS. At this condition the yield was 55.5% CH_4 molar fraction, only traces of CO_2, and SMY of 0.21 (N L CH_4 kg-1).

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