Biohydrogen production via reforming of anaerobic Digestion Biogas

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Abstract: Hydrogen (H_2) can be produced through different pathways including steam reforming of natural gas, gasification of coal, and electrolysis of water. Perhaps a more sustainable pathway is Bio H_2 which can be produced by biophotolysis of water, photo fermentation and dark fermentation of organic maters (OM). However, these routes have shortcomings which can be either high specific energy requirement or process sluggishness or both. To overcome these shortcomings, bio H_2 is produced by tapping into the biogas of the anaerobic digestion process – a rich CH_4 source. In this work, we undertake the reforming modeling of the biogas considering two different anaerobic digestion sources, i.e. landfill and wastewater sludge anaerobic digester, and benchmark the analysis against natural gas reforming. Process metrics such as conversion percentage as well as thermal process efficiency will be delineated and compared. A equilibrium based model is developed by considering several reaction constants, elemental mass balance and formation and sensible energy balance. Results shows that methane concentration has the most pronounced influence on the produced hydrogen and the overall reforming efficiency. While these values for natural gas are respectively around 0.5 and 75% for H_2 mole fraction and reforming efficiency, they only reach 0.3 for H_2 molar fraction and reforming efficiency near 36%. Therefore, while this work states the technical feasibility of reforming the biogas stream, its drawback is the attained low efficiency that one needs to consider and find ways to improve it.

Keywords: Bio Hydrogen; Methane Reforming; Reforming Efficiency; Methane Conversion

1. Introduction:

The biological pathway to produce H_2 and CH_4 shares similarities. They both consist of four generation steps but are dominated by different microbial groups, which give rise to different end products. A preliminary major challenge in the utilization of hydrogen is its sustainable production. Current technology to produce hydrogen includes steam reforming of natural gas, gasification of coal, electrolysis of water and steam reforming of CH_4. These technologies involve a significant amount of energy for generating the required heat. Recently, studies have reported a low production cost of energy through dark fermentation. The hydrogen produced through biological route, termed bio hydrogen (bio H_2), is viewed as a low energy solution and taps into organic waste source. Common routes for bio H_2 production includes biophotolysis of water, photo fermentation and dark fermentation of OM, in which fermentation has the least technological complexity but produces comparably high yields (Ntaikou et al., 2010). Fig. 1 demonstrates the biological pathways for bio H_2 production.

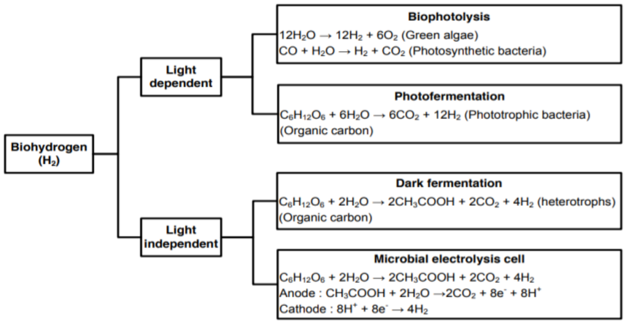


Fig. 1. The four major pathways to produce biohydrogen.

Fermentation or anaerobic digestion (AD) is a complicated and dynamic biological process which involves multiple physicochemical and biochemical reactions in sequential and parallel pathways. The AD process is governed by different microbes with varied specific cell growth rates, substrate consumption capabilities and preferred environmental conditions, such as pH and temperature. This complexity renders the sensitivity of the AD to changes in environmental conditions and, thus, parameters will need to be carefully monitored to prevent process failure. Generally, AD is characterized by four distinct phases, namely hydrolysis, acidogenesis, acetogenesis and methanogenesis as shown in Fig. 4. Some researchers have included a disintegration step before these four phases (Pontoni et al., 2015) and some categorized it into three phases, which are fermentation, acetogenesis and methanogenesis (Molino et al., 2013). Hydrolysis of carbohydrates, protein and lipid has a theoretical methane gas (CH_4) yield of 415 L CH_4/ kg VS, 496 L CH_4/ kg VS and 1014 L CH_4/ kg VS respectively (Moller et al., 2004).

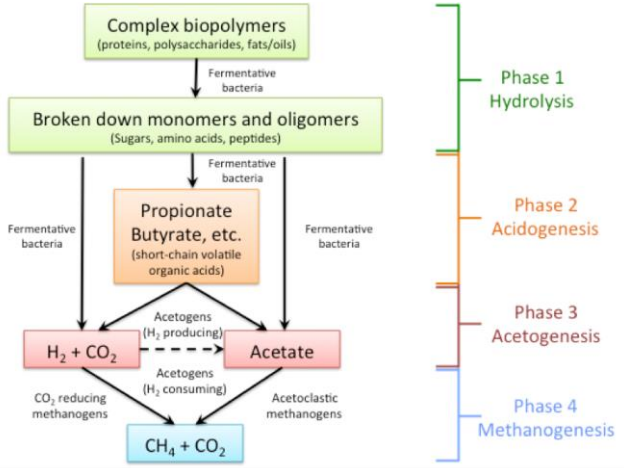


Fig. 2. The four major steps during the AD of complex organic substrates

A balanced anaerobic digestion process demands that in successive stages the rates of degradation must be equal in size. If the first degradation step runs too fast, the acid concentration rises, and the pH drops below 7.0 which inhibits the methanogenic bacteria. If the second phase runs too fast, methane production is limited by the hydrolytic stage. Thus, the rate-limiting step depends on the compounds of the substrate which is used for biogas production. Undissolved compounds like cellulose, proteins, or fats are cracked slowly into monomers within several days whereas the hydrolysis of soluble carbohydrates takes place within few hours. Therefore, the process design must be well adapted to the substrate properties for achieving complete degradation without process failure. Methane production from organic substrates mainly depends on their content of substances that can be degraded to yield CH_4 and CO_2 as main components. Composition and biodegradability are key factors for the methane yield from energy crops and animal manures. Crude protein, crude fat, crude fibre, cellulose, hemi-cellulose, starch and sugar markedly influence methane formation (Amon et al., 2002b, 2003, 2004a; Balsari et al., 1983).

Biogas from sewage digesters usually contains 55% to 65% methane and 35% to 45% carbon dioxide and <1% nitrogen by volume; biogas from organic waste digesters usually contains 60% to 70% methane, 30% to 40% carbon dioxide and <1% nitrogen while in landfills methane content is usually 45% to 55%, carbon dioxide 30% to 40% and nitrogen 5% to 15% [Jönsson O, et al 2013]. Typically, biogas also contains hydrogen sulphide and other sulphur compounds such as siloxanes and aromatic and halogenated compounds. The amount of methane and carbon dioxide in different biogases are found in previous studies in similar plants (Table 1). S. Rasi el al 2007 measured methane, carbon dioxide, oxygen, nitrogen, volatile organic compounds (VOCs) and sulphur compounds in biogas samples from a landfill, sewage treatment plant sludge digester and farm biogas plant. Methane content ranged from 48% to 65%, carbon dioxide from 36% to 41% and nitrogen from <1% to 17%. Oxygen content in all three gases was <1%. The highest methane content occurred in the gas from the sewage digester while the lowest methane and highest nitrogen contents were found in the landfill gas during winter.

Table 1. Methane, Carbon dioxide, nitrogen, hydrogen sulphide, benzene and toluene contents in biogas from different biogas producing plants

Biogas	CH_4 (%)	CO_2 (%)	O_2 (%)	N_2 (%)	H_2S (ppm)	Benzene(mgm^{-3})	Toluene(mgm^{-3})	Ref.
Landfill	47-57	37-41	<1	<1-17	36-115	0.6-2.3	1.7-5.1	S. Rasi et al. 2007
Sewage Digester	61-65	36-38	<1	<2	b.d.	0.1-0.3	2.8-11.8	S. Rasi et al. 2007
From Biogas Plant	55-58	37-38	<1	<1-2	32-169	0.7-1.3	0.2-0.7	S. Rasi et al. 2007
Landfill	59.4-67.9	29.9-38.6	n.a.	n.a.	15.1-427.5	21.7-35.6	83.3-171.6	Shin H-C et al. 2002
Landfill	37-62	24-29	<1	n.a.	n.a.	<0.1-7	10-287	Allen MR et al. 1997
Landfill	55.6	37.14	0.99	n.a.	n.a.	3.0	55.7	Eklund B et al. 1998
Landfill	44	40.1	2.6	13.2	250	n.a.	65.9	Jaffrin A et al 2003
Sewage digester	57.8	38.6	0	3.7	62.9	n.a.	n.a.	Spiegel RJ, Preston JL 2003
Sewage digester	62.6	37.4	n.a.	n.q.	n.a.	n.a.	n.a.	Stern SA et al 1998
Sewage digester	58	33.9	0	8.1	24.1	n.a.	n.a.	Spiegel RJ, Preston JL 2000

b.d. – Below detection limit 0.1 ppm ; n.a.- not analyzed

Pre-treatment methods such as acid, microwave and sonication have shown to improve the digestibility of organic waste through the disintegration of large particles and increase in its solubility. Zainal et al. (2018) has reported a H_2 yield of 28.47 mL H_2 / g COD removed where raw palm oil mill effluent (POME) and palm oil mill effluent sludge were used as a feed and inoculum respectively. The study showed that effective H_2 production could be achieved at a lower substrate concentration, with an optimum retention time of 8 h under thermophilic condition (50 °C). The impact of microwave (MW) irradiation of mixed sludge (MS) on biogas production has been investigated by Elagroudy and ElGohary (2013). Mixed sludge samples microwaved at the same temperature and at different MW intensities, consequently different exposure times, achieved almost equal degree of solubility, as expressed in terms of COD soluble/ COD total and VSS. In another study, ozonation was used as pre-treatment and showed an increase of 20 % of bio H_2 production and 25 % of COD removal than the raw POME, recording a maximum yield of 77.1 mL/ g COD (Tanikkul and Pisutpisal 2014). Budiman and Wu (2016) demonstrated that bio H_2 production at mesophilic temperature could be increased up to 8.72 mL H_2 / mL medium, with a 36.9 % of COD removal, at an amplitude of 30-90 % and ultrasonic duration of 5-60 min. The bio H_2 production was increased by 50 % compared to the untreated raw palm oil mill. Low frequency ultrasonication for 16.20 min as a pre-treatment for POME resulted in 16.10 % OM solubilization and an increase of soluble COD from 29,000 mg/L to 31,675 mg/L (Wong et al., 2018).

To the best of the authors' knowledge, reforming of biogas that considers the variation in the composition and its impact on the reforming metrics has not been done. This work fills this literature gap and undertakes the reforming modeling of biogas considering two different anaerobic digesting sources, i.e. landfill and wastewater sludge digester and benchmarks the analysis against natural gas reforming. Process metrics such as conversion percentage as well as thermal process efficiency will be delineated and compared. The model is based on equilibrium by considering three reaction constants, elemental mass balance and both chemical/formation and thermal/sensible energy balance.

2. Theoretical and modelling setup:

These models apply elemental balance, heat balance, and the equilibrium reaction constants equation for the CO shift and steam reforming reaction. The reaction constant method is applied versus the elemental potential due to the fact that it is simpler to code and more appealing to understand in determining the equilibrium species concentrations and the temperature and pressure products conditions [18]. The main assumption is that the process takes infinite residence time and occurs under chemical and thermodynamic equilibrium [25,26]. For that reason, reaction kinetics are neglected, and no intermediate species are accounted for as the models lack the concept of time, mixing and geometry to provide special distribution of species. Nevertheless, the reformer/reactor metrics such as the specific mole fraction of H_2 and conversion efficiency under "best" case scenario is evaluated. Practically, reforming is subjected to heat losses, kinetic limitations, flow and pressure drop losses due to the geometry as well as localized turbulence. These can result in variation in spatial and temporal species distribution, that, in general, yields lower efficiencies than the equilibrium assumption.

Reforming of biofuel is a series of homogeneous reactions and involves many species and their intermediates. Nevertheless, a summary of the main reforming reactions is given in Table 2. The quest is to determine the molar or mass fraction of each of H_2, CH_4, H_2O, CO, CO_2, and N_2 and the ratio of the feed streams as well as the required process heat over a sweeping range of operational temperatures (as well as pressures). Based on the evaluated and captured trend, the optimal operation condition can be identified. In the reforming, two feeds streams are considered, feed I and II. Feed I is represented by the methane rich stream whereas feed II is represented by the steam. Feed 1 can be a pure CH_4 or combination of CH_4 rich species as in the case of natural gas (CO, H_2, C_2H_4, C_3H_6, and traces of H_2) and the outcome of the digestion process (CH_4 and CO_2).

Table 2. Gasification reaction of the main species and their corresponding heat of reactions

Reaction#	Reaction Stoichiometry	Reaction energy (kJ/mol)	Description
R1	$CH_4 + H_2O \rightarrow CO + 3H_2$	$+206$	Methane steam reforming I
R2	$CO + H_2O \rightarrow CO_2 + H_2$	-41	CO Shift
R3	$CH_4 + 2H_2O \rightarrow CO_2 + 4H_2$	$+165$	Methane steam reforming II

Therefore, a total of 8 unknowns are generated governed by 8 equations and these are the *4* elemental balance of each of C, O, H, and N, the (*one*) total heat balance, the *three* equilibrium reaction of Steam Reforming (R1), CO-shift (R2) and Steam Reforming II (R3). Each of the above reaction equations is independent, and has an associated equilibrium equation in terms of the concentration K_c (or the partial pressure K_p) as follows:

$$K_c(T) = \frac{[C]^c[D]^d}{[A]^a[B]^b} \tag{1}$$

Where [X] is the molar concentration of species *X* and K_c is the Arrhenius rate and is written as:

$$k_c(T) = A_r T^{\beta_r} e^{-\frac{E_r}{RT}} \tag{2}$$

Where *A* is the pre-exponent constant, β is temperature exponent, E is the activation energy and is quoted to the Gibbs free energy of the reaction r, R is the universal gas constant (8.313kJ/kmol. K), *T* is the reaction absolute temperature. The steady form of the energy equation is written as:

$$\sum_{i=1}^{n\,product} \dot{n}_i\, h_i = \sum_{i=1}^{n\,react} \dot{n}_i\, h_i + \dot{Q} \tag{3}$$

Where *n* is the number of moles and the "*dot*" indicates time rate. Here *h* is the enthalpy term and includes the formation and sensible enthalpies. The 8 equations that cover the *six* species $(\chi_{H_2}, \chi_{CH_4}, \chi_{CO_2}, \chi_{CO}, \chi_{N_2}, \chi_{H_2O})$, the steam ratio per the methane feed, and process heat can be solved iteratively. The feedstock is defined according to its components composition additional to its lower heating value. The analyses are carried at a fixed pressure of 30 bars and at sweeping values of temperature between 650°C and 1250°C. Additional to species evaluation, the conversion and reforming efficiencies can be evaluated, without accounting to any of the product sensible heat. The conversion efficiency is defined as the ratio of the remaining CH_4 mass to the feed CH_4 mass while the reforming/thermal efficiency is the heating value of the resulting H_2 to the feed stream heating value, additional to any extra process heat.

3. Preliminary Results:

In these analyses, the reforming of pure methane and natural gas is conducted first to validate the model as far as the species yield and conversion metrics. The natural gas is represented by a mixture stream that consists of eight common species including CO_2, H_2, CH_4, N_2, C_2H_6, C_3H_8, C_4H_{10}, C_5H_{12} and per their concentrations given in Table 3.

Table 3: Natural gas composition and concentrations considered in this work

Species	CO_2	CO	H_2	CH_4	N_2	C_2H_6	C_3H_8	C_4H_{10}	C_5H_{12}
Molar Concentration	0.015	0	0.0642	0.7877	0.0379	0.0709	0.0166	0.0016	0.0379

There is plenty of literature dedicated to these specific streams and their results, rendering good fidelity of the model. The notion of the reactor is a continuous and open system type with two feed streams, i.e. the methane source and the steam, entering at specified temperature and pressure into the fixed temperature and pressure (28.5 bars baseline) reactor environment. Parametric study can be considered by varying any of the conditions of the feed streams or the reactor environment. Results of the molar concentrations of the six species are presented in Fig. 3 when fixing the feed stream condition at 540°C and 31 bars and the reactor pressure at 28.5 bar while sweeping on the reactor temperature from 600 °C to 1250 °C. Reactions R1 and R3 are strongly endothermic and according to Le Chatelier's principle, they favor the forward reaction and the production of H_2, CO and CO_2. Reaction R2, however, is mildly exothermic and hence the same principle favors the reverse reaction, penalizing CO_2 and H_2 production and favoring the production of CO. These trends are well presented in Fig.3 such that CO and H_2 continue to increase while CO_2 increases followed with a decrease. Overall, as the temperature is increased, the heat content of the system decreases, so the system would consume some of that heat by shifting the equilibrium to the right, thereby producing more H_2 and CO and consuming both CH_4 and H_2O. More H_2 would be produced if the reaction was run at a higher temperature. However, higher temperature also implies more heat added to the system and practically the temperature is set at a compromise value between the maximum desired CH_4 conversion and a reasonable H_2 production rate, which also determines the thermal process efficiency, before becoming unfavorable. As the overall reaction is endothermic, increase in temperature increases the K value. It should also be noted that the influence of increasing either stream (methane source or steam) concentration above process stoichiometry would favor the forward reaction, i.e. the production of H_2 and consumption of the CH_4 and H_2O reactants. The increase of the reactor pressure, however, is unfavorable according the same principle as reactions R1 and R3 would proceed backward and towards the lower molar to accommodate higher pressure. The R2 reaction remains insensitive to changes in the process pressure. The best conversion occurs near 1000°C as per Table 4 that results in a molar fraction of H_2 =0.511977 and residual fraction of CH_4 =0.004039 for the pure methane, but slightly lower for natural gas with H_2 = 0.50452 and CH_4 =0.003976. The attained efficiencies are 75.3% and 72.3% in favor of the pure methane stream. Nevertheless, the conversion was slightly better for natural gas reaching 99% compared to 97.5% for pure CH_4. The H_2 molar concentration is near 0.5 fraction and the other fractions is headed by the remaining steam near 0.32, CO near 0.11, and CO_2 near 0.04 and the balance is traces of unconverted methane.

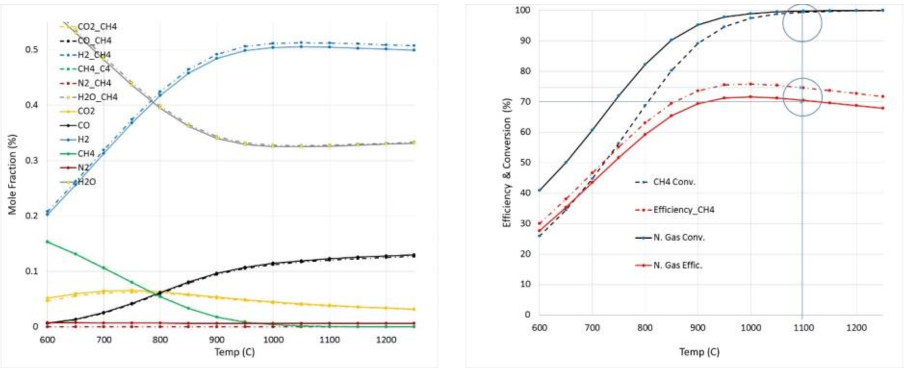


Fig. 3. Results of reforming pure methane and natural gas, L) species concentration, R) process metrics

Table 4: Species molar fractions and reforming thermal efficiency for methane and natural gas reforming

Reformed Stream	**CO_2**	**CO**	**H_2**	**CH_4**	**N_2**	**H_2O**	**Heat (MJ)**	**Effi. (%)**	**Conv. (%)**
Pure CH_4	0.043673	0.112428	0.511977	0.004039	na*	0.327882	215.141131	75.32633	97.48058
Nat. Gas	0.044963	0.114848	0.50452	0.003976	0.006046	0.325647	211.231063	72.08308	98.93861

*na not present

After establishing these baseline analyses, the anaerobic digestion streams are considered, and the conversion metrics are evaluated to assess the influence of the variation of the input stream composition. The results for molar fractions and process metrics are depicted in Fig. 4. These are also summarized in Table 6 at the best CH_4 conversion and the attained reforming efficiency. The trend with respect to temperature is well preserved but at higher yield of CO_2 due to its presence in the incoming stream. The exothermic R2 reaction also has stronger presence toward contribution of CO and reproduction of H_2O as depicted in Fig. 4. The landfill and anaerobic digester streams reforming efficiency however was at much lower value than the conventional stream (pure methane or natural gas) and they marked only 27% and 42% respectively when 99% methane conversion is sought. It should be noted that the composition of these streams is taken from Table 1 at average values as listed in Table 5. The lower attained efficiency is clearly read from the lower concentration of H_2 which marks near 0.3 compared to 0.5 in the case of the conventional streams.

Table 5: Composition of Biogas from Landfill and Anaerobic digester considered in this study

Species	**CO_2**	**CH_4**	**N_2**
Landfill	0.4	0.5	0.1
Anaerobic Digester	0.375	0.6	0.025

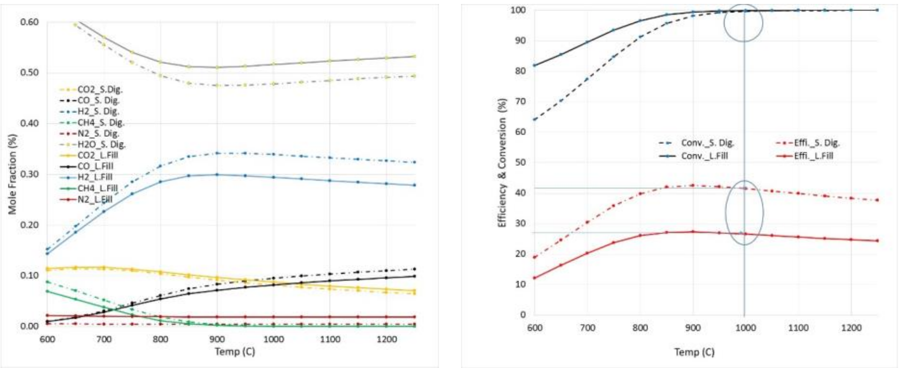


Fig. 4. Reforming of landfill and sewage digester biogas, L) species concentration and R) process metrics

Table 6: Reforming species molar fractions and thermal efficiency under biogas landfill and anaerobic digester streams

Species	CO_2	CO	H_2	CH_4	N_2	H_2O	Power	Heat (MJ)	Efficiency	Conversion
Landfill	0.088	0.082	0.294	0.000	0.019	0.517	3.672	140.883	26.566	99.894

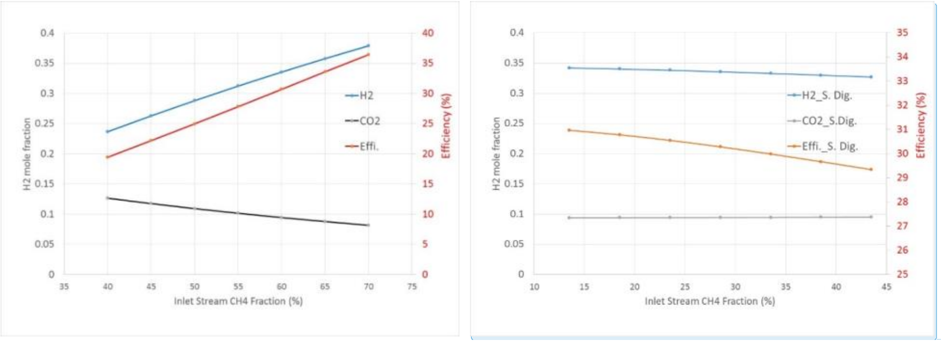
Anaerobic digester	0.082	0.095	0.339	0.001	0.005	0.479	3.808	157.278	41.514	99.672

To better assess reforming of the biogas source, a sensitivity study of increasing CH_4 fraction (thereby decreasing fraction of CO_2) at constant N_2 fraction is considered. Furthermore, a sensitivity study of the pressure is also carried out by varying the pressure from baseline values. Table 7 summarizes the values considered in these two sensitivity studies.

Table 7: Landfill and Anaerobic digester composition and concentrations considered in this work

Variable	**CO_2**	**CH_4**	**N_2**	**Pressure (bar)**	**Variable**	**Pressure(bar)**	**CO_2**	**CH_4**	**N_2**
Biogas concentration	0.3	0.69	0.01	28.5	Process Pressure	13.5	0.4	0.59	0.01
	0.35	0.64	0.01	28.5		18.5	0.4	0.59	0.01
	0.4	**0.59**	**0.01**	**28.5**		23.5	0.4	0.59	0.01
	0.45	0.54	0.01	28.5		**28.5 (baseline)**	**0.4**	**0.59**	**0.01**
	0.50	0.49	0.01	28.5		33.5	0.4	0.59	0.01
	0.55	0.44	0.01	28.5		38.5	0.4	0.59	0.01
	0.60	0.39	0.01	28.5		43.5	0.4	0.59	0.01

Results of varying CH_4 concentration are depicted in Fig.5. It shows that as the molar concentration of the former is increased, the molar fraction of H_2 increases while that of CO_2 decreases, increasing the reforming efficiency. In particular, increasing CH_4 fraction from 40 to 70% resulted in a reforming efficiency increase of 17 points, climbing from 20% to nearly 37%. On the contrary, the influence of pressure is less appealing and is in the declining trend as these results are shown in Fig. 5. This is once again satisfying le Chatelier principle as reforming produces more moles on the production side than the reacting side and hence higher pressure always would favor the backward reaction as Fig. 5 attests.



Με σχόλια [OM1]: The right graph of Fig. 5 shows CH_4 fraction instead of pressure

Fig. 5. Sensitivity studies of the reforming captured in terms of H_2 and CO_2 fractions and reforming efficiency for: L) CH_4 concentration R) Process pressure

4. Conclusion:

In this work, bio H_2 production through biogas reforming is carried out. Two sources are considered the landfill and anaerobic digester biogas source. The main difference in these streams are the concentration of the CH_4. A reforming model that is based on equilibrium is developed. The model is initially validated with respect to the two conventional streams, namely natural gas and pure CH_4. The model is then used to assess the molar concentration of the hydrogen produced and reforming efficiency under different conditions including the methane concentration and reactor temperature and pressure. Results shows that methane concentration has the most pronounced influence on the produced hydrogen and, consequently, the reforming efficiency. These values are around 0.5 molar fraction for H_2 and reforming efficiency nearly 75% for conventional stream while are near 0.3 molar fraction and best reforming efficiency near 36%. Therefore, while this work states the technical feasibility of reforming the biogas stream, its drawback is the attained low efficiency that one needs to consider and find ways to improve.

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