HYDROGEN AND METHANE PRODUCTION FROM PALM OIL MILL EFFLUENT THROUGH TWO-STAGE DARK FERMENTATION

SANTHANA KRISHNAN CHANDRASEKAR

Doctor of Philosophy

UNIVERSITI MALAYSIA PAHANG

SUPERVISOR'S DECLARATION

I hereby declare that I have checked this thesis and in my opinion, this thesis is adequate in terms of scope and quality for the award of the degree of Doctor of Philosophy in Energy and Environmental Engineering.

____

(Supervisor's Signature)

Full Name : PROFESSOR DATO' DR. ZULARISAM ABDUL WAHID

Position : DEAN FACULTY OF ENGINEERING TECHNOLOGY, UMP

Date : 07/07/17

____

(1^{st} Co-Supervisor's Signature)

Full Name : PROFESSOR DATIN' DR. MIMI SAKINAH ABDUL MUNAIM

Position : DEPUTY DEAN OF IPS, UMP

Date : 07/07/17

____

(2^{nd} Co-Supervisor's Signature)

Full Name : DR. LAKHVEER SINGH

Position : SENIOR LECTURER, FTEK, UMP

Date : 07/07/17

STUDENT'S DECLARATION

I hereby declare that the work in this thesis is based on my original work except for quotations and citations which have been duly acknowledged. I also declare that it has not been previously or concurrently submitted for any other degree at Universiti Malaysia Pahang or any other institutions.

____

(Student's Signature)

Full Name : SANTHANA KRISHNAN CHANDRASEKAR

ID Number : PKC15004

Date : 07/07/17

HYDROGEN AND METHANE PRODUCTION FROM PALM OIL MILL EFFLUENT THROUGH TWO-STAGE DARK FERMENTATION

SANTHANA KRISHNAN CHANDRASEKAR

Thesis submitted in fulfillment of the requirements
for the award of the degree of
Doctor of Philosophy in Energy and Environmental Engineering

Faculty of Engineering Technology
UNIVERSITI MALAYSIA PAHANG

JULY 2017

DEDICATION

Specially dedicated to my beloved parents, lovable brothers, and sister who constantly encouraged and supported me all the way since the beginning of the studies.

ACKNOWLEDGEMENTS

First and foremost I would like to thank God. You have given me the power to believe in myself and pursue my dreams. I could never have done this without the faith I have in you, the Almighty.

I am grateful and would like to express my sincere gratitude to my supervisor, Professor Dato' Dr. Zularisam Abdul Wahid for his germinal ideas, invaluable guidance, continuous encouragement and constant support in making this research possible. He has always impressed me with his outstanding professional conduct, his strong conviction for engineering, technology and science. I appreciate his consistent support from the first day I applied to graduate program till these concluding moments. I am truly grateful for his progressive vision during my training, his tolerance of my naive mistake, and his commitment to my future career. I also sincerely thanks for the time spent proofreading and correcting my many mistakes. I am very lucky to have him as my supervisor and I believe that he is the best supervisor in the world, indeed.

I would like to express my sincere thanks, gratitude and loyalty from the very bottom of my heart to my co-supervisor Professor Datin' Dr. Mimi Sakinah Abdul Munaim. From the very beginning of my research she helped and guided me. She supported me and gave her valuable ideas, suggestions, and advices during my tough times. I am lucky and thankful to god for showing such a wonderful co-supervisor. It gives me great pleasure to acknowledge to the guidance, valuable suggestions, and constructive criticism to Dr. Lakhveer Singh Thakur. Your suggestions helped me to make right decisions and had significantly contributed to this thesis.

I acknowledge my sincere indebtedness and gratitude to my parents Chandrasekar and Banumathy for their love, dream and sacrifice throughout my life. I am also grateful to my brothers Thiyagarajan, Murali krishnan, Kamala Kannan, and sister Santhana Lakshmi for their sacrifice, patience and understanding that were inevitable to make this work possible. I cannot find the appropriate words that could properly describe my appreciation for their devotion, support and faith in my ability to attain my goals. Also I am indebted and very much thankful to my Periyama Lakshmi Kantham and her son for her constant support and encouragement.

Special thanks should be given to all my friends Mr. Habib Jilani, Dr. Nazrullah Mohammad, Dr Natanam, Mr, Mishra, Dr. Ravinder, Ms. Rini Jarial, all lab mates, all postgraduate roommates and all staff members of Faculty of Engineering Technology, Universiti Malaysia Pahang, who help me in many ways and made me stay at UMP pleasant and unforgettable. I also would like to acknowledge their comments and suggestions, which was crucial for the successful completion of this study.

TABLE OF CONTENTS

LIST OF TABLES

LIST OF FIGURES

LIST OF SYMBOLS/UNITS

Q_F	Feed flow rate
V_{up}	Up-flow velocity
H(t)	Cumulative hydrogen production (mL)
P	Hydrogen production potential (mL)
R_m	Maximum hydrogen production rate (mL/h)
l	Lag phase time (h)
t	Cultivation time (h)
$H_{(Ac)}$	Acetic acid
$H_{(Bu)}$	Butyric acid
$H_{(Pr)}$	Propionic acid
$H_{(Va)}$	Valeric acid
EtoH	Ethanol
ºC	Degree Celsius
$FeCL_2$	Ferric chloride
$MgSO_4$	Magnesium sulphate
NaOH	Sodium hydroxide
w/v	Weight to volume
m^3	Meter cube
min	Minute
g/L or gL^{-1}	Gram per litre
%	Percentage
Mol	Moles
nm	Nanometre
mg/L	Milligram per litre
$L\ H_2\ L^{-1}\ d^{-1}$	Litre hydrogen per litre per day
$L\ CH_4\ L^{-1}\ d^{-1}$	Litre methane per litre per day
ml	Millilitre
L	Litre
C:N	Carbon to nitrogen
C:P	Carbon to phosphate
Na^+	Sodium ions
K^+	Potassium ions
Na_2HPO_4	Disodium mono hydrogen phosphate
$CaCO_3$	Calcium carbonate
d	Days
h	Hours
Kg/m^3	Kilograms per cubic meter
KJ	Kilojoule
MJ	Mega joule
L/d	Litre per day
m/h	Meter per hour
$L\ H_2\ kgCOD^{-1}$	Litre hydrogen per kilograms of chemical oxygen demand
$L\ CH_4\ kgCOD^{-1}$	Litre methane per kilograms of chemical oxygen demand
$L\ H_2$/L-POME	Litre hydrogen per litre of palm oil mill effluent
$L\ CH_4$/L-POME	Litre methane per litre of palm oil mill effluent
$Kg/m^3 \cdot d$	Kilograms per meter cube per day

LIST OF ABBREVIATIONS

POME	Palm oil mill effluent
UASB	Up-flow anaerobic sludge blanket reactor
CSTR	Continuous stirred tank reactor
H_2	Hydrogen
CH_4	Methane
HPR	Hydrogen production rate
MPR	Methane production rate
HY	Hydrogen yield
MY	Methane yield
N_2	Nitrogen
CO_2	Carbon dioxide
POME	Palm oil mill effluent
HPP	Hydrogen production potential
MPP	Methane production potential
OLR	Organic loading rate
HRT	Hydraulic retention time
CCD	Central composite design
RSM	Response surface methodology
COD	Chemical oxygen demand
BOD	Biological oxygen demand
VSS	Volatile suspended solids
VS	Volatile solids
TSS	Total suspended solids
TS	Total solids
MLVSS	Mixed liquor volatile suspended solids
VFA	Volatile fatty acids
SHPR	Specific hydrogen production rate
SMPR	Specific methane production rate
DGGE	Denatured gradient gel electrophoresis
PCR	Polymerase chain reaction
Fe	Iron
DOE	Department of Environment
EIA	Energy information administration
EC	European commission
MPOB	Malaysian palm oil board
TN	Total nitrogen
CPO	Crude palm oil
TAN or NH_4^+–N	Total ammonia nitrogen
TKN	Total kjeldahl nitrogen
BLAST	Basic local alignment search tool
HPG	Hydrogen producing granules
SRT	Solid retention time
F/M	Food to microbe ratio
DO	Dissolved oxygen

HYDROGEN AND METHANE PRODUCTION FROM PALM OIL MILL EFFLUENT THROUGH TWO-STAGE DARK FERMENTATION

SANTHANA KRISHNAN CHANDRASEKAR

Thesis submitted in fulfillment of the requirements
for the award of the degree of
Doctor of Philosophy in Energy and Environmental Engineering

Faculty of Engineering Technology
UNIVERSITI MALAYSIA PAHANG

JULY 2017

ABSTRAK

Dua peringkat penapaian gelap adalah teknologi hijau yang melambangkan satu peluang yang hebat untuk pertukaran tenaga yang tinggi dan kawalan pencemaran kedua-duanyaObjektif utama kajian ini adalah untuk menyiasat dua proses peringkat gelap penapaian anaerobik untuk hidrogen (H_2) berturutan dan penghasilan metana (CH_4) menggunakan cecair buangan dari kilang minyak sawit sebagai (POME) aliran atas selimut anaerobik enapcemar-reaktor tangki teraduk berterusan (UASB-CSTR). Semasa eksperimen, H_2 telah dihasilkan dalam reaktor UASB dalam keadaan termofilik (55ºC) pada peringkat pertama, manakala CH_4 dihasilkan daripada buangan dari reaktor UASB dalam CSTR dalam keadaan mesofilik (37ºC) pada peringkat kedua. Haba terawat dan tidak terawat enapcemar anaerobik digunakan masing-masing sebagai inokulum untuk reaktor UASB dan CSTR. Pada yang pertama, ujian kumpulan telah dijalankan untuk mengetahui H_2 dan CH_4 yang berpotensi bagi pengeluaran POME. Mengikuti, reaktor UASB yang dikendalikan secara berterusan pada keadaan termofilik dengan masa tahanan hidraulik (HRT) selama 2 hari dan kadar muatan organik (OLR) sebanyak 75 $kgCODm^{3}\cdot d^{-1}$ sehari untuk penghasilan H_2. Buangan dari reaktor UASB yang selalunya mengandungi asetat dan butyrate telah dimasukkan terus ke dalam CSTR untuk penghasilan CH_4 pada suhu mesofilik dalam masa 5 hari HRT. Kadar maksimum penghasilan H_2 dan CH_4 telah dicapai masing-masing pada 1.92 L H_2 L^{-1} dan 3.2 L H_2 L^{-1} sehari. Hasil terkumpul H_2 dan CH_4 adalah masing-masing 215 L $H_2/kgCOD^{-1}$ dan 320 L $CH_4/kgCOD^{-1}$ dengan jumlah kecekapan penyingkiran COD sebanyak 94%. Granul enapcemar dari kedua-dua reaktor UASB dan CSTR dianalisis dengan menggunakan mikroskop elektron pengimbas dan komuniti mikrob dianalisis menggunakan PCR denaturasi elektroforesis gel kecerunan (PCR-DGGE). Keputusan menunjukkan kehadiran spesies *Thermoanaerobacterium* dalam UASB dan *Methanobrevibacter* spesies, dan *Methanosarcina* spesies dalam CSTR. Kajian kedua menunjukkan kesan edaran semula buangan CH_4 ke dalam reaktor UASB bagi penghasilan secara berterusan untuk H_2 dan CH_4. HPP dari POME yang bercampur dengan buangan metanogen pada kadar edaran semula 50%, 40%, 35%, 30%, 20% dan 10% dikaji. Kedua-dua hasil dan kadar penghasilan H_2 meningkat dalam sistem edaran semula buangan metanogen. Edaran semula buangan metanogen pada kadar edaran semula 35% dapat mengimbangi kealkalian yang dikehendaki oleh reaktor UASB. Hasil H_2 dan CH_4 pula masing-masing 178 mL H_2/gCOD and 412 mL CH_4/gCOD. Pengaruh kadar loading organik (OLR) juga dikaji. Pelbagai OLR seperti 25, 50, 75, 100, 125 $kgCOD/m^3$ sehari telah dianalisis bagi meningkatkan kadar penghasilan dan hasil H_2 dan CH_4. Hasil yang lebih baik telah dicapai apabila OLR berada dalam lingkungan 75 kg-COD / m^3 sehari. Kadar maksimum penghasilan H_2 adalah 175.15 mL H_2/g MLVSS sehari, manakala kandungan H_2 tertinggi dan hasil H_2 adalah masing-masing 35% and 49.22 mL H_2/g $COD_{applied.}$ Kandungan dan hasil maksimum CH_4, dan SMPR adalah 68%, 155.87 mL CH_4/g $COD_{applied}$ dan 325.13 mL CH_4/g MLVSS sehari. Kajian terhadap pengaruh kadar aliran (Q_F) dan halaju aliran atas (VUP) diantara (1.7-10.2 L/ sehari) dan (0.5-3.0 m/jam) untuk penghasilan H_2 menggunakan kaedah gerak balas permukaan (RSM) menunjukkan bahawa hasil H_2 adalah 0.32 L H_2 g^{-1} COD pada Q_F dan V_{up} of 1.7 L sehari and 0.5 m sejam. Julat optimum untuk penghasilan penapaian H_2 POME ialah Q_F = 2.1-3.7 L sehari and V_{up} =1.5-2.3 m sejam. Keputusan eksperimen bersetuju sangat dengan ramalan model.

ABSTRACT

Two-stage dark fermentation is a green technology, presents an outstanding opportunity for both high energy conversion and pollution control. The main objective of this research is to investigate two-stage dark fermentation processes for sequential hydrogen (H_2) and methane (CH_4) production using palm oil mill effluent (POME) in up-flow anaerobic sludge blanket–continuous stirred tank reactor (UASB-CSTR). During the experiment, H_2 was produced in UASB reactor at thermophilic condition (55ºC) in the first stage, while CH_4 was produced from the effluents of UASB reactor in the CSTR at mesophilic condition (37ºC) in the second stage. The heat treated and non-heat treated anaerobic sludge was used as inoculum for UASB and CSTR reactor, respectively. In the first study, batch test was conducted to find out the hydrogen production potential (HPP) and methane production potential (MPP) of POME. Following, the UASB reactor operated continuously at thermophilic conditions with the hydraulic retention time (HRT) of 2 days and organic loading rate (OLR) 75 kgCOD $m^3 \cdot d^{-1}$ for H_2 production. The effluents from UASB reactor were directly fed into CSTR for CH_4 production at mesophilic temperature with the HRT of 5 days. The maximum H_2 and CH_4 production rate achieved was 1.92 L H_2 $L^{-1}d^{-1}$ and 3.2 L CH_4 L^{-1} d^{-1}, respectively. The cumulative H_2 and CH_4 yields were 215 L H_2/kgCOD^{-1} and 320 L CH_4/kgCOD^{-1}, respectively with the total COD removal efficiency of 94%. The sludge granules from both UASB and CSTR reactor were analyzed using scanning electron microscopy and the microbial community was analyzed using polymerase chain reaction denaturing gradient gel electrophoresis (PCR-DGGE). Results revealed that sludge granules were nearly round shaped with multiple cracks on the surface and UASB and CSTR reactor was enriched with *Thermoanaerobacterium* species and *Methanobrevibacter* species, *Methanosarcina* species, repectively. The second study addressed the effect of recirculation of methane effluent into UASB reactor for the continuous H_2 and CH_4 production. HPP from POME mixed with methanogenic effluent at recirculation rate of 50%, 40%, 35%, 30%, 20% and 10% was investigated. The recirculation of methanogenic effluent at 35% recirculation rate could compensate for alkalinity required by UASB reactor. The maximum H_2 and CH_4 yield were 178 mL H_2/gCOD and 412 mL CH_4/gCOD, respectively. Two-stage process with methanogenic effluent recirculation flavoured the UASB reactor and efficiently for energy recovery from POME. In the third study, influence of different organic loading rates (OLR) such as 25, 50, 75, 100, 125 kg-COD/$m^3 \cdot$d was analyzed for the improvement of hydrogen and methane production rate and yield. The better yield was achieved when the OLR was in the range of 75 kg-COD/$m^3 \cdot$d. The maximum H_2 production rate was 175.15 mL H_2/g MLVSS·d, while the highest H_2 content and yield were 35% and 49.22 mL H_2/g $COD_{applied}$, respectively. The maximum CH_4 content, CH_4 yield, and specific methane production rate (SMPR) were 68%, 155.87 mL CH_4/g $COD_{applied}$ and 325.13 mL CH_4/g MLVSS·d, respectively. The results indicated that OLR affected H_2-CH_4 production and substrate removal efficiency. Finally, the studies on the influence of flow rate (Q_F) and up-flow velocity (V_{up}) ranging (1.7-10.2 L/d) and (0.5-3.0 m/h), respectively on hydrogen production using response surface methodology (RSM) showed that H_2 yield was 0.32 L H_2 g^{-1} COD at Q_F and V_{up} of 1.7 L d^{-1} and 0.5 m h^{-1}, respectively The optimum ranges for the fermentative hydrogen production of the POME were Q_F = 2.1-3.7 L/d and V_{up} =1.5-2.3 m/h. The experimental results agreed very well with the model prediction.

REFERENCES

Abdelsalam, E., Samer, M., Attia, Y.A., Abdel-Hadi, M.A., Hassan, H.E., & Badr, Y. (2016). Comparison of nanoparticles effects on biogas and methane production from anaerobic digestion of cattle dung slurry. *Renewable Energy, 87,* 592-598.

Aguilar, M.R., Fdez-Guelfo, L.A., Alvarez-Gallego, C.J., & García, L.R. (2013). Effect of HRT on hydrogen production and organic matter solubilization in acidogenic anaerobic digestion of OFMSW. *Chemical Engineering Journal, 219,* 443-449.

Ahmad, A.L., Chong, M.F., & Bhatia, S. (2007). Mathematical modeling of multiple solutes system for reverse osmosis process in palm oil mill effluent (POME) treatment. *Chemical Engineering Journal, 132(1),* 183-193.

Ahn, J.H., & Forster, C.F. (2002). A comparison of mesophilic and thermophilic anaerobic upflow filters treating paper–pulp–liquors. *Process Biochemistry, 38(2),* 256-261.

Andre, L., Ndiaye, M., Pernier, M., Lespinard, O., Pauss, A., Lamy, E., & Ribeiro, T. (2016). Methane production improvement by modulation of solid phase immersion in dry batch anaerobic digestion process: Dynamic of methanogen populations. *Bioresource Technology, 207,* 353-360.

Angeriz-Campoy, R., Alvarez-Gallego, C.J., & Romero-García, L.I. (2015). Thermophilic anaerobic co-digestion of organic fraction of municipal solid waste (OFMSW) with food waste (FW): Enhancement of bio-hydrogen production. *Bioresource Technology, 194,* 291-296.

Antonopoulou, G., Gavala, H.N., Skiadas, I.V., & Lyberatos, G. (2011). Effect of substrate concentration on fermentative hydrogen production from sweet sorghum extract. *International Journal of Hydrogen Energy, 36(8),* 4843-4851.

Anzola-Rojas, M., Zaiat, M., & De Wever, H. (2016). Improvement of hydrogen production via ethanol-type fermentation in an anaerobic down-flow structured bed reactor. *Bioresource Technology, 202,* 42-49.

APHA. In. 2015. Standard methods for the examination of water and waste water. 23rd edition. Washington, DC, USA: American Public Health Association.

Argun, H., & Kargi, F. (2011). Bio-hydrogen production by different operational modes of dark and photo-fermentation: an overview. International *Journal of Hydrogen Energy, 36(13),* 7443-7459.

Argun, H., Kargi, F., Kapdan, I.K., & Oztekin, R. (2008). Biohydrogen production by dark fermentation of wheat powder solution: effects of C/N and C/P ratio on hydrogen yield and formation rate. *International Journal of Hydrogen Energy*, 33(7), 1813-1819.

Arikan, O.A., Mulbry, W., & Lansing, S. (2015). Effect of temperature on methane production from field-scale anaerobic digesters treating dairy manure. *Waste Management, 43,* 108-113.

Atif, A.A.Y., Fakhrul, A., Ngan, M.A., Morimoto, M., Iyuke, S.E., & Veziroglu, N.T. (2005). Fed batch production of hydrogen from palm oil mill effluent using anaerobic microflora. *International Journal of Hydrogen Energy, 30(13),* 1393-1397.

Badiei, M., Jahim, J.M., Anuar, N., Abdullah, S.R.S., Su, L. S., & Kamaruzzaman, M.A. (2012). Microbial community analysis of mixed anaerobic microflora in suspended sludge of ASBR producing hydrogen from palm oil mill effluent. *International Journal of Hydrogen Energy, 37(4),* 3169-3176.

Bakonyi, P., Nemestóthy, N., Lanko, J., Rivera, I., Buitron, G., & Belafi-Bako, K. (2015). Simultaneous biohydrogen production and purification in a double-membrane bioreactor system. *International Journal of Hydrogen Energy, 40(4),* 1690-1697.

Bisaillon, A., Turcot, J., & Hallenbeck, P.C. (2006). The effect of nutrient limitation on hydrogen production by batch cultures of Escherichia coli. *International Journal of Hydrogen Energy, 31(11),* 1504-1508.

Borja, R., & Banks, C.J. (1994). Anaerobic digestion of palm oil mill effluent using an up-flow anaerobic sludge blanket reactor. *Biomass and Bioenergy, 6(5),* 381-389.

Bowen, E.J., Dolfing, J., Davenport, R.J., Read, F.L., & Curtis, T.P. (2014). Low-temperature limitation of bioreactor sludge in anaerobic treatment of domestic wastewater. *Water Science and Technology, 69(5),* 1004-1013.

BP Statistical Review of World energy; 2011.

BP Energy Outlook 2030; 2012.

Buitron, G., Kumar, G., Martinez-Arce, A., & Moreno, G. (2014). Hydrogen and methane production via a two-stage processes (H_2-SBR+ CH_4-UASB) using tequila vinasses. *International Journal of Hydrogen Energy, 39(33),* 19249-19255.

Cail, R.G., & Barford, J.P. (1985). Thermophilic semi-continuous anaerobic digestion of palm-oil mill effluent. *Agricultural Waste, 13(4),* 295-304.

Cavinato, C., Bolzonella, D., Fatone, F., Giuliano, A., & Pavan, P. (2011). Two-phase thermophilic anaerobic digestion process for biohythane production treating biowaste: preliminary results. *Water Science and Technology, 64(3),* 715-721.

Cavinato, C., Giuliano, A., Bolzonella, D., Pavan, P., & Cecchi, F. (2012). Bio-hythane production from food waste by dark fermentation coupled with anaerobic digestion process: a long-term pilot scale experience. *International Journal of Hydrogen Energy, 37(15),* 11549-11555.

Chaisri, R., Boonsawang, P., Prasertsan, P., & Chaiprapat, S. (2007). Effect of organic loading rate on methane and volatile fatty acids productions from anaerobic treatment of palm oil mill effluent in UASB and UFAF reactors. *Songklanakarin Journal of Science and Technology, 2,* 311-323.

Chang, F.Y., & Lin, C.Y. (2004). Biohydrogen production using an up-flow anaerobic sludge blanket reactor. *International Journal of Hydrogen Energy, 29(1),* 33-39.

Chang, S. W., & Shaw, J.F. (2009). Biocatalysis for the production of carbohydrate esters. *New Biotechnology, 26(3),* 109-116.

Chen, Y., Cheng, J.J., & Creamer, K.S. (2008). Inhibition of anaerobic digestion process: a review. *Bioresource Technology, 99(10),* 4044-4064.

Cheng, X.Y., & Liu, C.Z. (2012). Enhanced coproduction of hydrogen and methane from cornstalks by a three-stage anaerobic fermentation process integrated with alkaline hydrolysis. *Bioresource Technology, 104,* 373-379.

Chong, M. L., Sabaratnam, V., Shirai, Y., & Hassan, M. A. (2009). Biohydrogen production from biomass and industrial wastes by dark fermentation. *International Journal of Hydrogen Energy, 34(8),* 3277-3287.

Chong, M. L., Yee, P. L., Aziz, S. A., Rahim, R. A., Shirai, Y., & Hassan, M. A. (2009a). Effects of pH, glucose and iron sulfate concentration on the yield of biohydrogen by Clostridium butyricum EB6. *International Journal of Hydrogen Energy, 34(21),* 8859-8865.

Chou, H.H., Huang, J.S., & Hong, W.F. (2004). Temperature dependency of granule characteristics and kinetic behavior in UASB reactors. *Journal of Chemical Technology and Biotechnology, 79(8),* 797-808.

Chu, C.Y., Tung, L., & Lin, C.Y. (2013). Effect of substrate concentration and pH on biohydrogen production kinetics from food industry wastewater by mixed culture. *International Journal of Hydrogen Energy, 38(35),* 15849-15855.

Chu, Y., Wei, Y., Yuan, X., & Shi, X. (2011). Bioconversion of wheat stalk to hydrogen by dark fermentation: effect of different mixed microflora on hydrogen yield and cellulose solubilisation. *Bioresource Technology, 102(4),* 3805-3809.

Chuang, Y.S., Lay, C. H., Sen, B., Chen, C. C., Gopalakrishnan, K., Wu, J. H., & Lin, C. Y. (2011). Biohydrogen and biomethane from water hyacinth (Eichhornia crassipes) fermentation: effects of substrate concentration and incubation temperature. *International Journal of Hydrogen Energy, 36(21),* 14195-14203.

Clean Malaysia (CM) magazine. (2015). Waste Management in Malaysia: In the Dumps, September issue.

Crutzen, P.J., Mosier, A. R., Smith, K.A., & Winiwarter, W. (2016). N_2O release from agro-biofuel production negates global warming reduction by replacing fossil fuels. In Paul J. Crutzen: *A Pioneer on Atmospheric Chemistry and Climate Change in the Anthropocene,* 227-238.

Dareioti, M.A., & Kornaros, M. (2014). Effect of hydraulic retention time (HRT) on the anaerobic co-digestion of agro-industrial wastes in a two-stage CSTR system. *Bioresource Technology, 167,* 407-415.

Dareioti, M.A., & Kornaros, M. (2015). Anaerobic mesophilic co-digestion of ensiled sorghum, cheese whey and liquid cow manure in a two-stage CSTR system: Effect of hydraulic retention time. *Bioresource Technology, 175,* 553-562.

DuBois, M., Gilles, K.A., Hamilton, J.K., Rebers, P. T., & Smith, F. (1956). Colorimetric method for determination of sugars and related substances. *Analytical Chemistry, 28(3),* 350-356.

EIA, U. (2016). Energy Information Administration. *International Energy Outlook report,* DOE/EIA-0484.

Elliott, A., & Mahmood, T. (2007). Whitewater (WW) quality and treatment options for closing value added WW loops. *In Annual Meeting Pulp and Paper Technical Association of Canada, 93,* 325.

Elreedy, A., Tawfik, A., Enitan, A., Kumari, S., & Bux, F. (2016). Pathways of 3-biofules (hydrogen, ethanol and methane) production from petrochemical industry wastewater via anaerobic packed bed baffled reactor inoculated with mixed culture bacteria. Energy Conversion and Management, 122, 119-130.

Elsamadony, M., Tawfik, A., Danial, A., & Suzuki, M. (2015). Optimization of hydrogen production from organic fraction of municipal solid waste (OFMSW) dry anaerobic digestion with analysis of microbial community. *International Journal of Energy Research, 39(7),* 929-940.

Elsgaard, L., Olsen, A.B., & Petersen, S. O. (2016). Temperature response of methane production in liquid manures and co-digestates. *Science of the Total Environment, 539,* 78-84.

Environmental Quality Act (Scheduled Wastes) Regulations (2005). P.U.(A) 294/2005.

Environmental Quality Act. (2013). "Environmental Quality Act (Act 127)": P.U. (A) 198/77.

European Commission, Life Focus/ A cleaner, greener Europe: LIFE and the European Union waste policy, ISBN 9289460180, Office for Official Publications of the European Communities, Luxembourg, 2014.

Fang, H.H., Liu, H., & Zhang, T. (2012). Characterization of a hydrogen-producing granular sludge. *Biotechnology and Bioengineering, 78(1),* 44-52.

Farajzadehha, S., Mirbagheri, S. A., Farajzadehha, S., & Shayegan, J. (2012). Lab scale study of HRT and OLR optimization in UASB reactor for pretreating fortified wastewater in various operational temperatures. *APCBEE Procedia, 1,* 90-95.

Fernandez, C., Cuetos, M.J., Martínez, E.J., & Gomez, X. (2015). Thermophilic anaerobic digestion of cheese whey: Coupling H 2 and CH 4 production. *Biomass and Bioenergy, 81,* 55-62.

Ferreira, S., Duarte, A.P., Ribeiro, M.H., Queiroz, J. A., & Domingues, F. C. (2009). Response surface optimization of enzymatic hydrolysis of Cistus ladanifer and Cytisus striatus for bioethanol production. *Biochemical Engineering Journal, 45(3),* 192-200.

Fezzani, B., & Cheikh, R.B. (2010). Two-phase anaerobic co-digestion of olive mill wastes in semi-continuous digesters at mesophilic temperature. *Bioresource Technology, 101(6),* 1628-1634.

Foo, K.Y., & Hameed, B.H. (2010). Insight into the applications of palm oil mill effluent: a renewable utilization of the industrial agricultural waste. *Renewable and Sustainable Energy Reviews, 14(5),* 1445-1452.

Frolund, B., Palmgren, R., Keiding, K., & Nielsen, P. H. (1996). Extraction of extracellular polymers from activated sludge using a cation exchange resin. *Water Research,* 30(8), 1749-1758.

Garg, K.K., Prasad, B. (2016). Development of Box Behnken design for treatment of terephthalic acid wastewater by electrocoagulation process: Optimization of process and analysis of sludge. *Journal of Environmental Chemical. Engineering, 4,* 178–190.

Ghouali, A., Sari, T., & Harmand, J. (2015). Maximizing biogas production from the anaerobic digestion. *Journal of Process Control, 36,* 79-88.

Giordano, A., Sarli, V., Lavagnolo, M. C., & Spagni, A. (2014). Evaluation of aeration pretreatment to prepare an inoculum for the two-stage hydrogen and methane production process. *Bioresource Technology*, *166*, 211-218.

Giuliano, A., Zanetti, L., Micolucci, F., & Cavinato, C. (2014). 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, 69(11),* 2200-2209.

Guiot, S.R., Pauss, A., & Costerton, J.W. (1992). A structured model of the anaerobic granule consortium. *Water Science and Technology, 25(7),* 1-10.

Guo, X.M., Trably, E., Latrille, E., Carrere, H., & Steyer, J.P. (2010). Hydrogen production from agricultural waste by dark fermentation: a review. *International Journal of Hydrogen Energy, 35(19),* 10660-10673.

Gupta, P., Sreekrishnan, T.R., & Ahammad, S.Z. (2016). Role of sludge volume index in anaerobic sludge granulation in a hybrid anaerobic reactor. *Chemical Engineering Journal, 283,* 338-350.

Hallenbeck, P.C., & Ghosh, D. (2009). Advances in fermentative biohydrogen production: the way forward?. *Trends in Biotechnology, 27(5),* 287-297.

Han, S.K., Kim, S.H., Kim, H.W., & Shin, H.S. (2005). Pilot-scale two-stage process: a combination of acidogenic hydrogenesis and methanogenesis. *Water Science and Technology, 52(1-2),* 131-138.

Harish, B. S., Ramaiah, M. J., & Uppuluri, K. B. (2015). Bioengineering strategies on catalysis for the effective production of renewable and sustainable energy. *Renewable and Sustainable Energy Reviews, 51,* 533-547.

Henze, M., & Harremoes, P. (1983). Anaerobic treatment of wastewater in fixed film reactors–a literature review. *Water Science and Technology, 15(8-9),* 1-101.

Intanoo, P., Chaimongkol, P., & Chavadej, S. (2016). Hydrogen and methane production from cassava wastewater using two-stage upflow anaerobic sludge blanket reactors (UASB) with an emphasis on maximum hydrogen production. *International Journal of Hydrogen Energy, 41(14),* 6107-6114.

Intanoo, P., Rangsanvigit, P., Malakul, P., & Chavadej, S. (2014). Optimization of separate hydrogen and methane production from cassava wastewater using two-stage upflow anaerobic sludge blanket reactor (UASB) system under thermophilic operation. *Bioresource Technology, 173,* 256-265.

Intanoo, P., Rangsunvigit, P., Namprohm, W., Thamprajamchit, B., Chavadej, J., & Chavadej, S. (2012). Hydrogen production from alcohol wastewater by an anaerobic sequencing batch reactor under thermophilic operation: Nitrogen and phosphorous uptakes and transformation. *International Journal of Hydrogen Energy, 37(15),* 11104-11112.

IPCC, (2016). IPCC Guidelines for National Greenhouse Gas Inventories. Reference Manual. Land-use change & Forestry.

Ishigaki, T., Yamada, M., Nagamori, M., Ono, Y., & Inoue, Y. (2005). Estimation of methane emission from whole waste landfill site using correlation between flux and ground temperature. *Environmental Geology, 48(7),* 845-853.

Ismail, I., Hassan, M.A., Rahman, N.A.A., & Soon, C. S. (2010). Thermophilic biohydrogen production from palm oil mill effluent (POME) using suspended mixed culture. *Biomass and Bioenergy, 34(1),* 42-47.

Jantsch, T.G., Angelidaki, I., Schmidt, J.E., de Hvidsten, B.B., & Ahring, B.K. (2002). Anaerobic biodegradation of spent sulphite liquor in a UASB reactor. *Bioresource Technology, 84(1),* 15-20.

Jariyaboon, R., Sompong, O., & Kongjan, P. (2015). Bio-hydrogen and bio-methane potentials of skim latex serum in batch thermophilic two-stage anaerobic digestion. *Bioresource Technology, 198,* 198-206.

Jung, K.W., Kim, D.H., & Shin, H.S. (2011). Fermentative hydrogen production from Laminaria japonica and optimization of thermal pretreatment conditions. *Bioresource Technology, 102(3),* 2745-2750.

Jung, K.W., Kim, D.H., Lee, M.Y., & Shin, H.S. (2012). Two-stage UASB reactor converting coffee drink manufacturing wastewater to hydrogen and methane. *International Journal of Hydrogen Energy, 37(9),* 7473-7481.

Khan, M.A., Ngo, H.H., Guo, W.S., Liu, Y.W., Zhou, J.L., Zhang, J., & Wang, J. (2016). Comparing the value of bioproducts from different stages of anaerobic membrane bioreactors. *Bioresource Technology, 214,* 816-825.

Kim, D.H., Son, H., & Kim, M.S. (2012). Effect of substrate concentration on continuous photo-fermentative hydrogen production from lactate using Rhodobacter sphaeroides. *International Journal of Hydrogen Energy, 37(20),* 15483-15488.

Kim, M., Liu, C., Noh, J.W., Yang, Y., Oh, S., Shimizu, K., & Zhang, Z. (2013). Hydrogen and methane production from untreated rice straw and raw sewage sludge under thermophilic anaerobic conditions. *International Journal of Hydrogen Energy, 38(21),* 8648-8656.

Kim, S. H., Cheon, H. C., & Lee, C. Y. (2016). Enhancement of hydrogen production by recycling of methanogenic effluent in two-phase fermentation of food waste. *International Journal of Hydrogen Energy, 37(18),* 13777-13782.

King, L. S., & Yu, L. C. (2013). A retrofitted palm oil mill effluent treatment system for tapping biogas. *European International Journal of Science and Technology, 2(5),* 106-114.

Kisielewska, M., Wysocka, I., & Rynkiewicz, M. R. (2014). Continuous biohydrogen and biomethane production from whey permeate in a two-stage fermentation process. *Environmental Progress & Sustainable Energy, 33(4),* 1411-1418.

Kobayashi, T., Xu, K. Q., Li, Y. Y., & Inamori, Y. (2012). Effect of sludge recirculation on characteristics of hydrogen production in a two-stage hydrogen–methane fermentation process treating food wastes. *International Journal of Hydrogen Energy, 37(7),* 5602-5611.

Kongjan, P., Jariyaboon, R., & Sompong, O. (2014). Anaerobic digestion of skim latex serum (SLS) for hydrogen and methane production using a two-stage process in a series of up-flow anaerobic sludge blanket (UASB) reactor. *International Journal of Hydrogen Energy, 39(33),* 19343-19348.

Koocheki, A., Taherian, A.R., Razavi, S.M.A., Bostan, A. (2009). Response surface methodology for optimization of extraction yield, viscosity, hue and emulsion stability of mucilage extracted from Lepidium perfoliatum seeds. *Food Hydrocolloids, 23,* 2369–2379.

Koroneos, C., Dompros, A., Roumbas, G., & Moussiopoulos, N. (2004). Life cycle assessment of hydrogen fuel production processes. *International Journal of Hydrogen Energy, 29(14),* 1443-1450.

Kosaric, N., & Blaszczyk, R. (1990). Microbial aggregates in anaerobic wastewater treatment. *In Bioprocesses and Applied Enzymology* Springer, Berlin, Heidelberg, 27-62.

Kotsopoulos, T.A., Zeng, R.J., & Angelidaki, I. (2006). Biohydrogen production in granular up-flow anaerobic sludge blanket (UASB) reactors with mixed cultures under hyper-thermophilic temperature (70°C). *Biotechnology and Bioengineering, 94(2),* 296-302.

Kraemer, J.T., & Bagley, D.M. (2005). Continuous fermentative hydrogen production using a two-phase reactor system with recycle. *Environmental Science & Technology, 39(10),* 3819-3825.

Kumar, G., Park, J.H., Kim, M.S., Kim, D.H., & Kim, S.H. (2014). Hydrogen fermentation of different galactose–glucose compositions during various hydraulic retention times (HRTs). *International Journal of Hydrogen Energy, 39(35),* 20625-20631.

Kumar, G., Sivagurunathan, P., Park, J. H., Park, J. H., Park, H. D., Yoon, J. J., & Kim, S. H. (2016). HRT dependent performance and bacterial community population of granular hydrogen-producing mixed cultures fed with galactose. *Bioresource Technology, 206,* 188-194.

Kvesitadze, G., Sadunishvili, T., Dudauri, T., Zakariashvili, N., Partskhaladze, G., Ugrekhelidze, V., & Jobava, M. 2012. Two-stage anaerobic process for bio-hydrogen and bio-methane combined production from biodegradable solid wastes. *Energy, 37(1),* 94-102.

Kwietniewska, E., & Tys, J. (2014). Process characteristics, inhibition factors and methane yields of anaerobic digestion process, with particular focus on microalgal biomass fermentation. *Renewable and Sustainable Energy Reviews, 34,* 491-500.

Lam, M.K., & Lee, K.T. (2011). Renewable and sustainable bioenergies production from palm oil mill effluent (POME): win–win strategies toward better environmental protection. *Biotechnology Advances, 29(1),* 124-141.

Lateef, S.A., Beneragama, N., Yamashiro, T., Iwasaki, M., & Umetsu, K. (2014). Batch anaerobic co-digestion of cow manure and waste milk in two-stage process for hydrogen and methane productions. *Bioprocess and Biosystems Engineering, 37(3),* 355-363.

Lau, I.W., & Fang, H.H. (1997). Effect of temperature shock to thermophilic granules. *Water Research, 31(10),* 2626-2632.

Lee, D. Y., Ebie, Y., Xu, K.Q., Li, Y.Y., & Inamori, Y. (2010). Continuous H_2 and CH_4 production from high-solid food waste in the two-stage thermophilic fermentation process with the recirculation of digester sludge. *Bioresource Technology, 101(1),* S42-S47.

Lee, D. Y., Li, Y.Y., Oh, Y.K., Kim, M.S., & Noike, T. (2009). Effect of iron concentration on continuous H 2 production using membrane bioreactor. *International Journal of Hydrogen Energy, 34(3),* 1244-1252.

Lee, I.S., Parameswaran, P., & Rittmann, B.E. (2011). Effects of solids retention time on methanogenesis in anaerobic digestion of thickened mixed sludge. *Bioresource Technology, 102(22),* 10266-10272.

Lettinga, G. (1995). Anaerobic digestion and wastewater treatment systems. *Antonie Van Leeuwenhoek, 67(1),* 3-28.

Lettinga, G.A.F.M., Van Velsen, A.F.M., Hobma, S.W., De Zeeuw, W., & Klapwijk, A. (1980). Use of the upflow sludge blanket (USB) reactor concept for biological wastewater treatment, especially for anaerobic treatment. *Biotechnology and Bioengineering, 22(4),* 699-734.

Lettinga, G., & Pol, L. H. (1991). UASB-process design for various types of wastewaters. *Water Science and Technology, 24(8)*, 87-107.

Li, J., Ai, B., & Ren, N. (2013). Effect of Initial Sludge Loading Rate on the Formation of Ethanol Type Fermentation for Hydrogen Production in a Continuous Stirred-Tank Reactor. *Environmental Progress & Sustainable Energy.* 32(4): 1271-1279.

Lier, J.B., Grolle, K.C., Stams, A.J., Macario, E.C., & Lettinga, G. (1992). Start-up of a thermophilic upflow anaerobic sludge bed (UASB) reactor with mesophilic granular sludge. *Applied Microbiology and Biotechnology, 37(1),* 130-135.

Limwattanalert, N. (2011). Hydrogen production from ethanol wastewater by using upflow anaerobic sludge blanket reactor. The Petroleum and Petrochemical College, Chulalongkorn University.

Lin, Q., De Vrieze, J., He, G., Li, X., & Li, J. (2016). Temperature regulates methane production through the function centralization of microbial community in anaerobic digestion. *Bioresource Technology, 216,* 150-158.

Lin, Y., Wu, S., & Wang, D. (2013). Hydrogen-methane production from pulp & paper sludge and food waste by mesophilic–thermophilic anaerobic co-digestion. *International Journal of Hydrogen Energy, 38(35),* 15055-15062.

Linke, B., Muha, I., Wittum, G., & Plogsties, V. (2013). Mesophilic anaerobic co-digestion of cow manure and biogas crops in full scale German biogas plants: a model for calculating the effect of hydraulic retention time and VS crop proportion in the mixture on methane yield from digester and from digestate storage at different temperatures. *Bioresource Technology, 130,* 689-695.

Liu, C.M., Wu, S.Y., Chu, C.Y., & Chou, Y.P. (2014). Biohydrogen production from rice straw hydrolyzate in a continuously external circulating bioreactor. *International Journal of Hydrogen Energy, 39(33),* 19317-19322.

Liu, D., Liu, D., Zeng, R. J., & Angelidaki, I. (2006). Hydrogen and methane production from household solid waste in the two-stage fermentation process. *Water Research, 40(11),* 2230-2236.

Liu, X., Li, R., Ji, M., & Han, L. (2013). Hydrogen and methane production by co-digestion of waste activated sludge and food waste in the two-stage fermentation process: substrate conversion and energy yield. *Bioresource Technology, 146,* 317-323.

Liu, Y., Xu, H.L., Yang, S.F. and Tay, J.H. (2003). Mechanisms and models for anaerobic granulation in upflow anaerobic sludge blanket reactor. *Water Research, 37,* 661–673.

Liu, Y., Zhang, Y., Quan, X., Li, Y., Zhao, Z., Meng, X., & Chen, S. (2012). Optimization of anaerobic acidogenesis by adding Fe 0 powder to enhance anaerobic wastewater treatment. *Chemical Engineering Journal, 192,* 179-185.

Luo, G., Xie, L., Zou, Z., Wang, W., Zhou, Q., & Shim, H. (2010). Anaerobic treatment of cassava stillage for hydrogen and methane production in continuously stirred tank reactor (CSTR) under high organic loading rate (OLR). *International Journal of Hydrogen Energy, 35(21),* 11733-11737.

Luo, J., Feng, L., Zhang, W., Li, X., Chen, H., Wang, D., & Chen, Y. (2014). Improved production of short-chain fatty acids from waste activated sludge driven by carbohydrate addition in continuous-flow reactors: influence of SRT and temperature. *Applied Energy.* 113: 51-58.

Lutoslawski, K., Ryznar-Luty, A., Cibis, E., Krzywonos, M., & Miskiewicz, T. (2011). Biodegradation of beet molasses vinasse by a mixed culture of microorganisms: Effect of aeration conditions and pH control. *Journal of Environmental Sciences, 23(11),* 1823-1830.

Lutpi, N.A., Jahim, J.M., Mumtaz, T., Harun, S., & Abdul, P.M. (2016). Batch and continuous thermophilic hydrogen fermentation of sucrose using anaerobic sludge from palm oil mill effluent via immobilisation technique. *Process Biochemistry, 51(2),* 297-307.

Ma, A.N. (1999). Innovations in management of palm oil mill effluent. Planter (Malaysia).

Ma, A.N., and Ong, A.S.H. (1985). Anaerobic digestion of palm oil mill. *PORIM Bulletin.* 4: 35–45.

Ma, D., Wang, J., Chen, T., Shi, C., Peng, S., & Yue, Z. (2015). Iron-oxide-promoted anaerobic process of the aquatic plant of curly leaf pondweed. *Energy & Fuels, 29(7),* 4356-4360.

Macario, A.J.L., Visser, F.A., van Lier, J.B., & de Macario Eerly, C. (1991). Topography of methanogenic subpopulations in a microbial consortium adapting to thermophilic conditions. *Microbiology, 137(9),* 2179-2189.

MacLeod, F.A., Guiot, S.R., & Costerton, J.W. (1990). Layered structure of bacterial aggregates produced in an upflow anaerobic sludge bed and filter reactor. *Applied and Environmental Microbiology, 56(6),* 1598-1607.

Madigan, M.T., Martinko, J.M., & Brock, T.D. (2009). Brock Mikrobiologie. Pearson Deutschland GmbH.

Malaysia A.I. (2013). National Biomass Strategy 2020: New wealth creation for Malaysia's biomass industry: Version 2.0, Agensi Inovasi Malaysia, Kuala Lumpur.

Malaysian Palm Oil Board (MPOB). Overview of the Malaysian oil palm industry 2009; 2010.

Mamimin, C., Singkhala, A., Kongjan, P., Suraraksa, B., Prasertsan, P., Imai, T., & Sompong, O. (2015). Two-stage thermophilic fermentation and mesophilic methanogen process for biohythane production from palm oil mill effluent. *International Journal of Hydrogen Energy, 40(19),* 6319-6328.

Mann, U. (2009). Principles of chemical reactor analysis and design: new tools for industrial chemical reactor operations. John Wiley & Sons.

Mao, C., Feng, Y., Wang, X., & Ren, G. (2015). Review on research achievements of biogas from anaerobic digestion. *Renewable and Sustainable Energy Reviews, 45,* 540-555.

Mariakakis, I., Bischoff, P., Krampe, J., Meyer, C., & Steinmetz, H. (2011). Effect of organic loading rate and solids retention time on microbial population during bio-hydrogen production by dark fermentation in large lab-scale. *International Journal of Hydrogen Energy, 36(17),* 10690-10700.

Maspolim, Y., Zhou, Y., Guo, C., Xiao, K., & Ng, W.J. (2015). Comparison of single-stage and two-phase anaerobic sludge digestion systems–Performance and microbial community dynamics. *Chemosphere, 140,* 54-62.

Massanet-Nicolau, J., Dinsdale, R., Guwy, A., & Shipley, G. (2015). Utilising biohydrogen to increase methane production, energy yields and process efficiency via two stage anaerobic digestion of grass. *Bioresource Technology, 189,* 379-383.

McHugh, S., Collins, G., & O’Flaherty, V. (2006). Long-term, high-rate anaerobic biological treatment of whey wastewaters at psychrophilic temperatures. *Bioresource Technology, 97(14),* 1669-1678.

Metcalf & Eddy, Burton, F.L., Stensel, H.D., & Tchobanoglous, G. (2003). Wastewater engineering: treatment and reuse. McGraw Hill.

Mohammadi, P., Ibrahim, S., & Annuar, M.S.M. (2012). Effects of biomass, COD and bicarbonate concentrations on fermentative hydrogen production from POME by granulated sludge in a batch culture. *International Journal of Hydrogen Energy, 37(23),* 17801-17808.

Mohammadi, P., Ibrahim, S., Annuar, M.S.M., Khashij, M., Mousavi, S.A., & Zinatizadeh, A. (2016). Optimization of fermentative hydrogen production from palm oil mill effluent in an up-flow anaerobic sludge blanket fixed film bioreactor. *Sustainable Environment Research.*

Montgomery, D.C. (2001). Design and Analysis of Experiments. United States: John Wiley & Sons Inc.

Morgan, J.W., Forster, C.F., & Evison, L. (1990). A comparative study of the nature of biopolymers extracted from anaerobic and activated sludges. *Water Research, 24(6),* 743-750.

Morimoto, M., Atsuko, M., Atif, A.A.Y., Ngan, M. A., Fakhrul-Razi, A., Iyuke, S.E., & Bakir, A.M. (2004). Biological production of hydrogen from glucose by natural anaerobic microflora. *International Journal of Hydrogen Energy, 29(7),* 709-713.

MPOB, Economic & Industry Development Devision. Oil Palm Planted Area. Available at: ⟨http://bepi.mpob.gov.my/index.php/statistics/area.html⟩; 2015.

Muhamad, M.H., Sheikh Abdullah, S.R., Mohamad, A.B., Abdul Rahman, R., Hasan Kadhum, A.A. (2013). Application of response surface methodology (RSM) for optimisation of COD, NH3-N and 2,4-DCP removal from recycled paper wastewater in a pilot-scale granular activated carbon sequencing batch biofilm reactor (GAC-SBBR). *Journal of Environmental Management, 121,* 179–190.

Muyzer, G., & Smalla, K. (1998). Application of denaturing gradient gel electrophoresis (DGGE) and temperature gradient gel electrophoresis (TGGE) in microbial ecology. *Antonie Van Leeuwenhoek, 73(1),* 127-141.

Myers, R.H. and Montgomery, D.C. (2002). Response Surface Methodology: Process and Product Optimization Using Designed Experiments. United States: John Wiley & Sons Inc.

Najafpour, G.D., Zinatizadeh, A.A.L., Mohamed, A.R., Isa, M.H., & Nasrollahzadeh, H. (2006). High-rate anaerobic digestion of palm oil mill effluent in an upflow anaerobic sludge-fixed film bioreactor. *Process Biochemistry, 41(2),* 370-379.

Nasr, M., Tawfik, A., Ookawara, S., Suzuki, M., Kumari, S., & Bux, F. (2015). Continuous biohydrogen production from starch wastewater via sequential dark-photo fermentation with emphasize on maghemite nanoparticles. *Journal of Industrial and Engineering Chemistry, 21,* 500-506.

Nasr, N., Elbeshbishy, E., Hafez, H., Nakhla, G., & El Naggar, M.H. (2012). Comparative assessment of single-stage and two-stage anaerobic digestion for the treatment of thin stillage. *Bioresource Technology, 111,* 122-126.

Nathoa, C., Sirisukpoca, U., & Pisutpaisal, N. (2014). Production of hydrogen and methane from banana peel by two phase anaerobic fermentation. *Energy Procedia, 50,* 702-710.

Nges, I.A., & Liu, J. (2010). Effects of solid retention time on anaerobic digestion of dewatered-sewage sludge in mesophilic and thermophilic conditions. *Renewable Energy, 35(10),* 2200-2206.

Ngo, T.A., & Sim, S.J. (2012). Dark fermentation of hydrogen from waste glycerol using hyperthermophilic eubacterium Thermotoga neapolitana. *Environmental Progress & Sustainable Energy, 31(3),* 466-473.

Nissila, M.E., Lay, C.H., & Puhakka, J. A. (2014). Dark fermentative hydrogen production from lignocellulosic hydrolyzates–a review. *Biomass and Bioenergy, 67,* 145-159.

Noparat, P., Prasertsan, P., & Sompong, O. (2012). Potential for using enriched cultures and thermotolerant bacterial isolates for production of biohydrogen from oil palm sap and microbial community analysis. *International Journal of Hydrogen Energy, 37(21),* 16412-16420.

Norfadilah, N., Raheem, A., Harun, R. (2016). Bio-hydrogen production from palm oil mill effluent (POME): A preliminary study. *International Journal of Hydrogen Energy, 41,* 3–7

Oswal, N., Sarma, P. M., Zinjarde, S.S., & Pant, A. (2002). Palm oil mill effluent treatment by a tropical marine yeast. *Bioresource Technology, 85(1),* 35-37.

Paepatung, N., Nopharatana, A., & Songkasiri, W. (2009). Bio-methane potential of biological solid materials and agricultural wastes. *Asian Journal on Energy and Environment, 10(1),* 19-27.

Pasupuleti, S.B., & Mohan, S.V. (2015). Single-stage fermentation process for high-value biohythane production with the treatment of distillery spent-wash. *Bioresource Technology, 189,* 177-185.

Patel, S.K., Kumar, P., Singh, M., Lee, J.K., & Kalia, V.C. (2015). Integrative approach to produce hydrogen and polyhydroxybutyrate from biowaste using defined bacterial cultures. *Bioresource Technology, 176,* 136-141.

Pattra, S., Sangyoka, S., Boonmee, M., & Reungsang, A. (2008). Bio-hydrogen production from the fermentation of sugarcane bagasse hydrolysate by Clostridium butyricum. *International Journal of Hydrogen Energy, 33(19),* 5256-5265.

Pishgar-Komleh, S.H., Keyhani, A., Mostofi-Sarkari, M., Jafari, A. (2012). Optimization of Seed Corn Harvesting Losses Applying Response Surface Methodology. *Research Journal of Applied Science Engineering Technology, 4,* 2350–2356.

Pisutpaisal, N., Nathao, C., & Sirisukpoka, U. (2014). Biological hydrogen and methane production in from food waste in two-stage CSTR. *Energy Procedia, 50,* 719-722.

Poh, P.E., & Chong, M.F. (2009). Development of anaerobic digestion methods for palm oil mill effluent (POME) treatment. *Bioresource Technology, 100(1),* 1-9.

Prajapati, S.K., Kaushik, P., Malik, A., & Vijay, V.K. (2013). Phycoremediation coupled production of algal biomass, harvesting and anaerobic digestion: possibilities and challenges. *Biotechnology Advances, 31(8),* 1408-1425.

Prasertsan, P., Sompong, O., & Birkeland, N.K. (2009). Optimization and microbial community analysis for production of biohydrogen from palm oil mill effluent by thermophilic fermentative process. *International Journal of Hydrogen Energy, 34(17),* 7448-7459.

Pretel, R., Shoener, B. D., Ferrer, J., & Guest, J.S. (2015). Navigating environmental, economic, and technological trade-offs in the design and operation of submerged anaerobic membrane bioreactors (AnMBRs). *Water Research, 87,* 531-541.

Qiao, W., Takayanagi, K., Niu, Q., Shofie, M., & Li, Y.Y. (2013). Long-term stability of thermophilic co-digestion submerged anaerobic membrane reactor encountering high organic loading rate, persistent propionate and detectable hydrogen in biogas. *Bioresource Technology, 149,* 92-102.

Rajan, S.S. (2002). *Research Trends in Modern Botany*, Anmol Publications Pvt. Ltd.

Ruggeri, B., Tommasi, T., & Sanfilippo, S. (2015). BioH_2 & BioCH_4 Through Anaerobic Digestion: From Research to Full-scale Applications. Springer.

Saeed, M.O., Azizli, K., Isa, M.H., Bashir, M.J.K. (2015). Application of CCD in RSM to obtain optimize treatment of POME using Fenton oxidation process. *Journal of Water Process Engineering, (8),* 7–16.

Salerno, M.B., Park, W., Zuo, Y., & Logan, B.E. (2006). Inhibition of biohydrogen production by ammonia. *Water Research, 40(6),* 1167-1172.

Schievano, A., Tenca, A., Scaglia, B., Merlino, G., Rizzi, A., Daffonchio, D., & Adani, F. (2012). Two-stage vs single-stage thermophilic anaerobic digestion: comparison of energy production and biodegradation efficiencies. *Environmental Science & Technology, 46(15),* 8502-8510.

Schmidt, J.E., & Ahring, B.K. (1996). Granular sludge formation in upflow anaerobic sludge blanket (UASB) reactors. *Biotechnology and Bioengineering, 49(3),* 229-246.

Sekiguchi, Y., Kamagata, Y., Nakamura, K., Ohashi, A., & Harada, H. (1999). Fluorescence in situ hybridization using 16S rRNA-targeted oligonucleotides reveals localization of methanogens and selected uncultured bacteria in mesophilic and thermophilic sludge granules. *Applied and Environmental Microbiology, 65(3),* 1280-1288.

Shen, Y., Linville, J.L., Urgun-Demirtas, M., Schoene, R. P., & Snyder, S. W. (2015). Producing pipeline-quality biomethane via anaerobic digestion of sludge amended with corn stover biochar with in-situ CO_2 removal. *Applied Energy, 158,* 300-309.

Sheng, T., Gao, L., Zhao, L., Liu, W., & Wang, A. (2015). Direct hydrogen production from lignocellulose by the newly isolated Thermoanaerobacterium thermosaccharolyticum strain DD32. *RSC Advances, 5(121),* 99781-99788.

Shin, H.S., Youn, J.H., & Kim, S.H. (2004). Hydrogen production from food waste in anaerobic mesophilic and thermophilic acidogenesis. *International Journal of Hydrogen Energy, 29(13),* 1355-1363.

Shofie, M., Qiao, W., Li, Q., Takayanagi, K., & Li, Y.Y. (2015). Comprehensive monitoring and management of a long-term thermophilic CSTR treating coffee grounds, coffee liquid, milk waste, and municipal sludge. *Bioresource Technology*, 192: 202-211.

Siang, L.C. (2006). *Biodegradation of oil and grease in upflow anaerobic sludge blanket reactor for palm oil mill effluent treatment.* (Master’s degree thesis). Universiti Teknologi Malaysia, Malaysia.

Siddique, N.I., Munaim, M.S.A., & Wahid, Z.A. (2015). Role of biogas recirculation in enhancing petrochemical wastewater treatment efficiency of continuous stirred tank reactor. *Journal of Cleaner Production, 91,* 229-234.

Singh, L., & Wahid, Z.A. (2015). Methods for enhancing bio-hydrogen production from biological process: a review. *Journal of Industrial and Engineering Chemistry, 21,* 70-80.

Singh, L., Siddiqui, M.F., Ahmad, A., Rahim, M.H.A., Sakinah, M., & Wahid, Z.A. (2013). Application of polyethylene glycol immobilized Clostridium sp. LS2 for continuous hydrogen production from palm oil mill effluent in upflow anaerobic sludge blanket reactor. *Biochemical Engineering Journal, 70,* 158-165.

Smith, M.R., & Mah, R.A. (1978). Growth and methanogenesis by Methanosarcina strain 227 on acetate and methanol. *Applied and Environmental Microbiology, 36(6),* 870-879.

Sofiah, M.K., Ong, H.L., Akil, H.M., & Ishak, Z.A.M. (2015). Effect of polypropylene-methyl polyhedral Oligomeric silsesquioxane Compatibilizer in polypropylene/Silica Nanocomposites: Mechanical, Morphological and Thermal Studies. *In Materials Science Forum, 803,* 265-268.

Sompong, O., Boe, K., & Angelidaki, I. (2012). Thermophilic anaerobic co-digestion of oil palm empty fruit bunches with palm oil mill effluent for efficient biogas production. *Applied Energy, 93,* 648-654.

Sompong, O., Prasertsan, P., Intrasungkha, N., Dhamwichukorn, S., & Birkeland, N.K. (2007). Improvement of biohydrogen production and treatment efficiency on palm oil mill effluent with nutrient supplementation at thermophilic condition using an anaerobic sequencing batch reactor. *Enzyme and Microbial Technology, 41(5),* 583-590.

Srinivasan, J. (1999). Ice ages. *Resonance, 4(8),* 25-35.

Stabnikova, O., Liu, X.Y., & Wang, J.Y. (2008). Digestion of frozen/thawed food waste in the hybrid anaerobic solid–liquid system. *Waste Management, 28(9),* 1654-1659.

Stafford, D.A. (1982). The effects of mixing and volatile fatty acid concentrations on anaerobic digester performance. *Biomass, 2(1),* 43-55.

Subramaniam, V., MaAn, N., ChooYuen, M., & Nik Meriam, N.S. (2008). Environmental performance of the milling process of Malaysian palm oil using the life cycle assessment approach. *American Journal of Environmental Sciences, 4(4),* 310-315.

Suksong, W., Kongjan, P., & Sompong, O. (2015). Biohythane production from co-digestion of palm oil mill effluent with solid residues by two-stage solid state anaerobic digestion process. *Energy Procedia, 79,* 943-949.

Sun, W., Yu, G., Louie, T., Liu, T., Zhu, C., Xue, G., & Gao, P. (2015). From mesophilic to thermophilic digestion: the transitions of anaerobic bacterial, archaeal, and fungal community structures in sludge and manure samples. *Applied Microbiology and Biotechnology, 99(23),* 10271-10282.

Syutsubo, K., Harada, H., Ohashi, A., & Suzuki, H. (1997). An effective start-up of thermophilic UASB reactor by seeding mesophilically-grown granular sludge. *Water Science and Technology, 36(7),* 391-398.

Tahti, H., Kaparaju, P., & Rintala, J. (2013). Hydrogen and methane production in extreme thermophilic conditions in two-stage (upflow anaerobic sludge bed) UASB reactor system. *International Journal of Hydrogen Energy, 38(12),* 4997-5002.

Thani, M.I., Hussin, R., Ibrahim, W.W.R., & Sulaiman, M. S. (1999). Industrial processes & the environment: crude palm oil industry, Handbook No.3. Department of Environment, Kuala Lumpur, 7-54.

Thong, O., Prasertsan, P., & Birkeland, N.K. (2009). Evaluation of methods for preparing hydrogen-producing seed inocula under thermophilic condition by process performance and microbial community analysis. *Bioresource Technology, 100(2),* 909-918.

Ueno, Y., Fukui, H., & Goto, M. (2007). Operation of a two-stage fermentation process producing hydrogen and methane from organic waste. *Environmental Science & Technology, 41(4),* 1413-1419.

Van Ginkel, S., & Logan, B.E. (2005). Inhibition of biohydrogen production by undissociated acetic and butyric acids. *Environmental Science & Technology, 39(23),* 9351-9356.

Venetsaneas, N., Antonopoulou, G., Stamatelatou, K., Kornaros, M., & Lyberatos, G. (2009). Using cheese whey for hydrogen and methane generation in a two-stage continuous process with alternative pH controlling approaches. *Bioresource Technology, 100(15),* 3713-3717.

Vijayaraghavan, K., & Ahmad, D. (2006). Biohydrogen generation from palm oil mill effluent using anaerobic contact filter. *International Journal of Hydrogen Energy, 31(10),* 1284-1291.

Walter, A., Franke-Whittle, I.H., Wagner, A.O., & Insam, H. (2015). Methane yields and methanogenic community changes during co-fermentation of cattle slurry with empty fruit bunches of oil palm. *Bioresource Technology, 175,* 619-623.

Wang, A.J., Cao, G.L., & Liu, W.Z. (2011). Biohydrogen production from anaerobic fermentation. In Biotechnology in China III: *Biofuels and Bioenergy,* 143-163

Wang, F., Hidaka, T., & Tsumori, J. (2014). Enhancement of anaerobic digestion of shredded grass by co-digestion with sewage sludge and hyperthermophilic pretreatment. *Bioresource Technology, 169,* 299-306.

Wang, J., & Wan, W. (2009). Factors influencing fermentative hydrogen production: a review. *International Journal of Hydrogen Energy, 34(2),* 799-811.

Wang, J., & Wan, W. (2011). Combined effects of temperature and pH on biohydrogen production by anaerobic digested sludge. *Biomass and Bioenergy, 35(9),* 3896-3901.

Wang, X., & Zhao, Y.C. (2009). A bench scale study of fermentative hydrogen and methane production from food waste in integrated two-stage process. *International Journal of Hydrogen Energy, 34(1),* 245-254.

Weiland, P. (2010). Biogas production: current state and perspectives. *Applied Microbiology and Biotechnology, 85(4),* 849-860.

Wong, Y.M., Wu, T.Y., & Juan, J.C. (2014). A review of sustainable hydrogen production using seed sludge via dark fermentation. *Renewable and Sustainable Energy Reviews, 34,* 471-482.

Wu, T.Y., Mohammad, A.W., Jahim, J.M., & Anuar, N. (2007). Palm oil mill effluent (POME) treatment and bioresources recovery using ultrafiltration membrane: effect of pressure on membrane fouling. *Biochemical Engineering Journal, 35(3),* 309-317.

Wu, T.Y., Mohammad, A.W., Jahim, J.M., & Anuar, N. (2010). Pollution control technologies for the treatment of palm oil mill effluent (POME) through end-of-pipe processes. *Journal of Environmental Management, 91(7),* 1467-1490.

Yacob, S., Hassan, M. A., Shirai, Y., Wakisaka, M., & Subash, S. (2005). Baseline study of methane emission from open digesting tanks of palm oil mill effluent treatment. *Chemosphere, 59(11),* 1575-1581.

Yang, Z., Guo, R., Xu, X., Fan, X., & Luo, S. (2011). Hydrogen and methane production from lipid-extracted microalgal biomass residues. *International Journal of Hydrogen Energy, 36(5),* 3465-3470.

Yang, Z., Xu, X., Guo, R., Fan, X., & Zhao, X. (2015). Accelerated methanogenesis from effluents of hydrogen-producing stage in anaerobic digestion by mixed cultures enriched with acetate and nano-sized magnetite particles. *Bioresource Technology, 190,* 132-139.

Yossan, S., Sompong, O., & Prasertsan, P. (2012). Effect of initial pH, nutrients and temperature on hydrogen production from palm oil mill effluent using thermotolerant consortia and corresponding microbial communities. *International Journal of Hydrogen Energy, 37(18),* 13806-13814.

Zamanzadeh, M., Hagen, L.H., Svensson, K., Linjordet, R., & Horn, S.J. (2016). Anaerobic digestion of food waste–effect of recirculation and temperature on performance and microbiology. *Water Research, 96*, 246-254.

Zhang, K., Ren, N.Q., & Wang, A.J. (2015). Fermentative hydrogen production from corn stover hydrolyzate by two typical seed sludges: Effect of temperature. *International Journal of Hydrogen Energy, 40(10),* 3838-3848.

Zhang, S., Lee, Y., Kim, T.H., & Hwang, S.J. (2013). Effects of OLRs and HRTs on hydrogen production from high salinity substrate by halophilic hydrogen producing bacterium (HHPB). *Bioresource Technology, 141,* 227-232.

Zhang, W., Wei, Q., Wu, S., Qi, D., Li, W., Zuo, Z., & Dong, R. (2014). Batch anaerobic co-digestion of pig manure with dewatered sewage sludge under mesophilic conditions. *Applied Energy, 128*, 175-183.

Zhao, J.H., Zhang, B.H., Li, N., Wang, B., & Li, Y.F. (2013). Analysis of hydrogen-production performance in a UASB system at low pH. *Huan Jing Ke Xue, 34(11),* 4370-4375.

Zhao, M.X., Yan, Q., Ruan, W.Q., Miao, H.F., Ren, H.Y., & Xu, Y. (2012). A comparative study of sequential hydrogen-methane and independent methane production from kitchen wastes. *Energy Sources, Part A: Recovery, Utilization, and Environmental Effects, 34(11),* 1046-1054.

Zhong, J., Stevens, D.K., & Hansen, C.L. (2015). Optimization of anaerobic hydrogen and methane production from dairy processing waste using a two-stage digestion in induced bed reactors (IBR). *International Journal of Hydrogen Energy, 40(45),* 15470-15476.

Zhu, H., Stadnyk, A., Béland, M., & Seto, P. (2008). Co-production of hydrogen and methane from potato waste using a two-stage anaerobic digestion process. *Bioresource Technology, 99(11),* 5078-5084.

Zhu, X., Kougias, P.G., Treu, L., Campanaro, S., & Angelidaki, I. (2016). Microbial community changes in methanogenic granules during the transition from mesophilic to thermophilic conditions. *Applied Microbiology and Biotechnology,* 1-10.

Zinatizadeh, A.A.L., Salamatinia, B., Zinatizadeh, S.L., Mohamed, A.R., & Isa, M.H. (2007). Palm oil mill effluent digestion in an up-flow anaerobic sludge fixed film bioreactor.

Zinder, S.H. (1990). Conversion of acetic acid to methane by thermophiles. *FEMS Microbiology Reviews, 6(2-3)*, 125-137.

LIST OF PUBLICATIONS

[1] Krishnan, S., Singh, L., Sakinah, M., Thakur, S., Wahid, Z. A., & Alkasrawi, M. (2016). Process enhancement of hydrogen and methane production from palm oil mill effluent using two-stage thermophilic and mesophilic fermentation. *International Journal of Hydrogen Energy*. **41(30):** 12888-12898**. (Elsevier, IF-3.3).**

[2] Krishnan, S., Singh, L., Sakinah, M., Thakur, S., Wahid, Z. A., & Sohaili, J. (2016). Effect of organic loading rate on hydrogen (H_2) and methane (CH_4) production in two-stage fermentation under thermophilic conditions using palm oil mill effluent (POME). *Energy for Sustainable Development.* **34:** 130-138. **(Elsevier, IF-2.8).**

[3] Krishnan, S., Singh, L., Sakinah, M., Thakur, S., Wahid, Z. A., & Ghrayeb, O. A. (2017). Role of organic loading rate in bioenergy generation from palm oil mill effluent in a two-stage up-flow anaerobic sludge blanket continuous-stirred tank reactor. *Journal of Cleaner Production.* **142:** 3044-3049. **(Elsevier, IF-4.97).**

[4] Krishnan, S., Singh, L., Sakinah, M., Thakur, S., Wahid, Z. A., & Otenio.A. (2017). Investigation of hydrogen and methane production from palm oil mill effluent with the influence of HRT. *Environmental Progress and Sustainable Energy*. **(Wiley, IF-1.64).**

[5] Mishra, P., Thakur, S., Singh, L., Krishnan, S., Sakinah, M., & Wahid, Z.A. (2017). Fermentative hydrogen production from indigenous mesophilic strain Bacillus anthracis PUNAJAN 1 newly isolated from palm oil mill effluent. *International Journal of Hydrogen Energy.* **(Elsevier, IF-3.3).**