BIOHYDROGEN PRODUCTION FROM ANAEROBIC FERMENTATION OF PINEAPPLE PEELS BY IMMOBILIZED CO-CULTURED BACTERIA

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ABSTRACT

Pineapple peel is one of the potential biomass feedstocks for biohydrogen production. The most convenient way to produce biohydrogen from lignocellulosic materials is through fermentation. The process is environmentally friendly and consumes low energy but has low production yield. One of the ways to increase the biohydrogen production is by utilising more than one species of hydrogen-producing bacteria, also known as co-culture, and immobilising them in a stable anaerobic condition. The suitability of the bacterial co-culture and their stability in immobilisation matrix were tested to achieve maximum biohydrogen production. The objective of this study was to improve biohydrogen production from pineapple peels via batch fermentation process using the most suitable immobilised co-culture. Pineapple peel was chosen due to its abundance and availability. Three different H_2-producing bacteria were selected, namely *Escherichia coli*, *Enterobacter aerogenes*, and *Clostridium sporogenes*, which were used as a single culture or combined as a co-culture. Their performances in free cell and immobilised form were then compared. For the immobilisation, activated carbon sponge and loofah sponge were used and compared. Finally, the cumulative production of biohydrogen and biohydrogen production rate were analysed by kinetics study. The modified Gompertz equation was fitted to the kinetics of cumulative biohydrogen production via Excel solver application. All fermentation processes were carried out at pH 7 and 32 ± 1 °C, with 30 % v/v inoculum of working volume in batch process. In terms of biohydrogen production rate in immobilized co-culture fermentation, activated carbon sponge was found to be a better support material compared than loofah sponge. The obtained biohydrogen production rate using immobilized co-culture on activated carbon sponge was 0.768 L H_2/h/$L_{substrate}$ at 24 h fermentation, approximately 45 % higher than using loofah sponge for the immobilization. In comparison with fermentation of free co-culture, the average biohydrogen production rate using co-culture immobilized onto activated carbon sponge was 67 % higher than that without immobilization. The highest cumulative and production rate of biohydrogen were achieved by the co-cultured bacteria *Escherichia coli* and *Clostridium sporogenes*, with 15.42 L of H_2 and 1.416 L H_2/h/$L_{substrate}$. The best fitting curve result for the cumulative biohydrogen production prove that the modified Gompertz equation fitted well with most experimental results. This finding would be useful for scaling up of biohydrogen production. In conclusion, the combination of activated carbon sponge and co-culture enhanced the biohydrogen production from pineapple residues. The activated carbon sponge was identified as a reasonable, easily obtained, and durable support material, which is suitable to be used in any plug flow bioreactor system in the future.

ABSTRAK

Kulit nanas adalah salah satu bahan mentah biomas yang berpotensi untuk pengeluaran biohidrogen. Cara yang paling mudah untuk menghasilkan biohidrogen daripada bahan lignoselulosik adalah melalui proses penapaian. Proses ini mesra alam dan menggunakan tenaga rendah tetapi mempunyai pengeluaran hasil yang rendah. Salah satu cara untuk meningkatkan penghasilan biohidrogen adalah dengan menggunakan lebih daripada satu spesies bakteria penghasil hidrogen, juga dikenali sebagai kultur bersama, dan disekatgerak dalam keadaan anaerobik yang stabil. Kesesuaian kultur bersama bakteria dan kestabilan bakteria ini di dalam matriks penyekatgerakan diuji untuk mencapai pengeluaran maksimum biohidrogen. Objektif kajian ini adalah untuk meningkatkan pengeluaran biohidrogen daripada kulit nanas menerusi proses penapaian kelompok menggunakan kultur bersama bakteria tersekat gerak yang paling sesuai. Kulit nanas dipilih kerana kelimpahan sumbernya dan mudah didapati. Tiga spesies bakteria penghasil H_2 yang berbeza telah dipilih iaitu *Escherichia coli, Enterobacter aerogenes* dan *Clostridium sporogenes* dan digunakan sebagai kultur tunggal atau digabungkan sebagai kultur bersama. Prestasi ketiga-tiga bakteria ini dalam bentuk sel bebas dan tersekat gerak kemudian dibandingkan. Bagi tujuan penyekatgerakan, span karbon aktif dan span loofah telah digunakan dan dibandingkan. Akhirnya, pengeluaran kumulatif biohidrogen dan kadar pengeluaran biohidrogen dianalisis dengan kajian kinetik. Persamaan Gompertz yang diubah suai disuaikan kepada kinetik pengeluaran biohidrogen kumulatif melalui aplikasi 'Solver' di dalam Excel. Semua proses penapaian dijalankan pada pH 7 dan suhu 32 ± 1 ° C, dengan 30% v/v inokulum daripada jumlah substrat dalam proses kelompok. Span karbon aktif didapati merupakan bahan sokongan yang lebih baik berbanding span loofah. Kadar pengeluaran biohydrogen yang diperoleh menggunakan kultur bersama bakteria tersekat gerak pada span karbon aktif adalah 0.768 L H_2/jam/$L_{substrat}$ pada 24 jam penapaian, sekitar 45% lebih tinggi dari penapaian menggunakan span loofah. Sebagai perbandingan dengan penapaian kultur bersama sel bebas, kadar pengeluaran biohydrogen bagi kultur bersama tersekat gerak pada span karbon aktif adalah 67% lebih tinggi daripada penapaian menggunakan kultur bersama sel bebas. Kadar kumulatif dan pengeluaran biohydrogen tertinggi diperolehi melalui kultur bersama *Escherichia coli* dan *Clostridium sporogenes* yang menghasilkan sebanyak 15.42 L H_2 dan 1.416 L/jam/$L_{substrat}$. Keputusan lengkuk terbaik untuk pengeluaran biohidrogen kumulatif membuktikan bahawa persamaan Gompertz yang diubahsuai bersesuaian dengan semua keputusan eksperimen. Penemuan ini berguna untuk penambahan skala pengeluaran biohidrogen. Kesimpulannya, kombinasi span karbon aktif dan kultur bersama meningkatkan pengeluaran biohidrogen daripada sisa nanas. Span karbon aktif dikenal pasti sebagai bahan sokongan yang berpatutan, mudah dan tahan lama untuk digunakan dalam mana-mana aliran sistem bioreaktor aliran palam pada masa hadapan.

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LIST OF ABBREVIATIONS

AGSBR	-	Agitated Granular Sludge Bed Reactor
AC	-	Activated Carbon
AFBR	-	Anaerobic Fluidized Bed Reactor
BF	-	Batch Fermentation
BPR	-	Biohydrogen Production Rate
BOD	-	Biological Oxygen Demand
$BioH_2$	-	Biohydrogen
CIGSB	-	Carrier-Induced Granular Sludge Bed
COD	-	Chemical Oxygen Demand
CSTR	-	Continuous Stirred Tank Reactor
DOE	-	Design of Experiment
EFB	-	Empty Fruit Brunch
FBR	-	Fluidized Bed Reactor
GC-TCD	-	Gas Choromatography – Thermal Conductivity Detector
H_2	-	Hydrogen
HPLC	-	High Performance Liquid Chromatography
HRT	-	Hydraulic Retention Time
GAC	-	Granular Activated Carbon
MBR	-	Membrane Bioreactor
MPIB	-	Malaysia Pineapple Industry Board
MSW	-	Municipal Solid Residue
n.d	-	Not determined
OD	-	Optical Density
OLR	-	Organic Loading Rate
PAC	-	Powder Activated Carbon
PBR	-	Packed Bed Reactor
PEG	-	Polyethylene Glycol
PET	-	Polyetheylene Terephtalate
PMMA	-	Polymethyl Methacrylate
POME	-	Palm Oil Mill Effluent

PVA	-	Polyvinyl Alcohol
PVC	-	Polyvinyl chloride
PVDF	-	Polyvinylidene Flouride
SMR	-	Steam Methane Reforming
SEM	-	Scanning Electron Microscopy
TCD	-	Thermal Conductivity Detector
TS	-	Total solid
TVS	-	Total Volatile Solid
UV	-	Ultraviolet
UASB	-	Up-Flow Anaerobic Sludge Blanket Bioreactor
UASBR	-	Up-Flow Anaerobic Sludge Bed Reactor
VFAs	-	Volatile Fatty Acids
VS	-	Volatile Solid

LIST OF SYMBOLS

°C	-	Celcius
CH_3COOH	-	Acetic Acid
CO_2	-	Carbon Dioxide
$C_6H_{12}O_6$	-	Glucose
coA	-	coenzyme
C:N:P	-	Carbon:Nitrogen:Phosphorus
cm	-	centimetre
d	-	day
EJ	-	Exajoules
G	-	Gram
h	-	hour
H_2SO_4	-	Sulphuric Acid
Kg	-	Kilogram
Ktoe	-	Kilo tonne of oil equivalent
L	-	Litre
M	-	Molar
MJ	-	Millijoule
Min	-	Minutes
Mol	-	mole
Mmol	-	Milimole
OD_{600}	-	Optical Density at 600nm
R_m	-	Maximum Rate
P_t	-	Cumulative Biohydrogen
P_m	-	Maximum Biohydrogen Production
Rpm	-	Revolutions per minute
w/v	-	weight per volume
%	-	Percentage

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CHAPTER 1

INTRODUCTION

1.1 Study Background

Hydrogen is one of potential clean energy to replace fossil fuels as it burns without creating any pollution and produces only water when it combusts. The high specific energy (120-140 MJ/kg) considered as flammable which suggests a possibility of high performance in internal combustion engines (Shafie et al, 2011; Li et al, 2012; Das et al, 2014). The hydrogen demand is expected to increase for the chemical industries and refineries needs, biofuel production, application as synthetic fuels and in fuel cells (Thengane et al, 2014). Hydrogen has a wide variety of application including fuels for automobiles, distributed or central electricity and thermal energy generation (Kotay, 2008; Taylor, 2013). Hydrogen can be converted into electricity via the fuel cell, particularly in the transportation sector. Other than that, hydrogen is also used in industrial applications including removal of impurities in oil refineries, production of electronic devices, reformation of petroleum distillate, ammonia synthesis and methanol production (Sekoai, 2017).

Hydrogen can be produced by various methods, from either fossil fuels or renewable sources like biomass. The derivation from fossil fuels mainly involved hydrocarbon reforming or pyrolysis process. Hydrogen production is globally dominated by steam reforming technology. This is because the process has low operational and production costs but gives high efficiency (produced 95 % hydrogen with 65–75 % efficiency) (Kalamaras & Efstathiou, 2013; Straka & Bi, 2012). However, the whole process is regarded as a complex process which is accomplished in two stages. This includes the production of syngas (H_2/CO gas mixture) and conversion of carbon monoxide into carbon dioxide and hydrogen by steaming. The process also needed high temperature (750–800 °C) for the reaction of steam and

methane to occur in the presence of heat and catalyst in the fired reactor. It also required high energy input (Eker and Sarp, 2017; Orozco-pulido, 2011), and the process released carbon footprint (Orozco-pulido, 2011).

Although reforming processes were already established, alternative processes from renewable sources such as biomass and water splitting had attracted researchers' attention. The most prevalent process was water electrolysis. It converted electrical energy into chemical energy by splitting water to H_2 and O_2 by passing electric current. However, the low energy efficiency that depended on high temperature and electrical supply limit the process (Orozco-pulido, 2011). Pyrolysis is another existing technology under thermochemical process used to produce syngas comprising of high hydrogen content (Wu & Williams, 2010). This is a method where biomass is heated and gasified at temperature range of 500–900 °C under 0.1–0.5 MPa pressure which took place in the absence of air and oxygen. Meanwhile, gasification is a variation of pyrolysis based on partial oxidation of the feedstock into a producer gas consisting of a mixture of hydrogen, methane, carbon monoxide, nitrogen, carbon dioxide, and higher hydrocarbons.

The shortcoming of high energy consumption processes had shifted researchers' attention into utilising biological methods as a better option. The use of the biological process has increased due to the awareness on sustainable development and waste minimisation (Nikolaidis & Poullikkas, 2017). The biological process, including biophotolysis, dark, and photo fermentation only required ambient temperature and pressure to operate. These processes could also utilise various waste materials as feedstock and contributed to waste utilisation. These different types of processes are shown in Figure 1.1 as adapted from (Nikolaidis & Poullikkas, 2017).

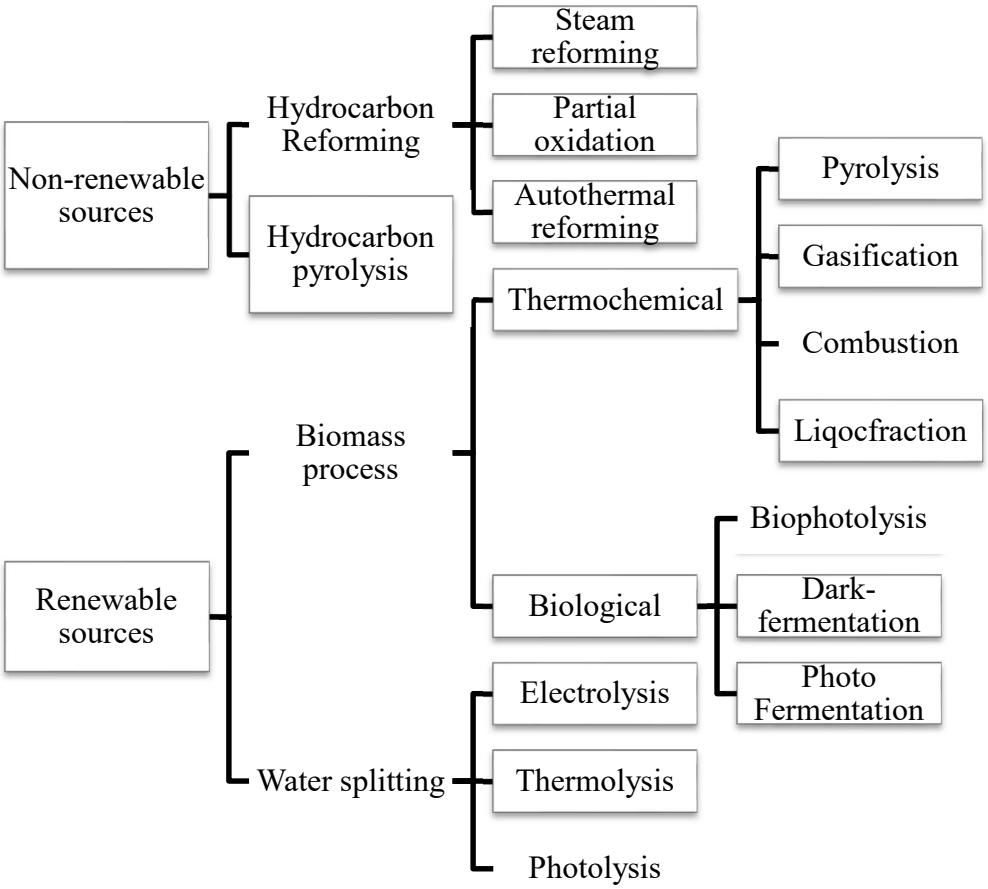

Figure 1.1 Hydrogen production technologies (Nikolaidis & Poullikkas, 2017)

Among all hydrogen production technologies, fermentative hydrogen production was considered an efficient and versatile biological process. It dealt with a wide range of biomass feedstock such as agricultural, agro-industrial, municipal, and food waste. In comparison with other hydrogen production technologies, biomass fermentation has 60–80 % overall efficiencies compared to the steam methane reforming (SMR) process (Khan et al, 2010). Fermentative hydrogen production from organic substrates has the advantage of clean energy production and waste reduction.

Over the years, there was an interest to use pineapple waste extract as a feedstock in the fermentation process to produce biohydrogen. The pineapple residues including peels, crown, skin, and pulp have high glucose content that could be suitable as a substrate for fermentation process (Kongjan, 2010). Approximately 70% of residues were usually found in pineapple plants, mainly from the leaves. Only 60% of the pineapple fruit was used by most pineapple processing industries, with majority of

the peels discarded. The high content of cellulose-based fibre in pineapple produced a lignocellulosic biomass which could be good and used as a green and cheap carbon source for hydrogen generation. In Malaysia, pineapples were grown in more than 10 thousand hectares' land with a total production of 250 to 300 thousand tons of pineapples between 2013 to 2015 (Halim, 2016). Pineapple industry in Malaysia has tremendous potential for development and expected to have high demand which was up to 700,000 metric tonnes per year in 2020 (Halim, 2016). The abundance of pineapple wastes will be produced in the future.

The most common fermentation process for hydrogen production are dark fermentation, photo fermentation, and combination of both. In the fermentation process, bacteria is mixed with the carbohydrate-based substrate to produce the desired products such as biohydrogen. Obligate and facultative anaerobes had the ability to convert carbohydrates under anaerobic fermentation to soluble gases product (Wu et al, 2008). The main criteria for this valuable outcome are hydrogen-producing bacteria which existed in: (i) pure culture known as H_2-producing bacteria species; (ii) co-culture, a combination of two or more H_2-producing bacteria species; and (iii) mixed culture which contained a variety of bacteria species found in natural communities like landfills, waste-water, and sludge compost (Philpott, 2011). Due to the productivity issue when utilising pure culture, co-culture bacteria were commonly used. Co-culture, which involved two known species or population was a better option instead of a mixed culture to avoid the complexity in establishing a stable system when there was more population involved (Goers et al, 2014).

Despite being the most feasible biological way in producing hydrogen, there were still technical hurdles in the fermentation process that could limit the technology development. Among the common yet highly recognised challenges were associated with productivity. Notably, the conversion and productivity of the process were still unsatisfactory to be scaled up for industrial production.

The most common problem highlighted in the literature review is the low productivity of biohydrogen despite various strategies had been implemented including the combination of production processes (Choonut et al, 2014), addition of nutrient supplement (Reungsang and Sreela-or, 2013; Cahyari, 2018), and a combination of multiple type of feedstocks (Robledo-Narvaez, 2013).

Although the co-cultures were easier to control and operate with a wide range of complex feedstock without sterilisation (Zhang, 2017), they still needed a stable environment for rapid growth and metabolism. This stable environment could be provided by implementing immobilisation technique, where microorganisms or H_2 producing bacteria were immobilised onto various biological or synthetic materials by several methods of immobilisation, including gel entrapment, surface attachments, and self-flocculation. This immobilisation technique was one of the practical strategies to improve biohydrogen production (Lin, 2018). Some researchers had tested and verified the potential of immobilisation based on the type of materials for microbial support (Nakatani et al., 2018; Wu & Chang, 2007; Kao et al., 2014; Kirli & Kapdan, 2016; Zhang et al., 2017). Immobilised materials must have high specific surface area, lower toxicity properties, and durable to harsh environmental conditions. The porous structure of the materials enabled the bacteria to grow freely to sustain their cell viability. At the same time, it enhanced the cell density and provided an efficient release of hydrogen from liquid phase to the headspace of the bioreactor (Kirli & Kapdan, 2016; Zhang et al., 2017)

The potential of immobilised microbial cells in biohydrogen production was discussed by Sekoai et al. (2017), who underlined different techniques of immobilisation including adsorption on surface materials, encapsulation by the matrix, entrapment within a matrix, and containment within a polymer. Immobilisation was said to be able to solve the problems raised in the use of suspended cells which possessed several advantages such as, (i) ability to withstand harsh fermentation condition; (ii) potential to increase substrate conversion efficiency; (iii) minimisation of microbial contaminations; and (iv) protect the microbial cells against shear stress caused by stirring during the operation of fermentation process. All the advantages might encourage the enhancement of biohydrogen production. The effectiveness of

immobilisation could be achieved by a proper selection of the support materials or matrix or carrier employed for the immobilisation. The large surface area, non-toxic materials, inexpensive and resistance towards fermentative by-products were the main properties to be considered.

1.2 Problem Statement

Several studies have shown that pineapple residues could be utilised as feedstock for biohydrogen production via fermentation process (Choonut et al., 2014; Reungsang & Sreela-or, 2013; Robledo-Narváez et al., 2013 and Chima 2017). Though, there are still areas for improvement to achieve higher H_2 production via various strategies. Choonut et al. (2014), added cellulose enzyme into pineapple peel feedstock parallel with immobilisation of pure culture onto loofah sponge with the maximum production of 1416 H_2 mL/L. Furthermore, Reunsang & Sreela-or (2013), found the production yield of biohydrogen of 1.83 mol H_2/mol glucose was achieved from anaerobic mixed cultures with the addition of $FeSO_4$ to overcome high acidic content in the pineapple waste substrate which claimed could cause adverse effects on hydrogen production. Whereas, Robeldo-Narvaez et al. (2013), indicated that only 15 % pineapple peels were used together with the mixture of 70 % sugarcane bagasse and 15 % of waste activated sludge for a production of 3.0 mmol H_2/g total solid. Despite the findings, a simple and low cost process would make the process more practical to be adopted at the industrial scale. Another study by Chima (2017), used different types of agricultural waste including pineapple peels as substrate to be utilised by purple non-sulphur photosynthetic bacteria of *R. sphaeroides* under natural sunlight. This study used mineral-salt solution for the bacteria isolation to allow the nitrogenase activity and showed that the pineapple peel was the most potential among other substrates used which yielded the most amount of H_2 of 11.8 mL/g at 227 h. However, the photofermentation process in the study needed to be placed outdoor for adequate sunlight. In another study, it was reported that biohydrogen could be produced by immobilised co-cultures of *Enterobacter* onto activated carbon (Zhang et al, 2017). However, the study was performed by using glucose liquid medium as the substrate.

In response to the above mentioned problems, this work proposed on a biohydrogen production using immobilised co-cultured H_2-producing bacteria of different species onto activated carbon sponge from lignocellulosic-pineapple peels fermentation. This method was expected to increase the bacteria survival rate, hence increases biohydrogen production from pineapple residue. The integration of co-culture method and immobilisation technique could be effectively achieved comparably or a higher production with similar fermentation process (Kumar et al., 2018; Hu et al., 2018). Though, this process has yet to be explored.

1.3 Research Objectives

This study aims to maximise the production of biohydrogen via batch fermentation process using pineapple peel as substrate. To achieve that, the following objectives were met:

i. To evaluate the performance of three types of H_2-producing bacteria that were co-cultured for biohydrogen production from pineapple peels in a 500 mL batch experimental set-up.
ii. To analyse the effect of the immobilised co-cultured bacteria attached to different support materials of loofah sponge and activated carbon sponge by adsorption technique.
iii. To assess the performance of free cells and immobilised co-cultured bacteria for biohydrogen production via fermentation using the modified Gompertz kinetic model.

1.4 Scope of Research

The scope of works based on each objective in four experimental stages are as follows:

i) Commercial pineapple peels waste.
   Fresh pineapple peels collected from a local market in Johor Bahru were used to be the substrate of the fermentation process. This pineapple peel substrate was characterised for pH, moisture content, total solids, volatile solids, COD concentration, lignin content, and glucose concentration.

ii) Pre-treatment process of pineapple peels substrate.
   Heat-shocked pre-treatments was applied and analysed in terms of glucose content performance and characterization of lignin. The heat pre-treatment was used to pre-treat the pineapple peels for the whole process of this batch fermentation.

iii) Immobilisation of free cell culture and co-cultured bacteria on support materials.
   The selected H_2-producing bacteria, namely *Clostridium sporogenes*, *Enterobacter aerogenes* and *Escherichia coli* were used in the form of single culture and co-culture. These bacteria were immobilised on loofah and activated carbon in single and co-cultured forms before being used in the fermentation process. The most potential sponge was selected for further fermentation of different co-cultures based on the ability to produce the highest biohydrogen. Scanning Electron Microscopy (SEM) analysis was conducted to observe the attachment of the co-culture on the selected sponge materials.

iv) Evaluation of kinetic performance of biohydrogen production.
   The progress of biohydrogen production by fermentation was assessed using the modified Gompertz kinetic model to predict the maximum biohydrogen production, the rate of biohydrogen formation, and the lag phase of the bacteria.

1.5 Research Significance

Production of biohydrogen through anaerobic fermentation was regarded as non-energy intensive and environmentally friendly processes that could play a significant role in future green energy. The reuse of waste in other applications eventually assisted to mitigate the environmental problems by reducing the number of residues dumped into landfills. This approach will also indirectly reduce the footprint of petroleum. Hence, the main contributions of this study included the demonstration of useful information on fermentation of pineapple peels by three types of hydrogen-producing bacteria that were co-cultured and immobilised to produce biohydrogen.

This work specifically had established potential immobilised co-cultured bacteria attached on activated carbon sponge for efficient fermentation process which was able to produce a comparable or higher productivity of biohydrogen. This work used a simple method with no additional supplement and nutrient to the substrate or inoculum, which was found workable to produce biohydrogen without producing methane by-product. To the best of the author's knowledge, this method of immobilising culture on a novel material of activated carbon sponge was never used for biohydrogen production by others.

The findings from this work provided insights for scaling up fermentation of immobilised co-culture on packed bed bioreactor. The activated carbon sponge surface was a suitable support material because it was easy to be packed in an up-flow packed bed reactor for continuous system. The kinetic performance of the batch fermentation provided a good guide for the prediction of biohydrogen production from the fermentation process, which is useful for scaling up study.

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