UNIVERSITI PUTRA MALAYSIA

GASIFICATION OF MICROALGAE CHLORELLA VULGARIS FOR SYNTHESIS GAS PRODUCTION

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By

ABDUL RAHEEM

Thesis Submitted to the School of Graduate Studies, Universiti Putra Malaysia, in Fulfilment of the Requirement for the Degree of Master of Science

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DEDICATION

I would like to dedicate this thesis with love and gratitude to my parents, wife, my sons (Adeel Ahmed and Khalil Rahman), my sisters, my brothers and longtime friends for their encouragement, unconditional love and support during my study.

Abstract of thesis presented to the Senate of Universiti Putra Malaysia in fulfillment of
the requirement for the Degree of Master of Science

GASIFICATION OF MICROALGAE *CHLORELLA VULGARIS* FOR SYNTHESIS GAS PRODUCTION

By

ABDUL RAHEEM

October 2015

Chairman : Mohd Razif Bin Harun, PhD
Faculty : Engineering

Microalgae have been used as a substrate for biofuel production due to numerous advantages, including fast growth rate, ability to grow with/without land and accumulate substantial amounts of carbohydrates, lipids and proteins. However, current production of alternative fuel from microalgal biomass (biodiesel and bioethanol) involves lengthy processing steps. Thermochemical conversion is proven technology for higher conversion efficiency of biomass into biofuel and shorter production period compared to other conventional methods such as biochemical. However, low production yield of synthesis gas (syngas) produced from the process hinder their conversion into a higher value of fuel products. This can be realized by optimizing its process conditions for high production yield of the syngas.

In this study, microalgal biomass, *Chlorella vulgaris,* is thermochemically gasified for syngas production. The study involves three stages include characterization of the *C. vulgaris* biomass, identification of the process parameters (reactor temperature, *C. vulgaris* biomass loading, and heating rate) and optimization for high syngas production.

The characterization of *C. vulgaris* biomass explains that the thermal degradation behavior of the biomass can be divided into three major stages; (1) moisture removal, (2) devolatization of carbohydrates, protein and lipids and (3) degradation of carbonaceous material. A degradation rate of 80% was obtained at the second phase of the combustion process in the presence of air whilst a degradation rate of 60% was obtained under N_2 atmosphere at the same phase. The biomass was further gasified for syngas production using a Temperature Programed Gasifier (TPG). The effect of three different process parameters, temperature (700, 800, and 900), *C. vulgaris* biomass loading (0.1, 0.25, and 0.5 g), and heating rate (5, 10, and 20 min^{-1}) were investigated. The maximum H_2 production was found at 800°C (45.9 ± 0.5 mol %, 0.35 mmol g^{-1}) with a biomass loading of 0.5 g (39.9 ± 1.5 mol %, 0.62 mmol g^{-1}). No significant effect of heating rate was observed on H_2 production. The activation energy values in air atmosphere, based on the Kissinger method, were evaluated to be 45.38 ±

1.0 kJ mol^{-1} (1st stage), 61.20 ± 0.5 kJ mol^{-1} (2nd stage) and 97.22 ± 0.5 kJ mol^{-1} (3rd stage).

Furthermore, the optimization of microalgal gasification for syngas production was conducted using a high temperature horizontal tubular furnace. Four response variables (H_2, CO, CO_2, and CH_4) were optimized under varying conditions of temperature (500-900), *C. vulgaris* biomass loading (0.6 - 2.5 g), heating rate (5 - 25 min^{-1}), and equivalent ratio (ER = 0.1 - 0.35). The optimization study was carried out using Central Composite Design (CCD). Temperature was found as the most significant process parameter influencing H_2 production followed by *C. vulgaris* biomass loading and heating rate. A maximum H_2 yield of 41.75 (0.66 mmol g^{-1}) mol % was obtained at a temperature of 703 , *C. vulgaris* loading of 1.45 g, heating rate of 22 min^{-1}, and ER concentration of 0.29. Statistical analysis showed that the generated models were sufficiently in agreement with the experimental results.

It was concluded that direct gasification of *C. vulgaris* biomass in the presence of air has a significant potential for commercial-scale production of syngas products under optimum economy. Hence the findings from this study are expected to resolve the problems that are being encountered with current conversion methods through the provision of numerous environmental and cost benefits.

Abstrak tesis yang dikemukakan kepada Senat Universiti Putra Malaysia sebagai
memenuhi keperluan untuk ijazah Master Sains

PENGEGASAN MIKROALGA *CHLORELLA VULGARIS* BAGI PENGHASILAN GAS SINTESIS

Oleh

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Mikroalga telah digunakan sebagai substrat untuk penghasilan bahan api bio kerana mempunyai pelbagai kelebihan, termasuk kadar pertumbuhan yang cepat, kebolehan untuk membiak dengan/tanpa memerlukan tanah dan keupayaan untuk mengumpul sejumlah besar karbohidrat, lipid dan protein. Walau bagaimanapun, penghasilan bahan api alternatif dari biojisim mikroalga (biodiesel dan bioetanol) melibatkan langkah-langkah pemprosesan yang panjang. Penukaran termokimia adalah teknologi yang dibuktikan lebih cekap untuk penukaran efektif biojisim ke bahan api alternatif dalam tempoh pengeluaran yang lebih pendek berbanding dengan kaedah konvensional lain seperti biokimia. Walau bagaimanapun, hasil pengeluaran gas sintesis (syngas) yang rendah daripada proses tersebut menghalangnya daripada menjadi produk bahan api yang bernilai tinggi. Ini boleh direalisisasikan dengan mengoptimumkan proses tersebut untuk pengeluaran hasil gas sintesis yang lebih tinggi.

Dalam kajian ini, biojisim mikroalga, *Chlorella vulgaris*, telah menjalani proses termokimia untuk penghasilan gas sintesis. Kajian ini melibatkan tiga peringkat termasuk pencirian biojisim *C. vulgaris*, mengenal pasti parameter proses (suhu reaktor, kemuatan biojisim, dan kadar pemanasan) dan pengoptimuman untuk pengeluaran gas sintesis yang tinggi.

Pencirian biojisim *C. vulgaris* menjelaskan bahawa tingkah laku degradasi terma biojisim boleh dibahagikan kepada tiga peringkat utama; (1) pembuangan kelembapan, (2) proses ñdevolatizationò karbohidrat, protein dan lipid dan (3) degradasi bahan berkarbon. Kadar penurunan sebanyak 80% telah diperolehi pada fasa kedua proses pembakaran di dalam udara manakala kadar degradasi 60% telah diperolehi di dalam atmosfera N_2 pada fasa yang sama. Seterusnya biojisim terus diproses untuk pengeluaran gas sintesis dengan menggunakan Pengegas berprogram suhu. Kesan tiga parameter proses yang berbeza iaitu suhu (700, 800 dan 900), pemuatan biojisim *C. vulgaris* (0.1, 0.25 dan 0.5 g), dan kadar pemanasan (5, 10 dan 20 min^{-1}) telah disiasat. Pengeluaran H_2 maksimum ditemui pada 800 (45.9 ± 0.5 mol%, 0.35 mmol g^{-1}) dengan muatan biojisim sebanyak 0.5 g (39.9 ± 1.5 mol%, 0.62 mmol g^{-1}). Tiada kesan ketara terhadap kadar pemanasan diperhatikan pada pengeluaran H_2. Nilai tenaga pengaktifan dalam atmosfera udara, berdasarkan kaedah Kissinger, telah dinilai pada 45.38 ± 1.0 kJ mol^{-1} (peringkat 1), 61.20 ± 0.5 kJ mol^{-1} (peringkat ke-2) dan 97.22 ± 0.5 kJ mol^{-1} (peringkat ke-3).

Tambahan pula, pengoptimuman pengegasan mikroalga untuk pengeluaran gas sintesis telah dijalankan dengan menggunakan tiub melintang relau bersuhu tinggi. Empat pembolehubah tindakbalas (H_2, CO, CO_2 dan CH_4) telah dioptimumkan di bawah keadaan suhu yang berbeza (500 - 900), kemuatan biojisim mikroalga (0.6 - 2.5 g), kadar pemanasan (5 - 25 min^{-1}), dan nisbah setara (ER = 0.1 - 0.35). Kajian pengoptimuman telah dijalankan dengan menggunakan Rekaan Komposit Pusat (CCD). Suhu didapati sebagai parameter proses yang paling penting mempengaruhi pengeluaran H_2 diikuti oleh pemuatan biojisim *C. vulgaris* dan kadar pemanasan. Hasil H_2 optimum 41.75 mol% (0.66 mmol g^{-1}) telah diperolehi pada suhu 703 , pemuatan biojisim *C. vulgaris* 1.45 g, kadar pemanasan 22 min^{-1}, dan kepekatan ER pada 0.29. Analisis statistik menunjukkan bahawa model yang dihasilkan adalah seiring dengan keputusan eksperimen.

Ia dapat disimpulkan bahawa pengegasan langsung biojisim *C. vulgaris* dengan kehadiran udara mempunyai potensi yang besar untuk pengeluaran gas sintesis berskala komersial yang lebih ekonomikal. Oleh itu dapatan daripada kajian ini dijangka akan menyelesaikan masalah yang sedang dihadapi dengan kaedah penukaran semasa dengan memberi pelbagai faedah alam sekitar dan kos yang lebih efektif.

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This thesis was submitted to the Senate of the Universiti Putra Malaysia and has been accepted as fulfillment of the requirement for the Degree of Master of Science. The members of the Supervisory Committee were as follows:

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respective model versus the experimental values. ( ): experimental values used for the calculation of the model. ( ): prediction line

LISIT OF ABBERIVATIONS

BOD	Biochemical oxygen demand
CCD	Central composite design
COD	Chemical oxygen demand
CV	Calorific value
CG	Conventional gasification
DTG	Derivative thermogravimetry
DME	Diethyl ether
EFB	Empty fruit bunch
ER	Equivalence ratio
FB	Fluidized bed
FID	Flame ionization detector
FTS	Fischer-Tropsch synthesis
GC	Gas chromatography
GHGs	Greenhouse gases
HHV	High heating value
HCV	Higher calorific value
HTG	Hydrothermal gasification
SCWG	Super critical water gasification
TGA	Thermogravimetric analyzer
TCD	Thermal conductive detector
TPG	Temperature program gasifier

CHAPTER 1

INTRODUCTION

1.1 Background of study

The demand of energy to satisfy global needs is continuously escalating. With the emergence of fossil fuels such as coal, oil and gas, the world increasingly became dependent on fossil fuel. Currently, the total world's economy relies o fuels as a reliable fuel sources for generation of electricity, heat, transportation fuel and other goods (Sarkar et al., 2012). It has been reported that more than 80% of energy derives from burning of fossil fuels (Demirbas and Demirbas, 2011). This tremendous increase have led to many concerns such as emission of greenhouse gases (GHG) and other harmful pollutants such as NO_X, SO_X and CO_X, resulting to air pollution, acid rain and greenhouse effect (Chen et al., 2011; Marques and Fuinhas, 2011). Fossil fuel reserves are likely to be depleted and make the fuel as unsustainable and nonrenewable energy sources. The dependency of this fuel has caused escalation in diesel, petroleum and electricity prices and subsequently initiated the necessity to explore alternative energy resources to support global ever-growing energy demands.

Synthesis gas (syngas), produced via advance technologies such as fuel cell and gas turbine, is one of the potential alternatives to substitute the existing fuels. It is produced from a thermochemical conversion technology particularly through gasification route. The method is highly capable to convert biomass into syngas with very low tar contents by utilizing various types of biomass such as corn, wheat, cotton stalks, and sugarcane (Tavasoli et al., 2009; Zabaniotou et al., 2010; Ahmed and Gupta, 2012). The biomass is used to convert into syngas under partial oxidation conditions. The syngas consists of hydrogen (H_2), carbon monoxide (CO), carbon dioxide (CO_2), and methane (CH_4). The two later compounds are the byproducts from the gasification reactions. This type of fuel provides various advantages, as it is suitable for Fischer-Tropsch (FT) process to produce diesel as well as methanol by chemical synthesis**.**

Furthermore, syngas is a substantial energy carrier fuel source and less harmful as compared to fossil fuel, thus the use of syngas for different applications such as power generation, transportation, boiler fuels and residual heating reduces greenhouse gases (GHGs) emissions and other pollutants into the atmosphere (Saxena et al., 2008; Jhon et al., 2010). Although syngas offers many advantages, a more sustainable feedstock to produce this fuel is required to meet the global energy demands. The first generation biofuel feedstocks such as grains, sugarcane, oil seeds, corn, and vegetable oil have been utilized to produce syngas. Those materials have achieved a commercial scale of production; however, their influence towards high value products particularly for food applications and requirement of large arable land has restricted the production of fuels such as syngas, biodiesel and bioethanol. For instance, apart from the risk that higher food prices may have serious negative impacts on food security, the demand for biofuels could place considerable additional pressure on the natural resource base, by harming environment and causing social consequences. Second generation biofuels referring to lignocellulosic biomass (non-food materials) have been progressed to evade existing problems related to the first generation biofuel sources (Chen et al., 2010), but these have not been commercialized due to technological impediments, cost effectiveness, and biomass collection networks (Nigam and Singh, 2011). Other problems associated with the current feedstock sources are ecological effects of soil erosion and deforestation especially of vegetation crops. The availability of feedstock is not ample to maintain existing fuel demands as well as its commercial exploitation. Hence, this contributes to a significant reason in rising production of biofuel prices and making them less appealing as an effective alternative energy source. Table 1.1 shows various types of feedstocks used for syngas production.

Table 1.1: Gasification feedstock for syngas production

Type	Biomass	Limitations	References
Sucrose	Sugar cane, Sugar beets, Soyabean	High moisture content, high water and fertilizer demands	Ensinas et al., 2007; Demirbas, 2007; Yoon et al., 2010
Starch based	Corn, wheat, grain, potatoes, barley	Moisture, pretreatment needed, harvesting and transportation cost	Antal et al., 2000; Chen et al., 2015; Tavasoli et al., 2009
Lignocellulosic material	(Woods, green grasses, straws)	High temperature resistant due to lignin presence, high cost of pretreatment	Balat, 2006; Caro et al., 2015; Dudynski et al., 2015; Naik et al., 2010

In order to produce more sustainable biofuel, third generation biofuel feedstock needs to be considered some criteria include i) lower production cost than fossil fuel, ii) provide high yield of biomass by using low area of land, iii) capable to improve the environmental quality by using CO_2 produced from different sources (Brennan and Owende, 2010). With that, microalgae provide a potential feedstock to overcome the existing problems associated with syngas production (Clarens et al., 2010; Mata et al., 2010).

Microalgae are non-flowering, autotrophic and unicellular photosynthetic micro-organisms, found in saline or freshwater environments. It is typically growing via photosynthesis process just like land based plants. Compared to the conventional bio-fuel producing crops, microalgae are likely to have much lower environmental impacts, when they are used to produce syngas. Microalgae have a high calorific value (HCV) 21.88 MJ kg^{-1} as compared to 18.96 MJ kg^{-1} for Empty Fruit Bunch (EFB), low density and low viscosity, those makes this material more suitable for syngas production than lignocellulosic materials (Ma and Yousof, 2005; Miao et al., 2004; Chen et al., 2013). Also, due to their inherently high in protein (42 - 55 %), carbohydrates (12 - 17 %) and lipid content (26 ±28 %), semi-steady state production and suitability grow in variety of climates; therefore microalgae provide a potential feedstock for biofuel production (Becker, 2007; Clarens et al., 2010).

1.2 Problem Statement

Environmental problems and economic security concerns associated with fossil fuel have triggered interest in using biofuels as an alternative to fossil fuels. The primary sources for current biofuel production are first generation and second generation biofuels. The first generation biofuel includes biomass such as sugar, starch, sugarcane, oil seeds and etc. However, it has some disadvantages as 1) needs arable land for their cultivation, 2) not enough efficient to fulfill energy demand, 3) more life cycle time, 4) food vs fuel problem, 5) increase in food prices. The second generation biofuel (lignocellulosic) includes wood, solid waste and energy crops those contain lignin which leads to difficulties within the conversion process and high conversion cost. Microalgae, a plant like microorganism, provide a potential feedstock to overcome the existing issue on biofuel production.

In term of technology, current production of alternative fuel from biomass involves lengthy processing steps. Hence choosing the right conversion technology is an important factor to make biofuels produced in sustainable, environmentally friendly and cost efficient process. One of the key options is to produce biofuel by using thermochemical i.e. gasification. The method provides higher conversion efficiency of biomass into biofuels, and a shorter production period compared to the conventional method such as biochemical method. Current microalgal biomass gasification use either fixed or fluidized bed- type reactors without focusing on optimization of process parameters. These reactors create major issues relating to carbon loss and scalability hence makes the reactors less viable for commercial scale applications. Therefore, this study explores the gasification of microalgal biomass (*C. vulgaris*) for synthesis gas production using a horizontal tubular reactor. The horizontal tubular reactor provides advantages in terms of low cost, simple operation, easy to control reaction conditions and higher carbon conversion rate (Chopra and Jain, 2007). An engineering approach focusing on detailed optimization for various process variables to maximize the syngas yield production was employed in this study.

1.3 Research objectives

The main objective of this study is to investigate the potential of *C. vulgaris* biomass as a feedstock for synthesis gas (syngas) production via gasification process. The objectives are divided into two sub-objectives include:

1. To characterize the properties of *C. vulgaris* biomass as a potential feedstock for syngas production via gasification process.

2. To optimize the effect of different process variables such as reactor temperature, biomass loading, heating rate and equivalence ratio (ER) for high syngas production from *C. vulgaris* biomass.

1.4 Scope of study

The proposed research of *C. vulgaris* gasification for syngas production was carried out in three stages; characterization of the *C. vulgaris* biomass for the thermochemical conversion, investigation of the effect of different parameters such as reactor temperature, *C. vulgaris* biomass loading, heating rate and ER during gasification and optimization of syngas production from *C. vulgaris*. The details scope of each objective is stated below:

1.4.1 Characterization of the *C. vulgaris* biomass for thermochemical conversion

The characterization study covers four different types of analysis include proximate, ultimate, higher heating value and thermal degradation of *C. vulgaris* biomass**.** Different parameters are applied according to the analysis standard methods. At the end of the study, a thermogravimetric profile and biomass characteristic is established and applied to the gasification process.

1.4.2 Investigation of different parameters on *C. vulgaris* biomass gasification

The main objective of this study is to investigate the effect of different gasification process parameters include reactor temperature (500, 600, 700, 800 and 900 (), *C. vulgaris* biomass loading (0.6, 1, 1.5, 2, 2.5 g), heating rate (5, 10, 15, 20, 25 (min $^{-1}$) and ER (0.1, 0.2, 0.26, 0.3 and 3.5) on response variables from *C. vulgaris* gasification. The gasification experiments were carried out in two different reactor types; (i) Temperature Program Gasifier (TPG) and (ii) High temperature quartz tube furnace. Results from the biomass gasification under different treatment conditions in TPG were used as a reference for the optimization study.

1.4.3 Optimization of *C. vulgaris* biomass gasification

Central composite design (CCD) approach was used to design the optimization study of *C. vulgaris* biomass gasification. The optimized parameters resulting high syngas production from gasification of *C. vulgaris* biomass will ensure a right direction towards the achievement to use syngas for different applications.

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