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GASIFICATION OF LIPID-EXTRACTED ALGAE AND FUCOIDAN-EXTRACTED SEAWEEDS FOR SYNGAS PRODUCTION

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By

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

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April 2019

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Lipid-extracted algae (LEA) are the by-products of algal lipid extraction that are used in many applications, for instance, biogas generation, ethanol manufacturing, animal feed, fertilizer, methane and as hydrogen fuel. LEA are less utilized in gasification and fucoidan-extracted seaweed residue is usually disposed after the extraction, hence, this study employed these algae residues via gasification, which is a thermochemical conversion technology of biomass into synthesis gas (syngas) to minimize the underutilization issue. The potential of lipid-extracted *Nannochloropsis gaditana* algae (LEA) and fucoidan-extracted algae that consist of mixture of *Sargassum sp*., *Padina sp*., and *Enteromorpha sp*. algae for synthesis gas production via gasification is investigated in this study. The effects of varying different process parameters; temperature and biomass loading on the synthesis gas composition, mainly H_2 and CO yields are evaluated. The characterization of the samples were carried out in Fourier Transform Infra-Red (FTIR), Thermogravimetric analysis (TGA), elemental analysis and bomb calorimetry test. Kinetics study of both algae residues are carried out using Kissinger-Akahira-Sunose (KAS) and Flynn-Wall-Ozawa (FWO) models that showed the degradation behaviour of the algae residues at high temperature, as indicated by the activation energy (E_a) at different degradation (α), proximate analysis and average degradation rate (AR). The gasification experiments were performed at different process parameters; temperature (600, 700, 800, 900 and 1000 ^oC), sample loading (0.3, 0.4, 0.5, 0.6 and 0.7 g) and equivalence ratio (ER) (0.1, 0.2, 0.3, 0.4 and 0.5) at fixed holding time (30 minutes). Yield of H_2 and CO were found to increase with temperature due to the effect of oxidation, water-gas, water-gas shift and Bouduard reactions. Increasing ER value decreased H_2 and CO yields due to the boosted oxidation of carbon that produced more CO_2. Sample loading increment resulted in increment of H_2 yield and decrement in CO yield, however, it did not significantly affect the syngas compositions, since it did not involve in the gasification mechanism. The same phenomena were observed for the data obtained from the simulation. Experimental gasification via Central Composite Design (CCD) approach revealed that optimized conditions for both samples

gasification were 718.77℃, 0.3 g loading and 0.24 ER value and 766.99℃, 0.7g loading and 0.1 ER value for LEA and seaweeds residue, respectively. At this conditions, the yield of H_2 and CO were 36.38 mol% and 13.28mol% respectively, for LEA and 47.99 mol% and 26.05 mol% respectively, for seaweeds residue. The experimental H_2 and CO yields were largely deviated from the simulation data, as observed from the root mean square error (RMSE) values. Thus, enhancements of the experimental facilities and limitations of Aspen Plus simulation could be done to improve the RMSE values of the largely deviated data.

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

PENGEGASAN ALGA TEREKSTRAK LIPID DAN RUMPAI LAUT TEREKSTRAK FUKOIDAN UNTUK PENGHASILAN GAS SINTESIS

Oleh

SITI NUR ATIKAH BINTI MAHAMUD

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Alga terekstrak lipid (LEA) adalah produk sampingan daripada proses pengekstrakan lipid yang digunakan dalam banyak aplikasi, antaranya, penghasilan biogas, ethanol, makanan haiwan, baja, gas metana dan gas hidrogen. Penggunaan LEA dalam proses pengegasan adalah kurang manakala rumpai laut terekstrak fukoidan biasanya dilupuskan selepas proses pengekstrakan, oleh itu, kajian ini menggunakan kedua-dua residu alga ini untuk pengegasan, iaitu teknologi penukaran biojisim secara termokimia kepasa gas sintesis untuk menangani masalah ini. Potensi LEA dari spesis *Nannochloropsis gaditana* and residu rumpai laut yang mengandungi campuran daripada spesis *Sargassum sp*., *Padina sp*., dan *Enteromorpha sp*. dalam penghasilan gas sintesis disiasat dalam kajian ini. Kesan memanipulasikan parameter-parameter proses, iaitu suhu dan muatan terhadap komposisi gas sintesis; H_2 dan CO dinilai. Pencirian kedua-dua sampel telah dijalankan melalui Fourier Transform Infra-Red (FTIR), Thermogravimetric analysis (TGA), analisis elemental dan ujian kalori bom. Kajian kinetic telah dijalankan menggunakan model-model Kissinger-Akahira-Sunose (KAS) dan Flynn-Wall-Ozawa (FWO) yang telah menunjukkan kelakuan degradasi kedua-dua residu alga pada suhu tinggi, seperti yang dinyatakan oleh tenaga pengaktifan (Ea) pada degradasi (α) yang berbeza, analisis proksimat dan purata kadar degradasi (AR). Proses pengegasan telah dijalankan pada parameter proses yang berbeza; suhu (600, 700, 800, 900 dan 1000°C), muatan sampel (0.3, 0.4, 0.5, 0.6 dan 0.7 g) dan nisbah keserataan (ER) (0.1, 0.2, 0.3, 0.4 dan 0.5) pada tempoh masa 30 minit. Hasil H_2 dan CO telah meningkat bersama suhu akibat kesan proses-proses pengoksidaan, air-gas, penukaran air-gas dan Bouduard. Peningkatan nilai ER menurunkan hasil H_2 dan CO sebagai kesan daripada galakan proses pengoksidaan karbon yang telah meningkatkan hasil CO_2. Peningkatan muatan sampel mempamerkan peningkatan hasil H_2 dan CO, walaubagaimanapun, ianya tidak memberi perubahan yang signifikan kepada komposisi gas sintesis kerana ianya tidak terlibat secara lansung dalam mekanisma proses pengegasan. Fenomena yang sama ditunjukkan oleh data

daripada simulasi dalam Aspen Plus. Eksperimen-eksperimen pengegasan melalui Rekabentuk Komposit Tengah (CCD) telah menunjukkan keadaan optimum bagi pengegasan kedua-dua residu alga ialah suhu 718.77℃, muatan 0.3 g and nilai ER 0.24 dan suhu 766.99℃, muatan 0.7g dan nilai ER 0.1, masing-masing untuk LEA dan residue rumpai laut. Pada keadaan ini, hasil H_2 dan CO adalah masing-masing 36.38 mol% dan 13.28 mol% untuk LEA dan 47.99mol% dan 26.05 mol% untuk residu rumpai laut. Hasil H_2 dan CO daripada eksperimen mempunyai perbezaan yang besar daripada simulasi, seperti yang dapat diperhatikan dari nilai-nilai kesilapan akar min kuasa dua (RMSE). Oleh itu, penambahbaikan kemudahan eksperimen dan had dalam simulasi Aspen Plus boleh dilakukan untuk memnambahbaik nilai-nilai RMSE tersebut.

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Declaration by graduate student

I hereby confirm that:

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This is to confirm that:

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LIST OF ABBREVIATIONS

CCD	Central composite design
DTG	Derivative thermogravimetry
FTIR	Fourier Transform Infra-Red
GC	Gas chromatography
LEA	Lipid-extracted algae
NO_X	Nitrogen oxides
PEAR	Post-extracted algal residue
PM	Particulate matter
PUFA	Polyunsaturated fatty acids
SCWG	Supercritical water gasification
SO_X	Sulphur oxides
SWE	Subcritical water extraction
TGA	Thermogravimetric analysis
TPG	Temperature Program Gasifier

CHAPTER 1

INTRODUCTION

1.1 Background

Fossil fuels are the world's primary energy sources, accounted 80% of energy consumption for the globe, in which 50% is utilized in transportation sector alone (Chakraborty et al., 2012). fuels, including oil, coal and natural gas, are extensively used in many applications, such as fuel for transportations, electricity and thermal energy generation (Brennan & Owende, 2010a). The utilization of coal, oil and natural gas emits hazardous gases that have severe impact to the living things and the environment. Burning of fossil fuels emits CO, CO_2, CH_4, NO_X, SO_X and particulate matter (PM) that further contribute to acid rain, haze, climate change, global warming and greenhouse effects (Lehmann et al., 2006; Chisti, 2008; Benemann et al., 2002; Gouveia & Oliveira, 2009). Burning of oil such as petrol and diesel contributes up to 30% of the overall CO_2 emission into the atmosphere (Janssens-Maenhout et al., 2017). These facts showed that reliance upon fossil fuels must be escaped, since they are unsustainable energy sources due to the consequences to living things and the environment (Brennan & Owende, 2010a).

This phenomenon has caused exploration on renewable energy sources for energy production, targeting to find efficient, sustainable, and cost-effective fuels with lower emission of hazardous pollutants (A. Singh et al., 2010). Biomass-derived biofuels such as methanol, ethanol, biodiesel, hydrogen, methane and Fischer-Tropsch diesel have been reported as the potential option for alternative fuels (Chakraborty et al., 2012; A Demirbas, 2005). First generation biofuels derived from terrestrial food crops such as rapeseed oil, sunflower, maize, sugar cane, soybean, wheat, barley, palm oil and sugar beet is introduced to substitute fossil fuels, which then created food-fuel crisis (Vassilev & Vassileva, 2016; Biller, 2013). However, due to the crisis, the sustainability of the biofuels are questioned, hence, second generation biofuels are established (Moore, 2008; Chakraborty et al., 2012). Second generation biofuels originated mostly from non-food crops lignocellulosic biomass such as agricultural waste, wood chips, forest residues, municipal solid waste and sewage sludge (Vassilev & Vassileva, 2016; Noraini et al., 2014). The efficiency of second generation biofuels production became a serious issue to be solved, since they require more advanced technologies of producing the biofuels (Noraini et al., 2014; Chakraborty et al., 2012).

Algal biofuels, known as third generation biofuels, are introduced to overcome the drawbacks of first and second generation biofuels (Ranjith Kumar et al., 2015). Algae have gained interests from many researchers for alternative biomass for fuel production due to their unique features: (1) high reproduction rate (2) high accumulation of lipid (3) less/ no arable land requirement (4) easy to cultivate in fresh water, salt water, brackish water and wastewater (5) high carbon-fixation efficiency (6) high calorific value, low density and low viscosity and (7) ability to produce up to ten times biofuel per acre in comparison with traditional biofuel crops (Miao, Wu, & Yang, 2004).

The algal biofuels processing causes accumulation of post-extracted algal residue (PEAR), lipid-extracted algal (LEA), or extracted algal leftover that require high cost of disposal (Chang et al., 2014). The extracted algal leftover still contains carbohydrate, protein and fraction of un-extracted lipid, which can be further processed to produce value-added products (Maurya et al., 2015). In this study, the leftover biomass obtained from extraction process is used for synthesis gas (syngas) production via gasification process. The gasification process produces hydrogen (H_2) and carbon monoxide (CO) as main products in the producer gas and other gases, depending on the gasification conditions (Romar, 2015; Brennan & Owende, 2010b). Although microalgal gasification have been extensively studied by researchers at various operating conditions for different algae species to yield high quality fuel (Nurdiawati et al. 2018; Ebadi et al. 2017; Raheem et al. 2018) however, little work have been reported on utilising algal residue from the extraction process for synthesis gas production. This study also optimizes the effect of different process variables for high synthesis gas production from algal leftover biomass and further kinetically study on the behaviour of the gasification process.

1.2 Problem Statement

The dwindling of fossil fuels resources is a serious issue as the world's population is relying upon them as their main sources of energy. Fossil fuels such as oil, natural gas, and coal are burnt to release the chemical energy stored in them that emit hazardous pollutants, for instance, CO_2, CO, SO_X, NO_X, CH_4 and particulate matter (PM). Finding an alternative energy source to substitute fossil fuels is crucial to minimize the adverse effects of fossil fuels burning that led to the introduction of first generation biofuels derived from food crops that created food supply and prices crisis. Second generation biofuels are found to overcome the limitations of first generation biofuels. However, second generation biofuels are insufficient to meet global demand for fuel and there is no established technology that could do the biomass to fuel conversion effectively (Nigam & Singh, 2011; Noraini et al., 2014).

Hence, further exploration on renewable energy sources had created a new discovery on the potential of microalgae as biofuel sources. Microalgal-derived biofuels are known as the third generation biofuels and their introduction is aiming to overcome the disadvantages of first and second generation biofuels. Algal biofuels manufacturing created the lipid-extracted algae (LEA) as the by-product which require high cost of disposal. Therefore, utilizing the leftover is more cost-effective and cause less harm to the environment.

One of the option to utilize the LEA is converting them for synthesis gas production via gasification process. Lu and Savage investigated supercritical water gasification on *Nannochloropsis sp.* LEA that yielded CH_4 as the major gas, meanwhile high H_2 yields ranging from 27-74.7 mmol-H_2/g LEA were obtained from catalytic steam gasification of *Botrycoccus braunii* LEA, *Scenedesmus almerienis* LEA and *Nannochloropsis oculata* LEA (Watanabe et al., 2015; Diaz-Rey et al., 2014; Duman et al., 2014). However, tar formation is a major challenge in steam gasification, hence, the selection catalyst selection is crucial for high tar conversion to synthesis gas. Air gasification of *Chlorella sp*. LEA in a thermogravimetric analyser yielded activated carbon as the desired product (Chang, Tsai, Li, & Chang, 2015). LEA from green algae species are also directly used as fish and ruminants feed and as fertilizer in agricultural industry that reduced the dependence on chemical fertilizer (Mona Mirsiaghi et al., 2016; Maurya et al., 2015; Rothlisberger-Lewis et al., 2016). Application of LEA as feedstock for pyrolysis in a fluidized bed reactor has created bio-oil and bio-char (Sanchez-Silva et al., 2013). Anaerobic digestion and anaerobic fermentation of *Scenedesmus sp.* LEA has proven to produce biogas that consists of methane (CH_4) as the major gas (Ehimen et al., 2011; Hernández et al., 2014). For seaweeds residue, this biomass is underutilized in value-added products manufacturing due to unavailability of the information on the applications of seaweeds residue in literature. Therefore, the potential of the seaweeds residue for syngas production via gasification will be discovered in this study.

This study aims to convert the LEA and seaweeds residue obtained from different extraction processes for synthesis gas production via conventional air gasification process. This creates an alternative path of application of algae residues for synthesis gas production that does not only solving the underutilization issue of the algae residues, but makes the overall algal processing becomes more economically and environmentally sustainable.

Optimization studies are performed experimentally and theoretically in Design Expert 7.7 software and Aspen Plus V8.8 software, respectively. Experimental optimization is aiming to find the optimized conditions for high syngas yield, focusing on H_2 and CO yields. Theoretical optimization verifies the validity of the experimental data based on the simulated gasification process performed at similar conditions as the gasification experiments. Both optimization studies are crucial in determining optimum syngas yield and theoretical syngas yield at certain process parameters. The air gasification process is simulated in Aspen Plus V8.8 software which has been used for modelling, simulation, optimization, sensitivity analysis and economic evaluation of chemical processes (Sun, 2014). Aspentech provides a detailed guide on the modelling of fixed bed coal gasification that is used as the main reference in this study. There are a few gasification of biomass simulation carried out in other studies, for instance, brown seaweed gasification (Kaewpanha et al., 2014), biomass (Sekhar et al., 2012), municipal solid waste (Karthik Ramakrishnan and Yang, 2014). However, few Aspen Plus gasification modelling were carried out on algae biomasses and none of the literatures reported on the fixed bed air gasification of algae biomasses, hence, this study builds a gasification model for LEA and seaweeds residue that is reproducible for gasification of other algae residues.

1.3 Research objectives

The objectives of this study are divided into two parts:

1. To investigate the potential leftover LEA and seaweeds residue as feedstocks for synthesis gas (syngas) production via gasification process under different operating conditions.
2. To evaluate the effect of different parameters on synthesis gas production from LEA and seaweeds residue.

1.4 Research scope

The study on gasification of algal leftover are carried out in four stages; (1) samples preparation (2) characterization of the algae residues via elemental analysis, Fourier Transform Infra-Red (FTIR) for functional group determination, thermogravimetric analysis (TGA) for thermal decomposition study and bomb calorimetry test for calorific value determination (3) gasification at different parameters; temperature (600, 700, 800, 900 and 1000℃), loading (0.3, 0.4, 0.5, 0.6 and 0.7 g) and equivalence ratio, ER (0.1, 0.2, 0.3 0.4, 0.5) and (4) experimental and theoretical optimization of the gasification process for the algae residues in central composite design (CCD) approach in Design Expert 7.7 software and in Aspen Plus V8.8 software, respectively. Further detailed information about each stage is as described below:

1.4.1 Sample preparation

The solid residues from lipid extraction and fucoidan extraction were filtered out from the mixture of solid and liquid residue and were put in a separate container. Both algae residues were then underwent drying process for 48 h in an oven at 105 ℃. Next, both samples were then crushed in a mortar and pestle prior to sieving to 300 micron size.

1.4.2 Characterization of all algae residues

This study utilized algal leftover biomass obtained from different extraction processess; (1) Lipid extracted algae (LEA) from *Nannochloropsis gaditana* obtained from lipid extraction obtained from (Bernard, 2016) (2) Mixture of seaweeds residue from *Sargassum sp.*, *Padina sp.*, and *Enteromorpha sp.* from fucoidan extraction. The characterization includes ultimate and proximate analysis for C, H, N, S and O as well as moisture, volatile matter, fixed carbon and ash content determination, respectively, thermal degradation study via thermogravimetric analysis (TGA), calorific value determination via bomb calorimetry test and functional group determination via Fourier Transform Infra-Red (FTIR).

1.4.3 Gasification of algae residues at different parameters

Gasification of LEA and seaweeds leftover are carried out under three different experimental parameters; temperature (600,700, 800, 900, and 1000 ºC), algae residues loading (0.3, 0.4, 0.5, 0.6, and 0.7 g) and equivalence ratio, ER (0.1, 0.2, 0.3, 0.4, 0.5) using compressed air as the gasifying medium. The experiments are performed in two stages; conventional gasification experiments and experimental optimization of LEA and seaweeds residue gasification to study the effect of varying the process parameters; temperature (600, 700, 800, 900 and 1000℃) and loading (0.3, 0.4, 0.5, 0.6 and 0.7 g) to the syngas compositions (H_2 and CO). All gasification experiments are carried out in a vertical fixed bed temperature programmed gasifier (TPG) and the compositions of syngas collected in gas collection bag obtained is analysed in a gas chromatography with flame ionization detector (GC-FID).

1.4.4 Experimental and theoretical optimization of the gasification process

The second part of the gasification experiments is the experimental optimization carried out via central response surface composite design (CCD) approach in Design Expert 7.7 software, aiming to obtain optimized conditions that produce high syngas yield for both LEA and seaweeds residue. Aspen Plus simulation excludes the ER, hence, the effect of ER on syngas compositions is not studied and the range of the ER values are determined from Raheem et al.

Theoretical optimization is performed in Advanced System for Process Engineering (Aspen) Plus V8.8 software using similar process parameters as used in the experiments and the results are compared with the ones obtained experimentally. However, the input parameters in Aspen Plus are scaled up 100 times from the experiment, since Aspen Plus is used to simulate chemical processes in large scale. Root mean square error (RMSE) calculation is performed for data validation of both experimental and theoretical optimization.

In the later part of this thesis, lipid-extracted *Nannochloropsis gaditana* and fucoidan-extracted mixture of seaweeds are addressed as LEA and seaweeds residue, respectively.

1.5 Significance of study

This study converts LEA and seaweeds residue to synthesis gas (syngas) that can be used in the production of biofuels, e.g. pure hydrogen fuel, bioethanol, dimethyl ether (DME), biodiesel and bioethanol. The feedstocks for gasification used are by-products of microalgal lipid extraction and seaweed fucoidan extraction. Hence, utilizing these sources is seen to be advantageous to solve the underutilization of algae residues after lipid and fucoidan extraction, thus, producing value-added products from the algae waste. In addition, most of polysaccharides are expected to remain in the LEA after lipid extraction, therefore the conversion of LEA into value-added products enhances the effectiveness of algal processing. As for seaweed residue, to the best of our knowledge, no study on the consumption of the residue after bioactive compounds extraction available in literature, thus, this study discovers the potential of the seaweeds residue for syngas production.

1.6 Thesis outline

Chapter 1 briefly introduces the background of the algae and points out the need of performing gasification of the algae leftovers. Chapter 2 presents more detailed information on the feedstock of gasification and the gasification process for syngas production. This chapter also elaborates on using Aspen Plus software in the gasification process optimization. Chapter 3 provides methodology for the whole study that can be divided into characterization of algae leftovers, gasification experiments and optimization of the gasification process. Chapter 4 presents and discusses the results from all parts of the study as well as validates the optimization studies done experimentally and in Aspen Plus software. Chapter 5 concludes all findings from the study and points out recommendations to improve the accuracy of the results obtained from the study. References are listed out according to the literatures cited in the thesis. Last but not least, appendices are also provided at the end of the thesis for more reference on the results presented in chapter 4.

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Conference Papers Presented (Orally)

R.A. Ilyas, S.M. Sapuan, M.R. Ishak, E.S. Zainudin and **M.S.N. Atikah**, Degradation and physicochemical properties of SPNFCs/SPS Bionanocomposite, Presented at *the First International Conference on Safe Biodegredable Packaging Technology (SafeBioPack 2018),* 24-26 July 2018, Might Partnership Hub, Cyberjaya, Malaysia, Abstract published in **Book of Abstract**, pp. 45.

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R.A. Ilyas, S.M. Sapuan, M.R. Ishak, E.S. Zainudin and **M.S.N. Atikah** Nanocellulose reinforced starch polymer composites: A review of preparation, properties and Application, *Proceedings of the 5th International*

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