Utilization of Empty Fruit Bunch Fiber of Palm Oil Industry for Bio-Hydrogen Production

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Abstract— Empty fruit bunch fiber (EFB_f) is a type of biomass waste generated by the palm oil industry, which accounts for approximately 22-23% of the total fresh fruit produced to make CPO. Currently, the total waste of EFB_f in Indonesia stands at about 5 million tons per year. It is necessary, therefore, to find solutions for utilizing this abundance. One alternative is to produce bio-hydrogen from the EFB_f bio-mass waste. The early stage of the process was to convert EFB_F biomass waste into bio-ethanol, which could then be subjected to high temperatures in a 'cracking' process to produce bio-hydrogen. The characteristics of the bio-ethanol were analysed, and it was used as Bio-ethanol Reformer, a raw material in the cracking process. The results showed that a pre-treatment process using sodium hydroxide at a concentration of 6% for 60 minutes resulted in an 86.69% elimination of lignin. Pre-treated EBF_f bio-mass waste hydrolysed using sulphuric acid at a concentration of 6% for 60 minutes, produced 0.6054% glucose. The fermentation process showed 6.58% bio-ethanol. Based on the characteristics of the bio-ethanol produced in this experiment, a simulation calculation for the production of bio-hydrogen was then performed using the cracking process of bio-ethanol with specified process conditions. The simulation calculations showed that the yield of bio-hydrogen in the cracking process reached 21.4%.

Keywords— empty fruit bunch; bio-ethanol; cracking; bio-hydrogen; simulation

I. INTRODUCTION

Continuous use of fossil fuels has led to dwindling supply and has triggered an energy crisis. Efforts are being made to avoid this crisis with the development of alternative energy sources. New technologies in alternative energy from renewable resources are in the process of being studied more widely. An example is the conversion of lignocellulosic feedstock to bio-energy, known as second-generation alternative energy. Production of bio-ethanol from lignocellulosic raw materials such as oil palm empty fruit bunch, water hyacinth, palm frond, banana bark, straw morning, maize straw, wheat straw and others is now being studied.

At the same time, the automotive industry is also developing various kinds of products using conventional fossil fuels such as gasoline and diesel, while development continues in the field of electric vehicles and with alternative fuels such as hydrogen. In order to support the use of hydrogen fuel in a fuel cell, many researchers are developing a variety of methods for producing hydrogen. Hydrogen is an environmentally friendly fuel which does not cause air pollution, the end product of the combustion process being water.

One method for producing bio-hydrogen is the development of lignocellulosic feedstocks to support the automotive industry in the production of hydrogen-fuelled vehicles. This research develops the production of bioethanol from oil palm empty fruit bunch, which then undergoes a termination process for cracking the carbon chain to produce hydrogen for use to support the development of the automotive industry.

A. Oil Palm Empty Fruit Bunch (EFBf)

Indonesia is one of the largest producers of palm oil in the world. Annual production of crude palm oil (CPO) in Indonesia has reached 19.76 million tons [1]. Indonesia also produces approximately 20.7 million metric tons / year [1] of waste oil palm empty fruit bunch (EFB_f). EFB_f is a biomass waste produced by the milling of palm oil. Total EFB_f produced ranges from 200-600 tonnes/day depending on the

volume and capacity of palm oil produced [1]. EFB is very abundant and has not been optimally utilised [2]. It is, therefore, necessary to find a solution to increase the economic value of the EFBf without causing environmental pollution. Typically, EFBf is simply burned or dumped in the area around the oil palm plant. Utilisation is still conventional, with the product normally only being used as compost. Efforts to discover a solution to optimal utilisation of EFBf is still being carried out in order to render the EFBf a valuable product.

The composition of the compounds found in EFB_f is approximately 34.37% lignin, 39.13% cellulose and 23.40% hemicellulose [3]. Total carbohydrates contained in EFB_f are approximately 62.53%. This relatively high number means that it can be converted into other derivative products such as glucose by hydrolysis of the cellulose. The glucose can then be fermented into biofuels such as biogas, bio-ethanol and other bio-hydrogen products [3], [4].

B. Bio-ethanol Production from Oil Palm Empty Fruit Bunch (EFB_f)

Conversion of oil palm empty fruit bunch into bio-ethanol can be carried out by various methods, whether physical, chemical, biological or any combination thereof. In this study, the production of bio-ethanol was divided into three stages: pre-treatment, hydrolysis, and fermentation. Pretreatment aims to eliminate lignin, while also aiming to convert the EFB_{f} into glucose by cellulose hydrolysis, and glucose in the subsequent fermentation into bio-ethanol.

The pre-treatment process uses a combination of chemical and physical methods with an autoclave. This method is known to be effective in the removal of lignin from the EFB. Chemicals used in this method are in the alkali classification (NaOH, KOH, Ca(OH)₂), ammonia, peroxide, organosolv and others. In general, lignin does not react chemically with the substances used, but it can be dissolved in the chemicals. At the beginning of the process of lignin termination from carbohydrates, the termination process happens very quickly. Loose lignin will first be dissolved by the chemicals, it then condenses and undergoes polymerisation returning to bond with other lignin. At the end of the pre-treatment process, insoluble lignin remains. The solubility of lignin in the chemical used in the pre-treatment process is influenced by the molecular weight of the lignin, the temperature, the ratio of solid to liquid in the process and material properties of ionic strength [5]. The advantage of chemical pre-treatment is speed, but a disadvantage is that it still requires a pretreatment laundering process for separating the lignin or further processing. This requires high utility and the presence of wastewater containing lignin, thus adding costs for waste treatment [6], [7].

Typically, the chemical process uses acid hydrolysis. Dilute acid hydrolysis is carried out using acids such as H_2SO_4 and HCl at temperatures of between 120-200 ° C. Hydrolysis is the process of breaking the poly-saccharides chain into mono-saccharides using water. Complete hydrolysis of cellulose will produce glucose, while hemicellulose produces several monomer pentose sugars (C5) and hexoses (C6).

Fermentation to convert glucose and xylose into ethanol uses *Saccharomyces cerevisiae*. The optimum concentration of *Saccharomyces cerevisiae* starter is 10% [8]. In this fermentation process factors that affect the bio-ethanol produced are temperature, fermentation time and shaker speed. Optimal fermentation temperature is $38^{\circ}C$ [9] and the best fermentation time is 72 hours for EFB_f [10]. Optimal shaker speed is 150 rpm [11].

C. Conversion of Bio-ethanol to Bio-hydrogen

Bio-ethanol produced from lignocellulosic materials is the raw material for making hydrogen in the future. Advantages of the use of bio-ethanol in the steam reforming process to produce bio-hydrogen are seen during storage and use of fuel cell applications. Bio-ethanol is considered a compound that is less toxic than methanol. Previous research to produce hydrogen from ethanol, done [12], [13] tested the thermodynamic equilibrium in the steam reforming of ethanol to produce hydrogen. Calculations made at pressures of 1-9 atm, temperatures of 400-800 K and water content in ethanol of 0.1 - 10% resulted in optimal conditions for producing hydrogen being at a temperature of 650 K. Vasudeva [14] conducted research on steam reforming of ethanol in equilibrium conditions which resulted in the formation of stable carbon and a very high hydrogen yield of about 5.5. mol per mol of ethanol in the feed. Development research was conducted by [12] on conditions in the formation of stable carbon done by [15]. In this development, re-forming is considered to have the requirements of the increase in response to the reaction at temperatures of 700-800 K and a high ratio of water to ethanol is predominantly expected to speed up the reaction of ethanol into hydrogen. Previous analysis of thermodynamics conducted by [16] provided an analysis of the thermodynamics of hydrogen made from ethanol with the application of solid-polymer fuel cells to be very good. Both the steam reforming and partial oxidation reactor have linkages to water-gas-shift, and Reactor CO-oxidation deemed to affect operating parameters on hydrogen yield. In addition, the study of thermodynamics and numerous catalytic experiments have been conducted by several researchers throughout the world.

D. Mathematical Equation Model and Reaction

The conversion of bio-ethanol from EFB_f into biohydrogen requires that a variety of data and reactions become a reference in the calculation. Some mathematical equations and chemical reactions are used in the simulation of bio-hydrogen production from bio-ethanol, such as the following

1) Ethanol Reforming (ER): Reaction on the ethanol reformer [16] following:

$$C_2H_5OH + H_2O \longrightarrow 2CO + 4H_2$$

Reaction rate following equation:

$$r_{1} = C1 \ k \exp\left(\frac{-Ea}{RT}\right) P_{a}^{1.25} P_{b}^{-0.125}$$
(1)

The equation of Mass Balanced:

$$\frac{dF_{C_2H,OH}}{dz} = -r_1 \tag{2}$$

$$\frac{dF_{H_2O}}{dz} = -r_1 \tag{3}$$

$$\frac{dF_{co}}{dz} = 2r_1 \tag{4}$$

$$\frac{dF_{H_2}}{dz} = 4r_1 \tag{5}$$

2) Water Gas Shift (WGS):

$$O + H_2O \longrightarrow CO_2 + H_2$$

Reaction rate:

$$r_1 = k \frac{P_{CO} P_{H_2O} - K_p^{-1} P_{CO_2} P_{H_2}}{1 + 4.4 P_{H_2O} + 13 P_{CO_3}}$$
(6)

The equation of Mass Balanced:

$$\frac{dF_{co}}{dz} = -r \tag{7}$$

$$\frac{dF_{CO_2}}{dz} = r \tag{8}$$

$$\frac{dF_{H_2}}{dz} = r \tag{9}$$

$$\frac{dF_{H_2O}}{dz} = -r \tag{10}$$

3) Membrane Separation

The equation on the membrane separation unit [17]:

$$J = AP\left(\overline{P}_{H_2O}^n - \overline{p}_{H_2O}^n\right) \tag{11}$$

II. MATERIAL AND METHOD

This research was carried out with a combination of experimental and simulation calculations using a computer program on the process of making bio-ethanol from EFB_f done in laboratory experiments to determine operating conditions for the process of pre-treatment, hydrolysis, and fermentation. In addition, simulation calculations were performed for the production of bio-hydrogen. Simulations were carried out by calculating the reaction in the reactor/reformer and developing programs with a module combination of reformer and separator. The separator used in this study was a gas membrane.

A. Determination of Operating Conditions for Production of Bio-ethanol Converted from EFB_f

Determination of operating conditions in the pre-treatment and hydrolysis process verify the results using fermentation with Saccharomyces cerevisiae.

1) Operating Conditions in Pre-treatment Process

The pre-treatment process in this study used Sodium Hydroxide. The variations in concentrations of sodium hydroxide used were 1-15%. The variations in lengths of time of the pretreatment heating using an autoclave with an operating temperature of about 121° C and a pressure of 1 atmosphere were between 30-150 minutes. Analysis of lignin, cellulose, and hemicellulose of EFB_f after pretreatment process was made using *Chesson Datta Methode*.

2) Operating Conditions in the Hydrolysis Process

The hydrolysis process in this study was carried out using sulfuric acid. Variations of concentrations of sulfuric acid used were 0.5 - 10%. Variations in the lengths of time of hydrolysis by heating using an autoclave with an operating temperature of around 121°C and at 2 atmospheres of pressure were between 30-150 minutes. Analysis of glucose after the hydrolysis process was made using Somogyi-Nelson. After the pre-treatment and hydrolysis, a fermentation process was then performed using Saccharomyces cerevisiae, with a fermentation time of between 1-6 days. Analysis of the bio-ethanol fermentation results was made using Gas Chromatography (GC) Shimadzu type FID Detector.

B. Performance and Optimization

Performance of each scheme is shown by the acquisition of hydrogen gas. Optimisation of operating parameters and design parameters was conducted. Operating parameters were stated by the effect of pressure inside the separation shell membrane while the influence of the design parameters was stated by the length of the reactor. Optimisation measures used are shown in Fig. 1.



Fig. 1 Stage design optimization on bio-ethanol cracking process into bio-hydrogen

For the simulation and optimisation, the values of operating parameters and kinetics used were obtained from the results of research performed by Brian James and Jeff Kalinoski (2008).

C. Optimization and Design Process for Bio-ethanol Converted from EFB_f to become Bio-hydrogen

Optimal conditions for production of bio-ethanol from EFB_f were established in part A; these bio-ethanol characteristics were then used in simulation calculations using computer programs for conversion to bio-hydrogen.

3) Design Simulation

Stages of design in the simulation calculations conversion bio-ethanol into bio-hydrogen can be seen in Fig. 2.



Fig. 2 Design simulation for hydrogen production

4) Equipment

Simulation calculations to produce bio-hydrogen from bioethanol made from raw EFB_f consists of some equipment is a tool with Ethanol Reformer (ER), Water Gas Shift (WGS) and concretes membrane as in Eig. 2







III. RESULTS AND DISCUSSION

Analysis of substances in the EFB_F can be seen in Table 1.

TABLE I
CHEMICAL SUBSTANCES IN EFB_F

No	Zat	Rata-rata (%)
1	Hemicellulose	18.0703
2	cellulose	24.1201
3	lignin	33.5055

Empty fruit bunch fiber (EFB_f) used are as follow :



Fig. 4 Empty Fruit Bunch Fiber (EFB_f)

A. Operating Conditions for Converting Bio-ethanol Produced from EFB_f

1) Pre-treatment Process: At this stage, the lignin is removed from EFB_f using NaOH in various concentrations. The results of this study indicating lignin loss during pre-treatment with NaOH can be seen in Fig. 5.



Fig. 5 Profile of lignin reduction on $\mbox{EFB}_{\rm f}$ during pretreatment using 0.5% of NaOH

Fig. 5 shows that lignin reduction increased by increasing the length of pre-treatment time. In general, the higher the concentration of sodium hydroxide used, the higher the increase in lignin reduction. Pre-treatment times of 15 to 60 minutes showed that lignin degradation by NaOH increases with higher concentrations of sodium hydroxide and longer pre-treatment times. This is in contrast to the 75-90 minutes of pre-treatment time, where it appears that lignin loss does not change with time. In this case, lignin loss remains stable for the pre-treatment time from 75 to 90 minutes. Increasing the concentration of sodium hydroxide from 0.5 N to 1 N also shows no significant effect on the amount of lignin loss. This can be seen from the near equal amounts of lignin missing for both concentrations. Evaluation of process conditions shows a concentration of 0.5 N sodium hydroxide with a pretreatment time of 75 minutes to be the optimum process conditions for lignin removal from lignin TKKS with a maximum loss amount reaching 57.14%.

2) *Hydrolysis Process*: Data from the analysis of sugar in this study can be seen in Table 2.



 TABLE II

 DATA OF YIELD OF GLUCOSE ON HYDROLYSIS WITH SULFURIC ACID

In the table above it can be seen that the highest glucose concentration occurs with a sulphuric acid concentration of 6% and with a hydrolysis time of 60 minutes, which produces a glucose solution of 0.6054 gr/100, or approximately 0.6054%.

The fermentation process is done to achieve optimum fermentation in the production of bio-ethanol from oil palm empty fruit bunch and the optimum amount of acquisition of ethanol resulting from fermentation. Hydrolysis with a 6% concentration of sulphuric acid and a hydrolysis time of 60 minutes is fermented to obtain ethanol. Data obtained during the six days of the fermentation process of ethanol production can be seen in Fig. 6.



Fig. 6 Profile of bio-ethanol production from $\mbox{EFB}_{\rm f}$

Fig. 6 shows that the fermentation time is likely to increase to 4 days of fermentation, but bio-ethanol produced decreases further if the fermentation time is extended. Optimum bio-ethanol production from oil palm empty fruit bunch was three days with an acquisition amount of ethanol of 6.58%. Overall, for production of bio-ethanol from oil palm empty fruit bunch, a pre-treatment process is necessary using 0.5 N NaOH concentration and a pre-treatment time of 75 minutes, while the hydrolysis process used 6% sulfuric acid and a 60 minutes process time. Furthermore, optimal ethanol fermentation results were obtained with a

concentration of 6.58% sulfuric acid, 60 minutes hydrolysis, and fermentation for 3 days.

B. Simulation Results for Conversion of Bio-ethanol into Bio-hydrogen

A combination of equipment consisting of a reformer, WGS and membranes were used. Combining these tools into a single tool that serves as a reactor can simultaneously separate the reaction products. The performance of this tool can be evaluated from the conversion, the hydrogen gas produced. Optimisation of reactor length is a parameter for evaluation of its effect on the production of hydrogen gas. This tool serves as a reactor as well as a separator, so it can also be a Separation Membrane Reactor (RMS). Co-relation of the length of the reactor with conversion can be seen in Fig. 8.







Fig. 9 Profile concentration of component in RMS

The maximum conversion of ethanol in the RMS was 70.2%. The H_2 gas yield obtained was 15.2%. The result of every stage can be seen:

1) Stage 1: The result of mass balance at the operating condition and design process on the stage 1 show in the Tabel 3.

TABLE III THE RESULT OF STAGE 1 CALCULATION

Component	F1	F2	F3
C ₂ H ₅ OH	0.0079	0.002354	-
H ₂ O	0.0237	0.01267	-
СО	-	0.005608	-
CO2	-	0.005484	-
H ₂	-	0.015851	0.011888
Р	-	-	-
Т	-	-	-
Yield H ₂	-	-	-
H ₂ Recovery	-	-	-

2) Stage 2: The result of mass balance at the operating condition and design process on the stage 2 show in the Tabel 4.

TABLE IV THE RESULT OF STAGE 2 CALCULATION

Component	F1	F2	F3
C ₂ H ₅ OH	0.0079	0.002147	-
H ₂ O	0.0237	0.012084	-
СО	-	0.005643	-
CO2	-	0.005864	-
H ₂	-	0.01082	0.018153
Р	-	-	-
Т	-	-	-
Yield H ₂	-	-	_
H ₂ Recovery	-	-	-

3) Stage 3: The result of mass balance at the operating condition and design process on the stage 3 show in the Tabel 5.

THE RESULT OF STAGE 5 CALCULATION			
nent	F1	F2	
ł	0.0079	0.000593	

TABLE V

Component	F1	F2	F3
C ₂ H ₅ OH	0.0079	0.000593	-
H ₂ O	0.0237	0.006754	-
СО	-	0.004974	-
CO2	-	0.009639	-
H ₂	-	0.002802	0.0361
Р	-	-	-
Т	_	_	-
Yield H ₂	-	-	-

H ₂ Recovery	-	-	-
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The results of each step were compared to the optimisation measures to get the operating conditions and parameters of the best design for the reactor and separator in combination. Table 6 shows a comparison of H₂ and H₂ recovery yield for each step of optimisation.

TABLE VI Comparison OF Yield OF H_2 and $H_2 \, Recovery$ for each Optimization STAGE

No	Parameter	Stage 1	Stage 2	Stage 3
1	Yield of H ₂	15.20%	15.90%	21.40%
2	H ₂ Recovery	42.80%	62.6	92.8
3	Flow rate of H ₂	0.011888	0.018153	0.0361

From the table above it can be stated that step 3 is the best option for hydrogen production and membrane reactors in combination. The results of the design for the production of bio-hydrogen gas using a combination of reactor and separator membrane can be seen in Table 7.

TABLE VII THE RESULT OF DESIGN REACTOR MEMBRANE SEPARATOR FOR BIO-HYDROGEN PRODUCTION FROM BIO-ETHANOL

No	Keterangan	Nilai
1	Yield of H ₂	21.40%
2	H ₂ recovery	92.80%
3	Flow rate of H ₂ (mol/s)	0.0361
4	Design Parameter :	
	a. Diameter of Reactor	0.0508 m
	b. Lenght of Reactor	RMS = 10 cm
5	Operating Parameter :	
	a. Pressure	20 bar
	b. Temperature	973 K
	c. Pressure shell in membrane separation	Vakum 2 bar

IV. CONCLUSIONS

The conclusions on the implementation of this research are: (1) To produce bio-ethanol from oil palm empty fruit bunch, optimal conditions for a pre-treatment process occur when using a concentration of 0.5 N NaOH for 75 minutes. The hydrolysis process was carried out using sulphuric acid at a concentration of 6% for 60 minutes. The fermentation process was carried out for 3 days. (2) To produce hydrogen from the conversion of bio-ethanol made from empty oil palm bunch using a combination of reactor and separator membrane in optimal conditions for generating maximum hydrogen yield, the design of the data obtained was as follows: Hydrogen Yield (H2) of 21.4%, H2 Recovery of 92.8%, flow rate of Pure H₂ gas of 0.0362 mol / sec, Diameter reactor of 0.0508 m, Reactor (RMS) length of 10 cm, Operating Pressure of 20 bar, Operating temperature of 973 K, Shell in membrane separation pressure of 2 bar vacuum.

ACKNOWLEDGMENT

We thank the Indonesian Directorate General of Higher Education (DP_2M DIKTI) for financial support of this work through the research Grant for lecturer "National Strategic Research Grant 2009". We are grateful to all participants of the Sultan Ageng Tirtayasa University, Cilegon Banten for all their support of this work.

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