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Effect of Temperature on Gasification of Biomass Using Zeolit

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Abstract. Biomass gasification converts into syngas through the gasification process is promising for renewable energy utilization. Although gasification is a sustainable and environmentally-friendly technology for value-added utilization of biomass, yield percentage is the major problem during biomass gasification. An optimized gasifier and highly active catalyst were proved to be effective for biomass production. Furthermore, the decomposition pathway was also important to advance the gasification reactors and catalyst design. This paper summarized the fundamentals, such as gasifier types, zeolite catalysts, and reaction and deactivation mechanisms. This review also sheds light on other excellent catalysts, effective gasifier, and catalyst reaction mechanisms are also discussed in detail. At last, the paper ends with a conclusion and a prospective discussion of the lab-scale research. At the contact time of 240 minutes, with a column height of 30 cm, at a temperature of 450 oC, the concentration of syngas promise well.

Keywords: D-Fructose, adsorption, fructose concentration, natural zeolite

1. Introduction

Indonesia is an agricultural country with a subtropical climate which is suitable for multicultural plants, especially is sugarcane. Sugarcane is a plant as raw material for producing sugar and is a type of grass. Sugarcane fabrication need around 34.5 million tons sugarcane to produce 2.2 million tons of sugar [1]. The age of the sugarcane plant from planting until it can be harvested reaches approximately 1 year. In Indonesia, sugarcane plantations are spread across ten provinces, namely: North Sumatra, South Sumatra, Lampung, West Java, Central Java, DI Yogyakarta, East Java, West Nusa Tenggara, South Sulawesi and Gorontalo [2]. Nowadays, there are approximately 60 sugar factories operating and most of them are located in Java, especially East Java. All the sugar factories need sugar cane as main material.

Sugarcane waste (biomass) could convert solar energy to chemical energy due to its cellulose structure. Compared to other similar crops, sugarcane yields much higher carbohydrates and dry matter around 30 tonnes of dry matter per hectare [3]. The opportunity for this sugarcane biomass as raw material for the chemical industry is quite large, especially as renewable resource. In addition, the Indonesian government estimates that Indonesia's petroleum reserves will be exhausted of 15 years, natural gas of 60 years, and coal will be exhausted for 150 years [4]. These problems can affect energy security in Indonesia, base on this situation we need alternative energy as a substitute for fossil energy.



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Fig. 1. Image of Sugarcane Biomass (Bagasse)

Modification of the gasification process with biomass raw materials from biomass using zeolite as catalyst, in this study is bagasse, which aims to utilize biomass waste to methane and increase the conversion of biomass waste into alternative energy. Gasification is a process of converting solid fuels into combustible gases (CO, CH₄, and H₂) through a combustion process with a limited air supply (20 -40%) by breaking long hydrocarbon chains into short hydrocarbons [5].

Zeolite is an aluminosilicate material, which has pore structure with two types of pores were micropores (< 2 nm) and mesoporous (2-50 nm) [6]. Zeolites possess unique and outstanding physical and chemical properties. These characteristics make them very useful in a variety of applications including agronomy, ecology, certain manufacturing, industrial processes, medicine, and cosmetics. Recently, the application of a specific natural zeolite material, clinoptilolite, has been documented in veterinary and human medicine as adsorbent. Subsequently, the market of clinoptilolite-based products for use in vivo has been continuously growing. Clinoptilolite shows a relatively high CO₂ adsorption capacity of 4 - 6 mmol/g [7]. The adsorption ability of clinoptilolite is influenced by its high porosity and regular pore structure. However, the surface area of this zeolite type is relatively lower compared to other adsorbents such as carbon, that has a high surface area of 400 - 1000 m²/g. Alabadi *et al.*, (2015) reported that activated carbon has been used to adsorb CO₂, which has the adsorption capacity of 3.3 mmol/g. Activated carbon has also better regenerative ability than zeolites[8]. However, the pore structure of carbon is irregular and the porosity is low. Therefore, the idea emerged to make zeolite as a template on carbon in order to obtain a carbon material with a large surface area, regular pore structure and porosity and also has high adsorption capacity of CO₂. Zeolites have a shorter life-time in water compared to the resins and the stability of zeolites in water may improve significantly[9]. The following presentation deals with adsorption of carbohydrates in the liquid phase, which means that the saccharide molecules are enriched in the pores of the zeolite [10]. High specific adsorption of carbohydrates on hydrophobic Y-zeolites with only negligible fraction of ionic sites have already been reported [11], and also beta zeolites were shown to have similar effects [12] [13].

The overall combustion process can take place continuously, the drying process and pyrolysis/gasification are the initial stages in the solid fuel combustion process. The combustion process in terms of the amount of oxidizer (air/oxygen) can be divided into complete combustion and incomplete combustion. For example, the reaction of burning methane gas with oxygen and burning hydrogen gas with oxygen is as follows



Meanwhile, the incomplete combustion process occurs when the air or oxygen required is not sufficient to completely burn the fuel, to produce carbon dioxide and water. In incomplete combustion,

the carbon in the fuel is converted into carbon monoxide gas, while the nitrogen will react to NO at high temperature.



Boudouard reaction



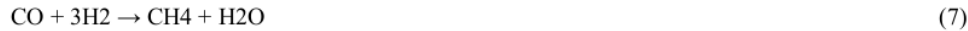
Water gas reaction



Shift conversion



Methanation



This reaction occurs in heated gasifier at certain reaction temperature and then the produced fuel went through the combustion process by reacting oxygen to produce combustible gas and other combustion products. Gasification generally consists of four processes, namely drying, pyrolysis, reduction and oxidation with a temperature range for each process, namely: Drying: $T < 150\text{ }^\circ\text{C}$; Gasification: $150\text{ }^\circ\text{C} < T < 700\text{ }^\circ\text{C}$; Reduction: $800\text{ }^\circ\text{C} < T < 1000\text{ }^\circ\text{C}$; Oxidation: $700\text{ }^\circ\text{C} < T < 1500\text{ }^\circ\text{C}$. The gasification has several factors that can affect the process and the syngas composition. The parameters of gasification are biomass characteristics, gasifier design, flow direction, type of gasification agent and air-fuel ratio (AFR) [7]. Based on the flow direction, the gasification process is divided into 4 types of gasification, namely the updraft, downdraft, fluidized bed and entrained bed as shown in Figure 2.



Fig. 2. Updraft type of Gasifier

Figure 2 showed the used reactors in gasification namely fixed bed and fluidized bed reactors. The effect of operation and design parameters in gasification process has been studied by Jean and Pierre (2009) [14]. Fluidized bed reactor is a technology commonly used to convert fuel into energy. In the last decade, internal circulating fluidized bed (ICFB) or so-called dual bed technology was successfully developed for biomass gasification along with CO₂ adsorption. The advantage of ICFB is the gas production, which has high net heating value (NHV), rich in flammable components and minimizes dilution by N₂. Several researchers have used air, oxygen and steam as gasification agent [15] [16]. In this research, zeolite was used as adsorbent in varied ratio of air and water to produce

syngas. The effect of air, water and contact time on the gasification process to produce combustible syngas (Methane, CO and H₂).were analysed.

2. Experiments

2.1. Sugarcane Biomass (Bagasse) Preparation

Firstly, baggase was dried at temperature 40°C by oven for 5 hours in order to reduce its water content, then chopped it of 7-10 cm, and put into the reactor. The gasification is processed to produce the desired gas product, namely methane, carbon monoxide and carbon dioxide.

2.2. Sugarcane biomass gasification

Dried baggase of 1.7 kg, which was ready to gasification process was entered into updraft type of gasifier with following steps as shown in Figure 3 below.

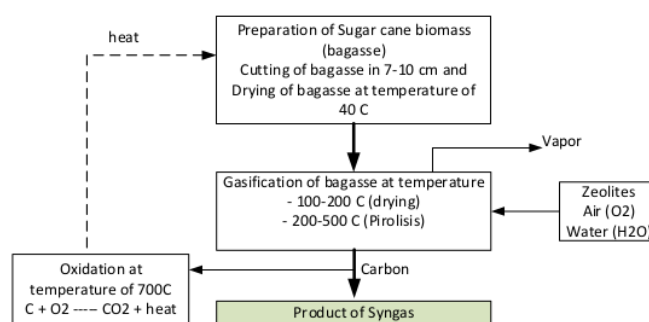


Fig. 3. Flowchart of Bagasse Gasification

The updraft type gasifier (fig.2) was consisted of a thermocouple, thermometer, data logger, condenser, venojet tube, and gas chromatography. The operating conditions of this gasifier were atmospheric pressure, temperature of 300-500°C and air flow rate of 10 L/minute and 20 L/minute using a zeolite. The sugar cane biomass (bagasse) put into gasifier. The drying process of bagasse was started at temperature of 100-200 °C for 25 minutes and the process continued with increasing the temperature from 200-500°C.

The addition of air to the gasifier is carried out at temperature of 300°C with an increase temperature for each range is 50°C until temperature of 500°C. The added air is then replaced by water in the same way. Furthermore, in Figure 4, we can see the distribution of reaction zones in gasification.

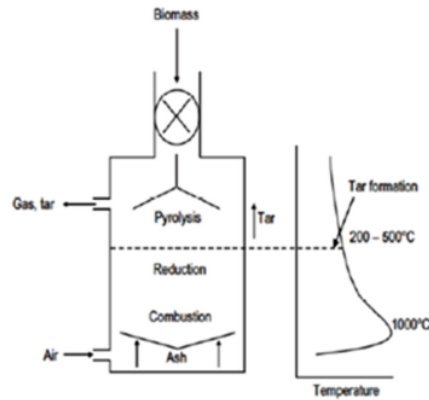


Fig. 4. Gasification Reaction Zone

The temperature distribution in each zone occurs vertically, where the highest temperature gasification zone occurs due to the reaction time. In the combustion zone, the temperature of 1000°C will be reached and above the combustion zone, the reduction process occurs as tar has formed. On the top of zone is pyrolysis, which the gasification produces syngas. The gasification process is complete after the sample is obtained at each predetermined condition at temperature of 300°C, 350°C, 400°C, 450°C, 500°C.

2.3. Bagasse Characterization

The morphology of the surface of the bagasse were examined using a *Field emission scanning electron microscope (FESEM, ZEISS SUPRA 35 VP)*. All specimens were freeze-dried and coated with an ultra thin layer of gold before conducting an analysis. In order to obtain a perfect structures, all sample fractured by immersing in liquid nitrogen. Fourier Transformation Infrared (FTIR) spectroscopy is frequently used to investigate the structure of constituents and the chemical changes in the bagasse biomass during pretreatment.

2.4. Proximate Analysis

The analysis data obtained from Gasification is divided into proximate analysis and ultimate analysis. Proximate analysis is carried out by measuring the caloric value, moisture content, ash content, levels of flying substances. The caloric value of bagasse was measured with a caloric meter bomb (ASTM D5865-2013). Inherent moisture content (ASTM D 3173-17) was measured by weighing bagasse and put into an oven at temperature of 110°C for 1 hour and cooled for 15 minutes then weighed. The moisture content (IM) was calculated by following equation,

$$\text{Moisture Content, \%} = ((a-c)/(a - b)) \times 100\% \tag{10}$$

where a is weight of cup and sample bagasse (gr); b is weight of empty cup (gr); c is weight of plate and sample bagasse after heating (gr). Ash content (ASTM D 3174-12) was measured by weighing bagasse and heating in an AAF furnace at temperature of 750°C for 2 hours and cooled in an excavator for 10 minutes then weighed. Water content can be calculated by following equation

$$\text{Ash content\%} = ((a-c)/(a - b)) \times 100\% \tag{11}$$

where a is weight of cup and sample bagasse (gr); b is weight of empty cup (gr); c is weight of plate and sample bagasse after heating (gr)

Volatile matter (ASTM D 3165-17) was measured by weighing bagasse and heating without oxidation at temperature of 900°C for 7 minutes, then corrected for moisture content. Volatile matter content (VM) could be calculated by following equation,

$$\text{Volatile matter, \%} = ((a-c)/(a-b)) \times 100\% \tag{12}$$

where a is weight of cup and sample bagasse (gr); b is weight of empty cup (gr); c is weight of plate and sample bagasse after heating (gr).

The fixed carbon content is determined by the following formula:

$$\text{Fixed Carbon (\%)} = 100 - (\text{IM} + \text{Ash} + \text{VM}) \tag{13}$$

where IM is moisture content, Ash is ash content and VM is Level of volatile substances.

2.5. Ultimate Analysis

The ultimate analysis was carried out to measure the value of carbon, hydrogen and nitrogen using gas chromatography.

3. Results and Discussion

3.1. Morfology and structural of bagasse

a) Field emission scanning electron microscope (FESEM) analysis

FESEM analysis was conducted to determine the effect of temperature on the material structure. The FESEM image presented the surface of the bagasse in the best condition. This image clearly shows that the material of bagasse smooth and continuous surface. Figure 5 shows basic and compact of fiber surface structure. By drying for 120 minutes at temperature 100-200°C, the gradual breakdown of fiber cell wall and the reduction of dimensions were apparent on the surface of solid product. The accessibility of cellulose fibrils to separate was enhanced by preheated of biomass (bagasse). The result indicated that heat induces morphological changes in the plant cell wall studies.

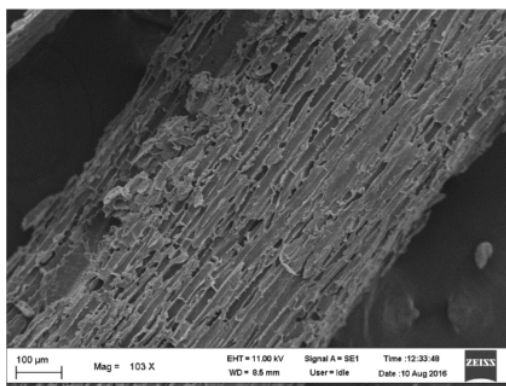


Fig. 5. SEM image of Bagasse

b) FT-IR analysis of functional groups of bagasse

FT-IR spectroscopy is frequently used to investigate the structure of constituents and the chemical changes in the bagasse during treatment. The representative chemical changes related to the water released and feed material was dehydrating during heating process. According to [17], the peak approximately recorded at 3300 cm⁻¹ was attributed to -OH groups that indicated hydroxyl group. The characteristic bond of C=C stretching aromatic skeletal mode of lignin, observed in raw bagasse around 1900 cm⁻¹. The band at 1740 cm⁻¹ was related to the C=O stretching of carbonyl and acetyl

groups attributed mainly to presence of hemicellulose. The absorption band of cellulose at 879 cm^{-1} showed the composition of cellulose in the raw material (bagasse).

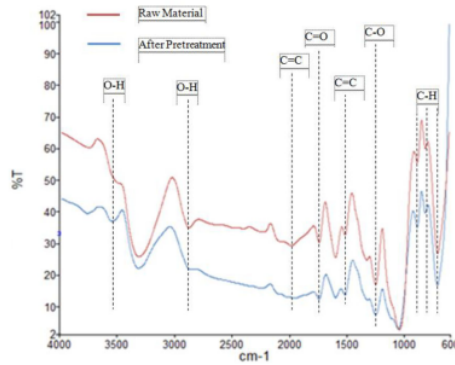


Fig. 6. FTIR-spectra of bagasse

3.2. Proximate and Ultimate Analysis of Bagasse

Based on the data in table 1, it can be observed the physically and chemically percentage of present elements of biomass. the caloric value of bagasse was obtained from 263 gr of dry weight of bagasse was 1830 kcal/kg. The results of the proximate and ultimate analysis of bagasse can be seen in Table 1 below. The content of C and H were high enough to produce the gas. The moisture content of bagasse is relatively low and does not require too much energy to remove it. The water content contained will be removed from the biomass by heating. If the moisture content is too high, a high drying activation energy is also required [11]

Table 1. Proximate and Ultimate Analysis of Bagasse

| Component | Amount, unit | Standard |
|--------------------------|--------------|------------------|
| Caloric Value | 1830 ccal/kg | ASTM D 5865-2013 |
| Proximate analysis, % | | |
| -Total moisture | 15.32 | ASTM D 3302M-17 |
| -Ash content | 4.19 | ASTM D 3173-12 |
| -Volatile matter | 43.68 | ASTM D 3175-17 |
| -Fixed carbon | 35.81 | ASTM D 3172-13 |
| -Sulphide | 0.04 | ASTM D 4239-2016 |
| Ultimate analysis, % | | |
| Carbon (C) | 24.12 | ASTM D 5373 |
| Hydrogen (H) | 3.12 | ASTM D 5373 |
| Nitrogen (N) | 0.12 | ASTM D 5373 |
| Oxygen (O ₂) | 53.09 | ASTM D 5373 |

3.3. Syngas Composition Analysis

The result of syngas composition analysis shows the ratio value of combustible gas (CG) and non-combustible gas (NCG) as shown in Table 2 below.

Table 2. Composition of syngas and ratio of combustible gas and noncombustible gas

| Temperature, °C | CH ₄ | H ₂ | O ₂ | N ₂ | CO | CO ₂ |
|-----------------|-----------------|----------------|----------------|----------------|-------|-----------------|
| 300 | 1.03 | 3.28 | 58.52 | 18.84 | 16.59 | 0.28 |
| 350 | 0.89 | 3.35 | 57.79 | 19.78 | 16.27 | 0.29 |
| 400 | 0.54 | 3.46 | 56.00 | 21.24 | 16.44 | 0.32 |
| 450 | 1.84 | 3.24 | 49.19 | 18.84 | 16.59 | 0.35 |
| 500 | 2.01 | 3.11 | 48.32 | 18.62 | 16.64 | 0.36 |

3.4. Effect of temperature on gasification of bagasse

The long contact time, the adsorbed syngas tends to increase. It is proven that contact time affects the adsorption process.

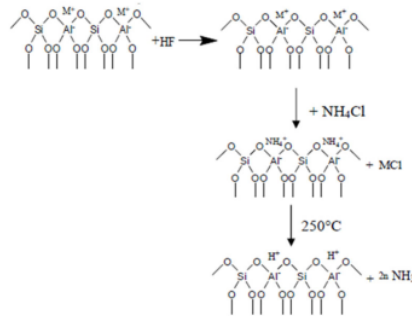


Fig. 7. Reaction mechanism of zeolite

The zeolite is a subject to proceed an ion-to-ion exchange ammonium. Ion exchange between cations in zeolite and NH₄⁺ aim to replace all impurities in the form of cations in the zeolite because it is possible inside the zeolite there are still alkaline or alkaline earth cations such as K⁺, Na⁺, Ca²⁺, Mg²⁺ that play a role as balancer of zeolite that could be exchanged with other cation to produce CH₄. The H-zeolite- is formed thermally, after the ion exchange process heated at a temperature of 250°C. The NH₃ could release after heating for 2 hours.

Based on Figure 3, the gasification process was produces syngas at temperature of 300°C, which was produced fluctuated syngas concentration (CO, H₂ and CH₄) and with increasing the temperature. This condition is influenced by the temperature and the distribution of feed combustion. At 300°C the gasification process, there is still a lot of fuel (feed) that can be converted into syngas, so that a relatively high concentration of syngas (CO, H₂ and CH₄) is obtained. In Table 2, it was showed that the syngas concentration in the temperature of 300° C was produced the CO of 18.84 mol%, H₂ of 1.03 mol % and CH₄ of 1.74 mol % , respectively. Table 2 was also showed the increase of temperature from 350°C to 450°C tend to increase of the produced syngas. As shown, the produced CO increase from 19.78 mol % to 21.24 mol %. However, at temperature of 450° C, the syngas concentration specifically for CO concentration decreased. This phenomenon was due to the decreasing of the feed with increasing temperature used during the gasification process. Reduced feed meant less feed being converted into CO syngas so that the resulting concentration will be lower [18]

3.5. Effect of zeolite on gasification of bagasse

The analysis of syngas resulted syngas composition and the ratio of combustible gas (CG) and noncombustible gas (NCG) with addition of zeolite of 200 gr and water flow rate of 20, 30 and 40 mL/minute at temperature of 500°C as shown in Figure 6.

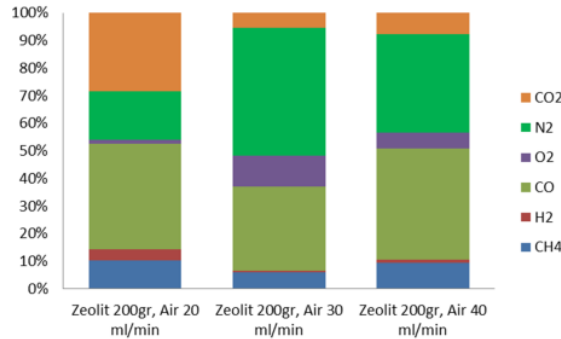


Fig. 8. Produced syngas composition using zeolit

Clinoptinolite embedded carbon is a potential material for adsorption of CO₂ gas. Based on the discussion above, the factors that determine the height CO₂ adsorption capacity is the surface area and microporosity.

Based on Figure 6, the gasification produced syngas concentration at temperature of 500°C fluctuates. This condition was influenced by adding of zeolite and the amount of water in the gasification and also the distribution of feed combustion. By adding of water of 20 mL/minute in the gasification, there was a big amount of bagasse that can be converted into syngas, so that a relatively high concentration of syngas (CO, H₂, and CH₄) is obtained. In Figure 6, it is found that the concentration of syngas in the addition of water 20 mL/minute produced CO of 38.25% mol, H₂ of 4.01% mol and CH₄ of 10.17 mol%, respectively. Meanwhile, the water addition of 30 mL/minute, concentration syngas of CO of 30.78 mol%, H₂ of 0.53 mol% and CH₄ of 5.85% mole. Moreover, by water adding of 40 mL/minute, the produced syngas contained CO of 40.14 mol%, H₂ of 1.31 mol% and CH₄ of 9.26 mol%, respectively. This condition was because of the addition of water. Water addition was needed to produce H₂ gas and in the reduction process to produce H₂ and CO (water gas reaction). The added water must be in accordance with the requirements of the gasification process that is taking place. The addition of water aims to produce a gas product that has a better heating value [19]. The gasification process with the addition of water and zeolite produces a combustible gas ratio that is not that large, based on the data obtained that the use of zeolite was more effective in increasing the combustible gas ratio because zeolites have large pores with a wide surface and an intra-crystalline cavity. These structure could be able to adsorb a large number of substances other than water and are able to separate the molecules of substances based on their molecular size and polarity [20].

4. Conclusion

Based on the results of the research that has been carried out, it can be summarized with the conclusion that the more air is added, the more reactants react with the carbon in the bagasse, thereby increasing the Syngas product but the gas content has high nitrogen (N₂) so that it can interfere with the oxidation reaction to produce methane. Furthermore, the higher the gasification temperature tends to increase gas. Production of combustible gas (CG) as a result of gasification using a 200 gram zeolite catalyst and the water addition of 20 mL/min at a temperature of 500°C produced maximum combustible and noncombustible gas ratio of 1.10. This concluded that zeolite affected the gasification to produced syng gas. The ratio of combustibel and non combustibel directly equivalent to produce of a higher amount of gas.

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