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Sustainable bio energy: The potential of Ni-Fe/NZA as a catalyst for pyrolysis of sugarcane bagasse waste (*Saccharum officinarum L.*) for bio-oil production

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ABSTRACT

The depletion of fossil fuels and the increase in greenhouse gas emissions have sparked public concern. Along with the problems occurred, the need for energy continues to increase, while energy source reserves are decreasing. This research was conducted with an aim to determine the effect of the addition of Ni-Fe/NZA catalyst on bio-oil yield from the pyrolysis process of sugarcane bagasse waste. Chemical analysis was carried out using GC-MS instrument and physical analysis was carried out using a pycnometer, viscometer, pH meter, and bomb calorimeter. The results showed that the bio-oil produced had better quality as evidenced by a series of chemical and physical analysis, so bio-oil can be used as a renewable alternative fuel with dominant compounds in the form of furfural and acetic acid. The highest bio-oil yield was obtained at 1% catalyst weight percentage and at the temperature of 400°C of 48.1%. The density of bio-oil had a range of 0.93-1.01 gr/mL, bio-oil viscosity ranged from 25.75-30.01 cP and Bio-oil pH ranged from 2.62-3.27. The fastest first drop of bio-oil was obtained at 105°C. The calorific value had a range of 20.26-27.05 MJ/kg. It can then be concluded that Ni-Fe/NZA has great potential as a catalyst in the pyrolysis process and improves the quality of bio-oil produced in the development of sustainable technology.

1. Introduction

The depletion of fossil fuels and the increase in greenhouse gas emissions have sparked public concern. Along with the problems occurred, the need for energy continues to increase, while energy source reserves are decreasing. More than 80% of energy supply comes from fossil fuels such as coal, petroleum, and natural gas [1]. One of the promising biomass energy sources in the world and in Indonesia is sugar cane (*Saccharum officinarum L.*). In Indonesia the production of sugar cane was in 9th position 9th at 28.9 million tons [2].

Sugarcane processing produces solid waste in the form of bagasse, which has a fibre content of 35-40% of the sugarcane weight. Bagasse is waste from the sugar making process that still contains lignocellulose consisting of 40-50% cellulose, 20-30% hemicellulose, 20-25% lignin, and 1.5-3% ash [3]. The high cellulose and fibre content indicate that bagasse is quite potential as a biorefinery application and can be converted into high-value products such as bio-oil, which is an emulsion with a smoke-like odour produced through the condensation process of steam from the pyrolysis of biomass containing lignocellulose [4]. From its nature, bio-oil is more environmentally friendly and clean, and produces fewer SO_2, NO_x, and soot emissions compared to fossil fuels. Bio-oil is mostly used as boiler fuel and furnaces, or processed to produce fuel oil and chemical products [5].

Bio-oil can be produced using the pyrolysis process, which is the thermochemical decomposition of organic matter at high temperature without using oxygen [6]. The advantage of pyrolysis process is that it can be a promising alternative for biomass processing for having a high conversion ratio, high energy content in its products, able to be upgraded to raw materials for other purposes, and having an easier process control when compared to other biomass combustion processes [7]. Commonly, pyrolysis process uses NZA catalysts or commonly called as zeolites – the minerals consisting of hydrated aluminosilicate minerals containing alkali or alkaline earth cations in a three-dimensional framework. Zeolites have the molecular formula of M2nO.Al2O3.xSiO2.yH2O where x refers to the amount of silica oxide and y refers to the amount of water contained in the zeolite [8]. Zeolites have the advantage of having a large surface area and acidity, enabling them to be easily modified with other minerals in order to obtain maximum results in their utilization process [9].

Iron-nickel catalyst is one of the catalysts that can be used to accelerate the pyrolysis reaction rate. The addition of nickel (Ni) catalyst in the pyrolysis process can affect the electrical conductivity value in which this value is higher than the electrical conductivity at C and lower than Ni-HCl. Also, the addition of Ni catalyst can enable the pyrolysis process to be carried out at a fairly low temperature [10]. The addition of Fe catalyst can expand or increase the active surface [11]. The NiFe/NZA catalyst will be applied to the pyrolysis process that

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then produces the main product of bio-oil as a potential alternative fuel.

This research was conducted to observe the effects of the combination of NiFe and NZA catalysts into NiFe/NZA on the bio-oil production, which is more environmentally friendly and potential as sustainable energy with sugarcane bagasse waste biomass.

2. Materials and Method

2.1. Materials

Sugarcane bagasse waste, natural zeolite, hydrochloric acid (HCl), ammonium chloride (NH4Cl), silver (I) nitrate (AgNO3), nickel (II) nitrate hexahydrate (Ni(NO3)2.6H2O), and nonahydrate ferric nitrate (Fe(NO3)3.9H2O), distilled water.

2.2. Tools

Pyrolysis reactor, furnace, mortar and pestle, sieve, 2-neck flat-bottomed boiling flask, Liebig condenser, magnetic stirrer, thermometer, desiccator, evaporator cup, blender, measuring cylinder, measuring flask, spatula, Erlenmeyer flask, pycnometer, Ostwald viscometer, dropper pipette, beaker, glass funnel, filter paper, pH meter, Gas Chromathography-Mass Spectrometer (GC-MS), and bomb calorimeter.

2.2. Sugarcane bagasse pre-treatment

Preparing the raw materials was done by chopping sugarcane bagasse waste into small pieces. It was continued by drying the sugarcane bagasse waste under the hot sun until being dry. Sugarcane bagasse waste was ground using a blender and sieved using a 25-mesh sieve.

2.3. Sintesis Ni-Fe/NZA

The synthesis of Ni-Fe/NZA catalyst referred to Mutaqqi et al. [12] by combining Ni(NO3)2.6H2O and NZA compounds with an impregnation technique at the temperature of 120ºC for 12 hours. Subsequently, at a similar temperature and duration, Fe(NO3)3.9H2O was combined with Ni/NZA using a metal impregnation technique. The Ni-Fe/NZA catalyst resulted could be activated through calcination at a temperature of 550°C for 3 hours.

2.4. Pyrolysis of sugarcane bagasse waste

The treated sugarcane bagasse waste biomass was pyrolyzed using Ni-Fe/NZA catalyst with varied temperatures (350°C and 400°C) and the variation of catalyst weight percentage (1%; 1.5%, and 2%) for 30 minutes. The experiment was repeated on samples without using a catalyst at a temperature of 400°C.

2.5. Chemical analysis of bio-oil

The analysis of bio-oil composition was carried out using a combination of GC-MS methods. The compound components of pyrolysis bio-oil were separated using GC (Gas Chromatography), which was then analyzed for the types of components contained using MS (Mass Spectrometer) through its mass spectrum with an aim to identify the types of compounds.

2.6. Physical analysis of bio-oil

The test of the physical characteristics of bio-oil was carried out by determining its density, viscosity, acidity level, and calorific value. It was carried out using a pycnometer, Ostwald viscometer, universal pH paper, and bomb calorimeter.

3. Results and Discussion

3.1. Yield bio-oil

The pyrolysis process of sugarcane bagasse waste was performed with varied temperatures (350°C and 400°C); while the percentage of catalyst weight was varied by 0%, 1%, 1.5%, and 2%. Pyrolysis was carried out for 30 minutes by means of a pyrolysis reactor in connection to a cooling condenser to change the product gas phase into a liquid one.

Table 1. The effects of temperature and percentage of catalyst on *yield bio-oil*

Temperature (°C)	Catalyst Percentage (%)	Yield (%)
350	1	43.89
	1.5	46.03
	2	41.12
400	0	45.76
	1	48.1
	1.5	46.5
	2	46.44

At the temperature of 350°C, the yield bio-oil from pyrolysis increased along with the increasing percentage of catalyst weight to biomass. At a catalyst weight of 1%, the bio-oil yield was 43.89% and increased at a catalyst weight of 1.5% by 46.03%. This increase occurred since the catalyst determined the pyrolysis process. The higher the percentage of catalyst weight, the greater the yield that will be produced. However, at a catalyst weight of 2%, the yield of bio-oil produced decreased due to the presence of biogas products or short-chain hydrocarbons that could not be condensed during the pyrolysis process. [13].

Similarly, at the temperature of 400°C, from a catalyst weight of 0% to a catalyst weight of 1%, the yield of bio-oil produced increased, but the yield decreased at a catalyst weight of 1.5% and again increased at a catalyst weight of 2%. This decrease in yield occurred in view of the presence of biogas products or short-chain hydrocarbons that were not condensed during the pyrolysis process.

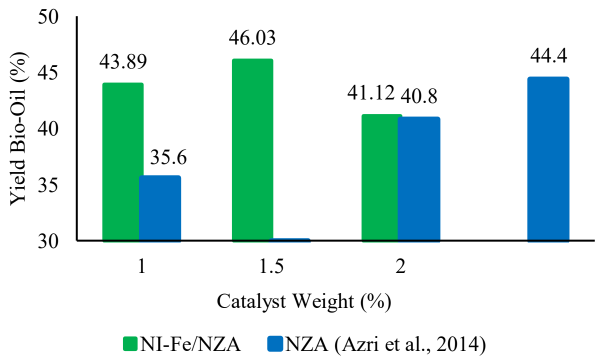

Fig. 1. The effects of temperature and weight of catalyst on *yield bio-oil*

Compared to research conducted by Azri et al. [14] that used the NZA catalyst, the comparison of the bio-oil yield of the three catalysts showed that the Ni-Fe/NZA catalyst produced a high bio-oil yield exceeding the zeolite and NZA catalysts. This was because the presence of Ni metal enabling the pyrolysis process of sugarcane bagasse waste to be carried out at a fairly low temperature and produced a large yield [10]. The presence of Fe metal also extended the active surface of the catalyst in which the pyrolysis process could take place rapidly and produced a large yield [11]. Thus, it can be concluded that Ni-Fe/NZA also has great potential as a catalyst for the pyrolysis process of biomass, particularly sugarcane bagasse waste.

3.2. The effects of Ni-Fe/NZA on the Biomass Decomposition

The Ni-Fe/NZA catalyst also has great potential as a pyrolysis catalyst as seen in the first drop of bio-oil that falls from the condenser.

Table 2. The effects of percentage of catalyst weight on the first drop of *bio-oil*

Temperature (°C)	Percentage of Catalyst Weight (%)	Temperature of First Drop (°C)
350	1	120
	1.5	116
	2	105
400	0	141
	1	116
	1.5	114
	2	113

As observed at the temperature of 350°C, the temperature of the first drop at each catalyst weight decreased. At a catalyst weight of 1%, the first drop occurred at the temperature of 120°C and at a catalyst weight of 2%, the first drop occurred at the temperature of 105°C. A decrease in temperature also occurred at a pyrolysis temperature of 400°C. In the variation without a catalyst, the first drop occurred at the temperature of 141°C and at a catalyst weight of 2% the first drop occurred at the temperature of 113°C. This proved that the addition of Ni-Fe/NZA catalyst had a significant effect on the rate of the pyrolysis process of sugarcane bagasse waste. The greater the percentage of catalyst weight added, the faster the reaction in pyrolysis to form the main product of bio-oil [15].

3.3. The effect of Ni-Fe/NZA on the bio-oil density

As seen in Figure 2, the bio-oil density has been measured using pycnometer.

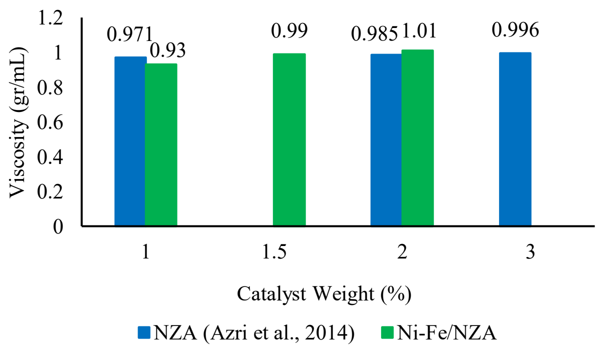

Fig. 2. The effects of catalyst weight percentage on the *bio-oil* density

The results of the research showed that the *bio-oil* density was in the value range of 0,93 gram/mL to 1,015 gram/mL, indicating that the bio-oil from the results of the research has fulfilled the standard of the bio-oil density in the range of 0,92-1,2 gr/mL [16]. The values of bio-oil density using the Ni-Fe/NZA catalyst, if compared to the values of density using NZA catalyst, was found not much different respectively with the range of 0,93-1,01 gr/mL, and 0,971-0,996 gr/mL. However, compared to the density of bio-oil using NZA catalyst, it was found much different with the range of 1,02-1,017. This difference was caused by the high water content in the resulted bio-oil in which the value of bio-oil density using the Ni-Fe/NZA catalyst became lower compared to the one using zeolite catalyst [17].

3.4. The effects of Ni-Fe/NZA on the bio-oil viscosity

The viscosity of bio-oil has been measured using Ostwald viscometer at each temperature variation and the percentage of catalyst weight used in the pyrolysis process, and the effect of using Ni-Fe/NZA on the quality of the bio-oil produced could be identified as seen in Figure 4.

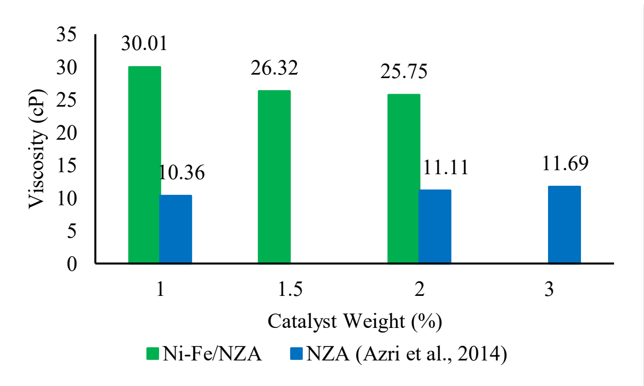

Fig. 3. The comparison in the effects of percentage of catalyst weight on the viscosity of bio-oil

The viscosity value of bio-oil using Ni-Fe/NZA catalyst when measured at room temperature decreased along with the increasing percentage of catalyst weight. The decrease in viscosity value was related to the presence of a catalyst accelerating the decomposition process of heavy molecules into light ones; as a result, the Ni-Fe/NZA catalyst could provide a good effect on the pyrolysis process and produce qualified bio-oil [18]. The decreasing viscosity value also showed the potential of bio-oil as an alternative fuel for being easier to flow [19].

Compared to the viscosity of bio-oil using NZA catalyst, the viscosity of bio-oil with Ni-Fe/NZA catalyst was found still higher as the pyrolysis process was conducted for 30 minutes making the process of cracking heavy molecules into light one not maximum. It was in contrast to bio-oil using NZA catalyst for 120 minutes that provided maximum time to convert the heavy molecules in bio-oil into the light ones.

3.5. The effect of Ni-Fe/NZA on bio-oil pH

Since the pH of *bio-oil* has been measured using pH meter in each variation of temperature and the percentage of catalyst weight used in the pyrolysis process, the effects of Ni-Fe/NZA use on the quality of resulted bio-oil could be identified as seen in Table 3.

Table 3. The effects of percentage of catalyst weight on *bio-oil* pH

Temperature (°C)	Percentage of Catalyst Weight (%)	pH
350	1	3.13
	1.5	3.25
	2	3.27
400	0	3.11
	1	2.62
	1.5	3.3
	2	3.02

At a temperature of 350°C, the pH range could be seen at 3.13-3.50 and at a temperature of 400°C it was at the pH range of 2.62-3.30. This pH range was suitable with the standard pH of bio-oil which is in the range of 2.3-3.3 [20]. The pH value also indicates the level of the process when pyrolysis takes place to decompose biomass into acidic compounds in bio-oil.

The acidity level also has a good effect on bio-oil as it enables the shelf life of bio-oil to last longer [11]. The compounds with an acidic effect on bio-oil are acetic acid and phenol compounds which are in accordance with the results of the GC-MS analysis proving that acetic acid and phenol are the dominant compounds in each bio-oil. Therefore, the Ni-Fe/NZA catalyst also has an effect on the quality of bio-oil.

3.5. Characterization with gas chromatography-mass spectrometry (GC-MS)

In the analysis of the components contained in bio-oil, the GC-MS instrument was used on the bio-oil product with the highest yield at the catalyst weight percentage of 1% with a pyrolysis temperature of 400°C. Figure 5 illustrates the chromatogram results.

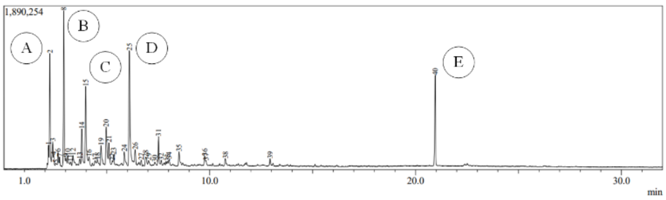

Fig. 4. Chromatogram of gas chromatography of bio-oil with the percentage of catalyst weight 1% at pyrolysis temperature of 400°C

Notes:
A : *Acetic Acid* C : *Phenol* E : *Dioctyl phthalate*
B : *Fufural* D : *4-ethyl-Phenol*

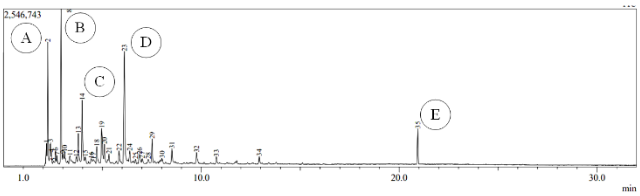

Fig. 5. Chromatogram of gas chromatography of bio-oil with the percentage of catalyst weight 2% at pyrolysis temperature of 400°C

Notes:

A : *Acetic Acid* C : *Phenol* E : *Phtalique Acid*
B : *Furfural* D : *4-ethyl-Phenol*

It can be seen that the use of 1% Ni-Fe/NZA catalyst at the temperature of 400°C could break down lignocellulose contained in sugarcane bagasse waste into 40 compounds with dominant compounds as shown at the highest peak including acetic acid of 12.09%, furfural of 17.47%, phenol of 8.95%, 4-ethyl-phenol of 13.53%, and dioctyl phthalate of 7.85%. In bio-oil products with variation of temperatures and weight percentage of other catalysts also had dominant main compounds of furfural, acetic acid, and phenol.

The simialr dominant compounds were also contained in bio-oil with the use of 2% catalyst at the temperature of 400°C with a percentage of acetic acid 9.72%, furfural 14.72%, phenol 8.06%, 4-ethyl-phenol 16.35%, and phtalique acid 3.63%. Looking at the use of 1% and 2% catalysts, there was a difference in the number of total compounds in which in the 1% catalyst there were 40 compounds and in the 2% catalyst the there were 35 compounds. The decrease in the total number of compounds was in accordance with the results of the calorific value obtained at a temperature variation of 400°C and the weight percentage of 1% and 2% catalysts, which also experienced an increase.

3.6. The effects of Ni-Fe/NZA on calorific value

The calorific test was conducted by means of bomb calorimeter on each bio-oil at each variation of temperatures and the percentage of catalyst weight as presented in Table 4.

Table 4. The effect of percentage of catalyst weight on calorific value

Temperature (°C)	Percentage of Catalyst Weight (%)	Calorific Value (MJ/kg)
350	1	20.79
	1.5	22.02
	2	22.23
400	0	20.26
	1	22.30
	1.5	25.06
	2	27.05

It can be found out that the calorific value of bio-oil products at a temperature of 350°C was in the range of 20.79-22.23 MJ/kg, and at a temperature of 400°C it was in the range of 20.26-27.05 MJ/kg. The calorific value obtained was in accordance with the ASTM D 7544-09 standard with the minimum of 15 MJ/kg [21]. At each temperature and increase in the percentage weight of the catalyst, the calorific value of the bio-oil increased, proving that the quality of bio-oil was good and potential to be used as a renewable alternative fuel.

The addition of Ni-Fe/NZA as a catalyst had a positive effect on the quality of bio-oil. Ni-Fe/NZA as a catalyst could remove any oxygen compounds, reduce viscosity, and break down heavy hydrocarbons into short-chain hydrocarbons [11]. The pyrolysis temperature also affected the calorific value contained in bio-oil. The higher the temperature, the higher the calorific value of bio-oil [22].

4. Conclusion

From this research, it can be concluded that the variation of temperatures and percentages of Ni-Fe/NZA catalyst could bring an effect on the bio-oil resulted from the pyrolysis process of sugarcane baggase waste. Physical tests of density, viscosity, and pH of the bio-oil produced showed that bio-oil had good quality and had great potential as an alternative renewable energy source. While, bio-oil products that did not use catalysts showed worse quality compared to those using Ni-Fe/NZA catalysts. The dominant compounds in each bio-oil include furfural, acetic acid, and phenol. The highest bio-oil yield was obtained at a catalyst weight percentage of 1% and at the temperature of 400°C of 48.1%. Density was in the range of 0.93-1.01 gr/mL, viscosity was in the range of 25.75-30.01 cP, and bio-oil pH was in the range of 2.62-3.27. The fastest first drop of bio-oil was obtained at a temperature of 105°C. Therefore, it can be concluded that the use of Ni-Fe/NZA catalyst for bio-oil production can provide benefits for producing bio-oil in greater quantities and better quality as an application of sustainable bioenergy in the future.

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