Performance and Characteristics of Bio-Oil from Pyrolysis Process of Rice Husk

by Sri Aulia Novita

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Abstract. This study aims to modify the pyrolysis device that produces bio-oil with methyl esters, determine the content of methyl esters with GC/MS analysis, and test the performance of biodiesel using a diesel engine. This research modified the pyrolysis tube wall by changing the thickness of the stainless-steel material to 1.5 mm to facilitate the combustion process and heat transfer and reduce the equipment weight. Meanwhile, the tube base still uses 3 mm stainless steel to prevent leakage during the process because of high temperature (300 – 400°C). Using wood and coconut shell could accelerate the incomplete combustion process and produce higher methyl ester than using a gas stove. The process using wood and coconut shell could produce 35.88% of bio-oil produced while the process using a gas stove only produces 30%. The GC/ MS analysis has discovered that the content of methyl ester and ethanol was 60.12% and 1.13%, respectively. The obtained methyl ester was separated from the tar using a rotary evaporator based on the boiling point difference. Methyl ester from this husk can turn on the diesel engine with B20-B60.

Keywords: Rice husk, pyrolysis, methyl ester, Bio-oil

1. Introduction

Indonesia's need for diesel fuel increases by 6% every year. Petroleum productivity does not suffice because more than 50% of the total domestic need is imported. Reliance on imported diesel fuel can be minimized in several ways; one of which is producing an alternative oil fuel comprising of biodiesel (methyl ester) and biomass from agricultural waste, such as corncob and rice husk. Biomass is one of the renewable energy sources, and it can be used as a source of heat energy, biofuel, chemicals, biomaterial, and transportation [3, 4]. Cellulose, hemicellulose, and organic compounds in biomass are determined as the elements of carbon, hydrogen, and oxygen and possess high energy content [1, 2]. Fuels, including biodiesel has physical and chemical characteristics akin to diesel fuel.

Novita, SA (2011) [5] has designed and produced bio-oil from agricultural waste raw materials, such as coconut shells, coconut husks, and rice husks. The GC/MS test has revealed that the bio-oil produced from rice husks and corn cobs contains high methyl ester content for ± 58-70%. Further research by Novita, SA et al. (2014) [6] investigated methyl esters from coconut shells to separate their boiling points and test diesel engines. The test was conducted by mixing methyl esters in B10–B60 concentrations to start a diesel engine. Rice milling process produced 20–30% husk, bran 8–12%, and 52% hulled rice weighing [7]. One alternative to increase the benefits of rice husk is the pyrolysis process. This process decomposite compounds from carbon-containing materials at a high temperature to produce charcoal, liquids, and gases [8].

Pyrolysis is a thermochemical method of converting waste biomass into solid fuel (char), gas (syngas), or liquid (bio-oil) in oxygen in the reactor [9]. The thermal conversion process for biomass valorization or pyrolysis is the thermal degradation of organic material by cracking chemical bonds in the partial or total absence of an oxidizing agent [10]. According to Basu (2010) [11], biomass pyrolysis generally occurs at a temperature of 300–600^{o}C. The pyrolysis process usually comes at above 300^{o}C for three to four hours, but this will depend on the feedstock and method used [12]. Bio-oil and charcoal are obtained from the pyrolysis process of biomass, which constitutes indirect combustion at high temperatures of 350-600^{0}C with a heating rate of 10^{0}C/min [13]. The type of biomass used in pyrolysis combustion will affect the bio-oil composition. Meanwhile, the amount of bio-oil produced is influenced by several interacting factors, such as temperature and temperature rise rate [14].

The liquid produced from pyrolysis is the initial formation of bio-oil, which, in the next process, is converted to biodiesel or bioethanol. This research aims to determine the characteristics of rice husk bio-oil, test biodiesel performance using a diesel engine, obtain the right biodiesel blend (B20–70), and evaluate exhaust emission from the diesel engine.

2. Materials and Methods

The raw material used in the research was rice husk from the Harau District, West Sumatera Indonesia. Rice husk comprises about 20% of the weight of rice with the following composition: cellulose (50%), lignin (25%–30%), silica (15%–20%), and moisture (10%–15%) [15]. The tools used in this research were pyrolysis, rotary evaporator, GC/MS instrument, workshop equipment, labor equipment, stopwatch, digital thermometer, diesel engine, and equipment for the biodiesel characteristics analysis. The research procedure was carried out in several stages: preparing raw materials, manufacturing bio-oil, purifying bio-oil, testing bio-oil compounds, analyzing equipment performance, and testing the characteristics of methyl esters.

3. Results and Discussion

3.1 Rice Husk Pyrolysis Process

This study used rice husk which is rich in cellulose, lignin, and hemicellulose to make the bio-oil. The pyrolysis process requires 8-11% of rice husk water content. [17] recommends that water content in raw material for bio-oil pyrolysis does not exceed 8%. Increased water content in raw material will lower the phenol, acids, and formaldehyde content in smoke, increase the carbonyl compounds, and produce more acidic flavor [6]. Pre-treatment with high temperatures can produce various chemical compositions and physical properties [12]. Biomass pyrolysis involves extremely complex chemical and physical processes, such as heat transfer, mass transfer, thermal dynamics, and their interactions influenced by temperature, heating rate, biomass particle size and density, as well as physical and chemical pretreatments of the process [18, 19].

3.2 Bio-Oil Pyrolysis Fabrication

The components of pyrolysis fabrication (Fig. 1) include pyrolysis tube, smoke duct, tar collector, condenser, coil pipe, exhaust flue, bio-oil collector, water drain drum, and furnace.

Figure 1. Modified Pyrolizer

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3.3 Bio-Oil Production

Bio-oil is a liquid fraction produced from a biomass pyrolysis process at a temperature of 200-500°C. The Factors that influence the quantity of bio-oil yields are time and temperature of combustion. Tools and materials used in the performance test were 8–10% of water content, 2.5 kg of material amount, and 100 minutes of pyrolysis time. This study has found that the average temperature in pyrolysis is 379.3°C and the yield is 26.076%.

The most influential factor in the characteristics and yield of bio-oil are temperature, higher temperature, and better amount of bio-oil produced [20]. The continuously fluidized bed reactor is pyrolyzed in pinewood in two separate experiments. The details of pyrolysis experiments and the discussion about experimentally observed trends refer to our previous works [21, 22].

Pyrolysis has various temperatures based on the environmental conditions and fuels used, such as coconut shells and firewood. Firewood resulted in higher temperatures than coconut shells. Various temperatures have affected the average amount of bio-oil yield. This study has also found that the resulted yield is still low; thus, further research is necessarily conducted to create an improvement. Bio- oil could yield the most compared to biochar and non-condensable gases [23]. The processes and effects of various temperatures on the yield are presented in Table 1.

Table 1. Pyrolysis Reaction at Different Temperatures

Temperature	Processes	Products
Below 350 °C	1. Forming free-radicals 2. Eliminating water 3. depolymerizing compounds	1. Carbonyl and carboxyl compounds 2. CO and CO_2 gases 3. Charcoal residue
350–450°C	1. Breaking down and substituting glycoside and polysaccharide chain compounds	1. A mix of levoglucosan, 2. Anhydride and oligosaccharide from tar fraction
450–500°C	1. Dehydrating water 2. Combining and splitting glucose compound	Carbonyl compounds, such as acetaldehyde, glyoxal, and acrolein
Above 500°C	1. Combining all processes	Combination of all compounds
Condensation temperature	1. condensed unsaturated compound products 2. Separating unsaturated compound products from charcoal	High-quality charcoal that can catch free radicals

3.4 Bio-Oil Refining

The pyrolysis process produced blackish-brown bio-oil with high tar content that should be separated from the bio-oil to obtain grade 2 bio-oil with the assistance of a rotary evaporator. This tool separated substances based on boiling point differences for three to five hours. After being refined, the bio-oil was filtered using zeolite rock and active charcoal. The GC/MS analysis revealed the compounds contained in rice husk bio-oil as presented in Table 2.

Table 2. The compounds contained in the rice husk bio-oil

No	Compound	Percentage (%)
1	Ethanol	0.55
2	2-Butyl-1-octanol	0.26
3	Silane	0.12
4	Acetic Acid	1.73
5	Oleic Acid	1.97
6	Methyl Ester	52.62
7	Phenol	1.13
8	Trans-2-undecenal	1.38
9	Palmitaldehide	1.86
10	Methyl 9.9 Dideutero Octadecanal	29.48
11	Others	8.9
	Total	**100**

GC/MS analysis shows that bio-oil is recommended as biodiesel-based fuel [24]. The characteristics of the bio-oil from the pyrolysis process are presented in Table 3.

Table 3. Physical Properties and Characteristics of Bio-Oil Resulted from the Pyrolysis Process

Physical Properties	Bio-Oil Characteristics	Causes
Appearance	Dark brown to dark green	Micro carbon and chemical compositions in the bio-oil
Smell	Distinct and strong smell of smoke	Low molecule weight for aldehyde and acidic compounds
Density	1. 1.2 kg/lit of bio-oil density 2. 0.85 kg/lit of fossil fuel density	High moisture and high molecule weight
Viscosity	Viscosity ranging from 25 to 1000 cSt	1. Various kinds of raw materials 2. KA and light materials accumulated in the condensation process
Calorific values	Containing significantly lower calorific values than that of fossil fuel	Requiring a greater quantity of oxygen
Storage time	1. Increased viscosity 2. Decreased volatility 3. Separation and sedimentation phase proportionate with time	1. Complex compound structure 2. High pH values
Blending	Easily blended with polar solvents but not with fossil fuel	Naturally polar compound

A bio-oil quality was tested at the Centre for Fuel Technology and Design Engineering (BTBBRD). The tested parameters included total acid, water content, caloric values, and density of bio-oil. The results of this test are presented in Table 4.

Table 4. Characteristics of Bio-Oil

No	Parameters	Units	Rice Husk	Methods
1	Total Acid Number	Mg KOH/g	20.3429	ASTM D 664
2	Water Content (Karl Fischer)	%	88.73 ± 0.32	ASTM D4017-15
3	Calorific Values	Cal/gram	268	ASTM D5865
4	Volumetric Mass Density	Kg/m^3	1.2	ASTM D 1298

This study has discovered that the total acid number of the rice husk of bio-oil was 20.3 mg KOH/gram. This value is quite high since the total acid number set by the SNI Biodiesel 7182-2012 is 0.6 mg KOH/gram. The volumetric mass density is 1.2 kg/m^3 while that of biodiesel is 0.85 kg/m^3. Meanwhile, the calorific value is 268 cal/g or lower than that of biodiesel. This study tested bio-oil performance using the blending method and has found that bio-oil could not be used as a pure biofuel because it has high viscosity, high water content, high oxygen content, and low heating values [25].

3.5 Bio-Oil Testing Using a Diesel Engine

The bio-oil was tested using a 7.5 HP diesel engine blended with diesel fuel at certain ratios (B20, B40, B50, and B60). Thermal conversion by fast pyrolysis converts 75% of the starting plant material and its energy content into an intermediate bio-oil that is suitable for upgrading to motor fuel [26]. The results of testing bio-oil using a diesel engine are presented in Table 5.

Table 5. Blending Bio-oil and Diesel Fuel

No	Blending	Description
1	B20	The engine vibration is high. The produced smoke is colorless. The smoke has a slight smell of diesel fuel. The ignition is quick. The engine's rpm is 12,340. The diesel engine is powered up after more than 1.5 hours.
2	B30	The engine vibration is low. The produced smoke is colorless. The smoke has a slight smell of diesel fuel. The ignition is quick. The engine's rpm is 10,450. The diesel engine is powered up after more than 1.5 hours.
3	B40	The engine vibration is low. The produced smoke is colorless and has a smell of rice husk. The ignition is quick. The engine's rpm is 10,230. The diesel engine is powered up after more than 1.5 hours.
4	B50	The engine vibration is low. The produced smoke is colorless and has a smell of rice husk. The ignition is slow, the engine's rpm is 10,050. The diesel engine is powered up after more than 1 hour.
5	B60	In the beginning, the engine could be powered up but later went out and could not be powered up anymore.

There was a slight difficulty to power up the diesel engine when the B60 biodiesel fuel blend was used. However, this blend was still able to start up the engine. The produced smoke has no smell, and appears white, and is more environment-friendly.

The bio-oil is less stable than conventional fuels due to highly oxygenated compounds, high density and viscosity, low pH value, low calorific value, neutral CO_2, the absence of SOX, and low NOX. Burning a 2.5% bio-oil and a 97.5% HFO exhibits similar furnace performance and has lower NO and SO2 emission levels than burning pure HFO. This blend produces the reductions of NO and SO2 emissions by 2.6% and 7.9%, respectively [27].

4. Conclusions

Components of bio-oil pyrolysis equipment include pyrolysis tubes, smoke conduit pipes, tar catchers, condensers, coil pipes, discharge pipes, bio-oil reservoirs, water supply drums, and furnaces. The instrument performance test has discovered that the average pyrolysis temperature is 379.3^{0}C, the yield is 26.076%, and the average yield is 652 grams. The main compounds of this bio-oil are methyl esters, ethanol, silane, acetic acid, oleic acid, phenol, trans-2-undecenal, etc. The characteristics of the tested bio-oil signify that the total number of bio-oil acids from rice husk is 20.3 mg KOH/gram. The acid number is 0.6 mg KOH/gram or higher than the total acid determined in the SNI Biodiesel by 7182-2,012. The density is 1.2 kg/m3 or higher than the biodiesel by 0.85 kg/m3. Meanwhile, the heating value is 268 cal/g, or lower than the value of biodiesel heating. The bio-oil test was conducted using a 7.5 HP diesel engine. The test shows that the B10-B50 could start a diesel engine for more than an hour without producing smoke or lower the engine vibration.

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