Zeolite catalytic pyrolysis of waste tire into fuel in gasoline hydrocarbon range

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Abstract. The increasing production of automotive vehicles has led to a significant increase in the rate of waste tire generation. Approximately 160 tons/day of waste tire are produced in Bandung City which will eventually become an environmental problem. However, the pyrolysis process can be applied as a technology to treat waste tire to produce valuable hydrocarbon products. This study addressed the description effect of used tire waste on the yield, properties, and composition of tire pyrolysis oil (TPO) products. The effect of zeolite catalysts on TPO was also studied. 500 g of the waste tire was pyrolyzed using a small tube reactor containing a zeolite catalyst at various temperatures of 300–450 °C for 60 minutes. From the present work, the highest TPO product yield of 36.6 %-wt was obtained during the pyrolysis of the waste tire. The characteristics of TPO as a fuel, such as viscosity and density are close to those of gasoline fuel with a heating value of about 44 MJ/kg. The compounds contained in TPO were classified into hydrocarbon groups compounds as commercial fuel which are included as compounds of aromatics, paraffin, naphthenes, and cycloparaffins through the results of GC-MS spectrum analysis. The TPO from waste tire pyrolysis is on par with the hydrocarbon range of gasoline (C7–C11).

1. Introduction
Rubber is a hydrocarbon polymer made of latex derived from the tree sap of several types of rubber plants. The rubber tree of Hevea Brasiliensis (Euphorbiaceae) is the main source of the sap used. The rubber can also be made and synthetically, tire is an example of synthetic rubber made from non-biodegradable materials such as styrene-butadiene polymers. The number of tires that have been produced currently creates environmental and economic issues due to dumping and landfilling of the waste tire in large quantities is the worst way [1]. Therefore, it is necessary to prevent the harmful environmental impacts associated with the waste tire.
One source of this waste tire in the city of Bandung is the use of tire on automotive vehicles. The number of vehicle users leads to an increase in the waste tire produced. Approximately 10% of waste tire are produced from 1600 tons/day of solid municipal waste (MSW) in this city [2]. Although the percentage of waste tire is small, the problem is that waste tire is not easily decomposed, if it is just disposed of in the landfill. All the time, recycling into craft was accomplished to overcome this pile of waste tire. However, this recycling process will only convert waste tire into new forms and will not resolve a large amount of waste tire when the recycled product has lost its function and will return to waste. The difficulty in recycling this waste tire is due to main constituents of Styrene-Butadiene Rubber (SBR), Butadiene Rubber (BR) or Polybutadiene Rubber (PR), Isoprene Rubber (IR), besides that they also contain other material components such as carbon, metals, several other organic and inorganic components [3]. Nevertheless, the higher heating value of waste tire of 20.9 MJ/kg makes it very potential to be used as refuse-derived fuel (RDF) [4]. Waste tire pyrolysis is one of the effective technologies in producing useful products such as pyrolysis oil, carbon black, and pyrolysis gas (flammable gas) [5]. This waste tire can be converted into potentially recyclable compounds such as tire pyrolysis oil (TPO). TPO produced have a high heating value of around 41-44 MJ/kg [6] can be utilized to substitute liquid fuel for heating purposes or as an internal combustion fuel. This research focuses on the potential of TPO as a fuel including estimation of the yield, properties, and composition of TPO.

2. Zeolite Catalytic Pyrolysis of Waste Tire

The pyrolysis of the waste tire has thrived as a decent alternative to overcome improper waste tire disposal practices. Pyrolysis can be an alternative as the better method to recycle waste tire, pyrolysis will decompose volatile matters in waste tire thermally into liquid or gaseous products. Pyrolysis technology contributes not only to environmental problems but also to economic aspects through processing the waste tire into TPO as an alternative fuel. The waste tire has the potential for third-order bioenergy after oil palm and coconut.

Waste tire pyrolysis is a process in which organic matter in waste tire is heated to a temperature of between 300-900 °C in the absence of air. In this process, there are two stages of the process; primary pyrolysis in which the decomposition of waste tire (feedstock), and secondary pyrolysis is the cracking of volatile gases resulting from primary pyrolysis. The yield and composition of pyrolysis product from pyrolysis depends on the physical-chemical properties of the waste tire input, temperature, type of pyrolysis reactor, heating rate, and pyrolysis time [7]. The pyrolysis processes can be performed in a closed airtight batch reactor, semi-batch or continuous. Small-batch reactors or semi-batch reactors are more advantageous and effective for experimental and demonstration purposes [5]. Despite its a fairly good method for recycling old waste tire, conventional waste tire pyrolysis is subject to certain limitations on the dependence of impurities such as sulfur in TPO [8]. Hence, catalytic pyrolysis is quite interesting to be developed in improving the performance of conventional thermal pyrolysis. Catalysts of various types have been used, including natural zeolite to enhance and to improve the quantity and quality of TPO in catalytic pyrolysis [8-9].

Wongkhorsub and Chindaprasert reported that the product of waste tire pyrolysis consists of 35%- wt. TPO, 56 %-wt. residue and the rest is incondensable gases. It was also informed that the maximum TPO yield was achieved at 350 °C and decomposes into gases rapidly above 400 °C [6]. Osayi and Osifo stated that the maximum TPO yield of 21.3 %-wt. was obtained from the pyrolysis of waste tire at a zeolite catalyst ratio to waste tire of 7.5 %-wt. and a pyrolysis temperature of 600 °C with a heating rate of 15 °C./minute. The yield of PLO was lower than the yield of non-catalyzed TPO of 34.40 %-wt., nevertheless, the quality in terms of the range of hydrocarbon fuels produced gasoline (C_{6}-C_{12}) is higher than that of non-catalyzed PLO [10]. The PLO yields from 31.6 to 12.7 %-wt. were investigated from the catalyst/waste tire ratio from 0.25 to 1.0 in two stages of waste tire pyrolysis reported by Shen et al. Two-stage pyrolysis is the pyrolysis of waste tire in a fixed bed reactor followed by the pyrolysis of the resulting gas which is passed through a secondary catalytic reactor. The obtained PLO contains predominantly single-ring aromatic compounds of benzene, toluene, etc. [11]. Some other similar research about the pyrolysis of the waste tire has also been reported by several previous researchers in
Refs. [5-6], etc. In general, TPO from uncatalyzed waste tire pyrolysis has been evidenced to be highly aromatic with limited use as a substitute for liquid fuels. The use of catalysts in the pyrolysis of waste tire is possible to increase single-ring aromatic compounds, as long as the main compounds contained in PLO are benzene, toluene, xylene, etc.

Since 60–80% of the waste tire can be converted into PLO through catalytic pyrolysis [8], in this study, zeolite as a catalyst was used for cracking the gases produced the pyrolysis process of a waste tire. Thus, the PLO yield could be increased with better quality for further extraction into other useful chemicals. Zeolite catalyst was chosen with consideration of being more economical due to its ease to create from natural zeolite which is abundantly available and easy to be found in the local area [9].

3. Experiment Work
The waste tire pyrolysis system and all elements of the experiment stand are shown in Figure 1. The pyrolysis process was carried out in a tubular pyrolysis reactor integrated with a heating and condenser unit. The pyrolysis unit was equipped with a stove-burner system using LPG as a heat source. In the experiment, a tubular pyrolysis reactor was connected to a condenser system to condense the pyrolysis vapor into PLO as the temperature decreased. Each connection between the reactor and the condenser was sealed with a chemical resistant paste to reduce leakage, withstand high temperatures up to 650 °C, and high pressures.

![Figure 1. The waste tire pyrolysis system and elements](image)

In this study, inner tube waste tire as feedstock for the pyrolysis process. An amount of inner tube waste tire was prepared by cleaning, drying, and weighing. The waste tire plate was cut to sheets by the size of 10 mm x 20 mm. 500 g of the waste tire was introduced into the reactor, then 50 g (10% by weight ratio of zeolite catalyst to waste tire) was placed on top in the reactor. Zeolite is made from solid natural zeolite granules with a diameter of about 10 mm by washing, drying, and calcining at 120 °C.
and 500 °C, respectively. There is no significant difference in TPO yield between the use of activated and inactivated zeolite catalysts in the pyrolysis process from previous studies [9]. The cooling water pump was turned on before the reactor was heated by a heat source from LPG fuel. By the heating rate of 22.5-37.5 °C/minute, the waste tire pyrolysis process was carried out for each experiment at temperatures of 300 °C, 400 °C, 450 °C for one hour, respectively. The experiment was executed 5 times run for each temperature variation in order to obtain accurate results. The resulting TPO was collected in a conical collecting flask at the end of the condenser and then the amount was measured, analyzed for its properties and composition as a fuel. The undecomposed solid residues were also collected from the pyrolysis reactor after a complete pyrolysis process.

The main product of TPO obtained from pyrolysis was calculated to find out how much waste tire could be converted into fuel oil through the pyrolysis process. Physiochemical properties of the TPO were evaluated and determined to meet the gasoline hydrocarbon range. The viscosity of TPO was measured following to procedure for measuring the density of fuels using pycnometers described in American Standard Testing and Material (ASTM) D1217 and repeated three times to ensure accuracy. PLO viscosity is measured and calculated in centistoke (pa.s) using a precise and accurate Brookefield viscometer. The heating value of TPO was through calculations using widely used, inexpensive, and accurate correlation for the prediction of higher heating values of liquid fuels [9, 12]. The TPO obtained from the pyrolysis of waste tire in different conditions was investigated for its composition by Gas Chromatography-Mass Spectrometry (GC-MS). Considering the complexity and many components in the TPO resulting from tire pyrolysis, the components resulting from the GC-MS spectra analysis are group into hydrocarbon compounds as fuel. Hereinafter, this group of hydrocarbon compounds was regroup based on the number of carbon chains as the fuel of gasoline, kerosene, and diesel.

4. Results and discussion
4.1 Products from waste tire pyrolysis
Waste tire pyrolysis temperatures of 300 °C, 400 °C, and 450 °C were reached in about 8, 16, and 20 minutes, respectively, with a heating rate of 22.5-37.5 °C/minute. The useful product yield of this waste pyrolysis after a complete pyrolysis time of one hour consists of 11.31%-36.63% TPO, 47.31%-76.38% and the rest is flammable non-condensable gas by weight (%-wt.). In this case, the non-condensable gas was calculated by the difference between the waste tire sample weights. Meanwhile, these non-combustible gases generally contain H₂, CO, CH₄, and hydrocarbon compounds from C₁ to C₅ that cannot be condensed were released into the ambient air. In this experiment, the TPO produced from the pyrolysis of tire waste was separated from the impurities by the layer method, where the bottom layer was the impurity and the top layer is the TPO. The yield of pyrolysis products is presented in Table 1.

| Temperature (°C) | TPO Yield (%-wt.) | Solid residue (%-wt.) |
|-----------------|-----------------|----------------------|
| 300             | 11.61           | 76.38                |
| 400             | 30.31           | 50.41                |
| 450             | 36.63           | 47.91                |

Figure 2. Solid residue test as adsorbent
Banar et al. recorded that waste tire pyrolysis at a temperature of 400 °C with a heating rate of 5 to 35 °C/minute provided the TPO of 33.8 and 35.1 %-wt., respectively. TPO yield decreased with the increasing heating rate [13]. Hossain et al. carried out pyrolysis of waste tire with a ratio zeolite catalyst to waste tire of 10 %-wt. at a pyrolysis temperature of 580 °C produce TPO yield of about 35.83 %-wt. [14]. As above described, Osayi and Osifo also informed that TPO yield of 21.3 %-wt. could be attained from the pyrolysis of waste tires at pyrolysis temperature of 600 °C and a heating rate of 15 °C./minute using zeolite catalyst ratio to waste tires of 7.5 %-wt. [10]. The different trends for TPO yields with varying heating rates and pyrolysis temperature have been reported by some researchers. Although the result of this study was in agreement with the results of previous researchers, the slight difference in TPO yields may be related to heat and mass transfer leading to the formation of different products that affect TPO yields [8]. Other influences may be residence time in reactor and reactor type.

As long as the solid residue resulting from the pyrolysis process is the most product. This residue can be considered as a value-added product for activated carbon. As a preliminary work in this study, the solid residue produced was tested by the color absorption method using butadiene solution. The result shows the yellow betadine solution becomes clear after adding solid residue, as presented in Figure 2. This shows that the residual solids from the pyrolysis of waste tire have the potential to be used as activated charcoal or absorbent.

### 4.2 Effect of temperature on TPO yield

The effect of temperature on the yield of TPO with and without impurities is depicted in Figure 3. This shows that the waste tire pyrolysis process produces varying amounts of TPO depending on the operating temperature. The average yield of PLO after separation from impurities at temperatures of 300 °C, 400 °C, and 450 °C was 10.16 %-wt., 30.17 %-wt., and 34.55 %-wt., respectively. The TPO yield increased rapidly when the temperature was increased from 300 °C to 400 °C, however slightly increased when the temperature was increased from 400 °C to 450 °C. This indicates that an increase in temperature above 450 °C will not obtain a significant TPO yield. This means that pyrolysis can be carried out through a slow pyrolysis process. Pyrolysis at low to medium temperatures produces more liquid products than gas products [15]. The pyrolysis temperature of 450 °C is a suitable condition considering the heat (energy) consumption.

![Figure 3. The effect temperature on TPO yield](image-url)
The pyrolysis temperature is obviously an important factor affecting the product yield. The yield of TPO and pyrolysis gas increased with increasing temperature while the yield of solid residue (charcoal) decreased with increasing temperature. High temperature also affects the reduction of the liquid product, while the resulting gas product increases [16]. The increase in yield of pyrolysis products due to increasing temperature is caused by the breaking of the C-C bonds from long carbon chains in waste tire with large molecular weights into short carbon chains (monomers) with small molecular weights. Usually during pyrolysis, it is initiated by a unimolecular dissociation reaction followed by the segregation of H atoms and addition reactions by hydrogen atoms (H) and methyl radical molecule (CH₃). The more bonds (hydrocarbon chains) that are broken, the more yield increases [9-16]. The high temperature also affects the reduction in TPO yield along with the increase in the yield of gas products. This is possible because of the secondary cracking process in the form of breaking long chains of organic compounds and hydrocarbons in the waste into shorter chains so that they cannot be condensed again. This is possible because the secondary cracking process is the breaking of long chains of hydrocarbons compounds in the waste tire into incondensable of shorter chains hydrocarbons compounds. Large hydrocarbon compounds are pyrolyzed until a threshold temperature, below this temperature at this pressure and residence time of pyrolysis the hydrocarbons do not decompose [17].

4.3 Effect of zeolite catalyst on TPO yield
Some researchers have stated that the addition of zeolite catalysts in the waste tire pyrolysis process promotes gas product yield while decreasing the TPO and char yield. Zeolite catalysts also affect the quality and quantity of TPO produced because the catalyst increases the decomposition reaction which results in a large number of long-chain hydrocarbons being broken down into short-chain hydrocarbons [8-9]. The zeolite catalyst to waste tire also impacts the yield of pyrolysis products. The ratio of zeolite catalyst to waste tires is 10 % wt. and 0.15 % wt. at an optimum pyrolysis temperature of 460 °C gave provided the increases in the char and TPO yields (35.83 % wt.) while decreasing gas yields were also reported by Hossain et al. [14]. This is in accordance with the results of the study that the average yield of PLO after separation from impurities at a temperature of 450 °C was 34.55 % wt. The presence of a zeolite catalyst also influences the properties of TPO as fuel, such as the density, viscosity, and energy content. Thus, zeolite catalyst is believed to be suitable to enhance the quantity and quality of TPO in the pyrolysis process.

4.4 Quality of TPO
TPO as fuel is identified by certain basic properties such as density, viscosity, and heat value. Density is one of the important parameters of a fuel, TPO density as a fuel indicates the content of aromatic compounds in TPO to estimate volumetric consumption in its application. While the viscosity of TPO is another key factor that must be considered to get good combustion. High-density fuel will affect engine performance and trigger increased emissions of gases such as carbon monoxide (CO) and soot from carbon dioxide (CO₂). High fuel viscosity will induce ignition delay, meanwhile, low viscosity fuels are ideal for contributing to pump pummability and engine efficiency [18-19]. Another important parameter of TPO as a fuel is the higher heating value (HHV). The high heating value of the TPO signifies that the TPO can be applied directly as a fuel. HHV of TPO of around 41-44 MJ/kg is suitable to substitute liquid fuels for heating purposes [6-8]. The characteristics of TPO in terms of density, viscosity, and calorific value compared to the characteristics of several fuels are listed in Table 2.

In Table 2, it can be seen that the TPO density was also very close to that of gasoline fuel and slightly lower than that of kerosene and diesel fuel. There is a slight difference in TPO viscosity obtained at each operating temperature, the higher the operating temperature, the higher the viscosity value obtained. At a temperature of 300 °C, the TPO viscosity is close to the gasoline viscosity value. The TPO viscosity obtained from the pyrolysis of the waste tire at temperatures greater than 300 °C is outside the range of values specified for gasoline (ie., 0.00040-0.00075 Pa.s). TPO fuel with high viscosity may induce improper atomization leading to incomplete combustion. On the other hand, the too low viscosity of fuel can induce excessive evaporation and the amount of unburned hydrocarbon compounds is very high.
It is advantageous to determine a slightly lower or higher viscosity of gasoline fuel to meet good combustion.

Table 2. The characteristics of TPO and commercial fuels

| Fuel Characteristics | Pyrolysis Temperature (°C) |
|----------------------|---------------------------|
| Tire pyrolysis oil (TPO) | 300 | 300 |
|                      | 400 | 400 |
|                      | 450 | 450 |
| Density (g/cm³)      | 0.63 | 0.65 |
|                      | 0.69 | 0.68 |
|                      | 0.0086 | 0.00136 |
| Viscosity (Pa.s), 20 °C | 0.00136 | 0.00136 |
|                      | 0.00258 | 0.00258 |
|                      | 44.8492 | 44.6014 |
| HHV (MJ/kg)          | 44.7688 | 44.7688 |
|                      | 43.2709 | 43.2709 |

The heating value of fuel oil is the energy content of the fuel when it is completely combusted with sufficient air. The HHVs of TPO are very similar to that of commercial gasoline and kerosene. Some researchers reported that tire pyrolysis oil has HHV in the 30-40 MJ/kg [14, 20-21]. Energy recovery from waste tire through pyrolysis of 49.5 and 46.7 MJ/kg was reported by Rada et al. [20]. Work done by Rodriguez et al. declares the HHV of TPO of 42 MJ/kg was obtained from pyrolysis of waste tire [21]. Thus, the HHV of TPO from this study is comparable to the results of previous researchers and on par with gasoline fuel.

GC-MS was used to investigate the effect of pyrolysis parameter and zeolite catalyst to identify the compounds contained in TPO and classify them into hydrocarbon groups compounds as commercial fuel which are included as compounds of aromatics, paraffin, naphthenes, and cycloparaffins. More than 500 compounds in TPO were identified through the peak chromatogram results of GC-MS. The percentage fraction of hydrocarbon groups compounds was determined based on the standard retention time. This group of hydrocarbon compounds was regroup based on the number of carbon chains (carbon range) as the fuel of gasoline, kerosene, and diesel. Gasoline standard retention time ≤ 18, standard kerosene ≥ 18 - ≤ 22.85 and standard diesel ≥ 22.85. With the retention time interval for each fraction, the percent of gasoline, kerosene, and diesel fractions in hydrocracking products can be calculated based on the percent area of the GC-MS analysis result by dividing by the total area of GC MS. The content of the hydrocarbon compounds group in TPO is as presented in Table 3.

Table 3. The content of hydrocarbon compounds group in TPO

| Hydrocarbon Compounds Group | 300 °C | 400 °C | 450 °C |
|----------------------------|--------|--------|--------|
| Aromatics (C₈H₈)          | 13.14  | 21.23  | 18.92  |
| Paraffins (C₆H₁₃)         | 3.7    | 3.6    | 1.88   |
| Naphthenes (C₇H₁₄)        | 6      | 4.37   | 4.41   |
| Olefin (C₇H₁₄)            | 0.25   | 0.15   | 0.25   |
| di-olefin (C₈H₁₄)         | 3.53   | 9.49   | 7.55   |
| Cycloparaffins            | 13.13  | 1.05   | 3.25   |
| Naphthalene               | 3.66   | 7.51   | 8.08   |
| D-limonene                | -      | 7.88   | 5.98   |
| etc.                      | 56.59  | 44.72  | 49.68  |
| Total                     | 100    | 100    | 100    |
The aromatic compounds in TPO from pyrolysis of wastes tire have the highest fraction concentration (13.14 %), and they are followed by naphthene (6 %), di-olefins (3.53 %), and cycloparaffins compounds (13.13 %). This result is in accordance with the results reported by Pavlopa et al. [22].

Hydrocarbon group compounds as fuel in TPO are regrouped into three types of fuel based on the number of carbon chains, namely gasoline (C₇-C₁₁), kerosene (C₁₂-C₁₅), diesel (C₁₆-C₂₀), and other compounds as presented in the above figure. In Figure 4, the higher the pyrolysis operating temperature, the lower the content of hydrocarbon products as commercial fuels, while other compounds increase due to the cracking of these hydrocarbon compounds. In Figure 5 it can be explained that the yield of hydrocarbon compounds in the range of kerosene and diesel is decreasing. This is possible because at high temperatures, the long hydrocarbon chains in TPO become shorter which triggers an increase in the gasoline yield fraction.

According to the description above, it can be stated TPO show similar fuel properties with gasoline from the point of density, viscosity, and HHV. The GC-MS analysis of TPO also shows the waste tire pyrolysis oil is wholly similar to gasoline hydrocarbon (C₇-C₁₁) than kerosene (C₁₂-C₁₅) with their significant concentrations of aromatic and cycloparaffins hydrocarbons.

Figure 4. Effect of temperature on compounds as fuel in TPO
5. Conclusions

The pyrolysis of waste tire with zeolite catalyst-tire ratio 0.1 %-wt. was successfully performed. The highest tire pyrolysis oil (TPO) yield of 36.63 %-wt. was achieved at 450°C, where increasing the pyrolysis temperature did not lead to a significant increase in yield. Based on fuel properties in terms of density, viscosity, and HHV denote similar fuel properties with gasoline. The GC-MS analysis of TPO signifies the major hydrocarbon compounds of aromatic and cycloparaffins. This TPO can be recommended as an environmentally friendly fuel to substitute commercial fuel due to its characteristics on par with commercial gasoline fuel.

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**Acknowledgment**
The authors would like to acknowledge the Indonesia Ministry of Education, Culture, Research, and Technology for providing funds to complete this work under the research project PTUPT grants for the year 2021-2022 with contract number of 310/SP2H/LT/DRPM/2021 and thank LPPM-UNJANI for financial support through competitive research.