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FTIR ANALYSIS OF PYROLYSIS OF POLYETHYLENE TEREPHTHALATE (PET) PLASTIC AND ITS PYROLYSIS MECHANISM COMPLETED WITH BIBLIOMETRIC LITERATURE REVIEW FOR SUPPORTING CURRENT ISSUES IN SUSTAINABLE DEVELOPMENT GOALS (SDGS)

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Abstract

The purpose of this study was to analyse the chemical components from pyrolyzed polyethylene terephthalate (PET) using Fourier transform infrared (FTIR). PET particles were pyrolyzed at temperatures between 120 and 277.7°C for 105 minutes in the batch reactor connected to two condensers (24°C). Condensers were set series to the output of the reactor and all systems were closed. The first condenser was connected directly to the reactor, whereas the second condenser was connected to the first condenser. From the pyrolysis process of PET, the liquid sample, residues, and gas were obtained. The colourless liquid sample with a distinctive odour was obtained in the first and the second condenser. The residue was black and solid. Both condenser samples contained similar groups, such as OH groups, C-H groups, C=O groups, and C-H groups of alkenes. The most dominant compounds in the product were benzoic acid and water as the side product. This pyrolysis process showed the occurrence of degradation and oxidation reactions by breaking the hydrocarbon chain into short chains. This causes terephthalic acid to oxidize to produce benzoic acid and water. This research had an impact on the management of PET plastic waste by understanding the compounds contained in PET plastic after pyrolysis. Finally, this study can be a problem solver to solve current issues in the sustainable development goals (SDGs).

Keywords: Characterization, FTIR, Plastic waste, Polyethylene terephthalate, Pyrolysis.

1. Introduction

Polyethylene terephthalate (PET) is one of the most widely available thermoplastic polymers on the trade market [1]. PET is a semicrystalline and transparent thermoplastic that has high rigidity, mechanical strength, and good chemical resistance. The molecular formula of PET is (-CO-C_6H_5-CO-O-CH_2-CH_2-O-)n. PET is widely used to make soft drinks and water bottles, synthetic fibre, video and audio tapes, photographic film, food packaging, and others [2]. The demand for PET in 2016 was 8400 kilotons, and it is expected 6.9% in 2017-2025 [3]. Although PET is useful, the presence of PET waste in the environment causes serious problems because the final product obtained from PET takes around 300–450 years to decompose naturally [4]. Figure 1 shows research trends in PET waste management based on bibliometric analysis using the Scopus database taken in May 2024. Bibliometric is one of the effective methods and has been used to understand current research trends in many subject areas [5-9], and even journals [10-12]. Detailed information on how to use bibliometrics is explained elsewhere [13].

Table 1 describes some research on plastic waste management. In the pyrolysis process, biomass and polymers undergo bond breaking to form molecules with smaller dimensions and sizes. Biomass pyrolysis is universally the decomposition of organic matter creating solid materials in the form of activated charcoal, gas as well as vapours and aerosols. Gas that can be condensed is known as biofuel or bio-oil [14]. Previous research showed that pyrolysis oil can be produced from 500 g of PET plastic within 6 hours. From the analysis performed, pyrolysis oil has a density of 0.688 g/mL, which is between the density of kerosene and fuel oil. The research shows that pyrolysis is an effective method to convert PET plastic waste into fuel oil, which not only reduces plastic waste but also offers an environmentally friendly fuel alternative. In addition, this study also shows that the quality of pyrolyzed oil can be similar to kerosene and fuel oil standards, showing the commercial potential and practical application of this technology [15].

From the results of plastic waste processing experiments, the type of plastic waste that produces the most oil is PET plastic waste, and the least oil is produced from the combination of PET and HDPE plastic waste. The plastic waste treatment that showed the greatest weight reduction was PET and the combination of PET and HDPE. Pyrolysis cannot produce oil less than 1200 mL from PET samples. The pyrolysis machine cannot produce less than 1200 mL of oil from HDPE samples. The use of pyrolysis equipment cannot produce oil below 1200 mL from oil that contains PET and HDPE [16].

Different from other studies, here, this study aims to analyse the chemical components from pyrolyzed PET using Fourier Transform Infrared. 400 g of PET particles with a size of 500 µm were pyrolyzed at temperatures of between 120 and 277.7°C for 105 minutes in the batch reactor (length = 12 cm x width = 8 cm x height = 5 cm) connected to two condensers (24°C). Condensers were set series to the output of the reactor and all systems were closed. The first condenser was connected directly to the reactor, whereas the second condenser was connected to the first condenser. The novelty of this study was the detailed information regarding the analysis of PET plastic after the pyrolysis process using Fourier Transform Infrared (FTIR). This study was done without any additional catalysts.

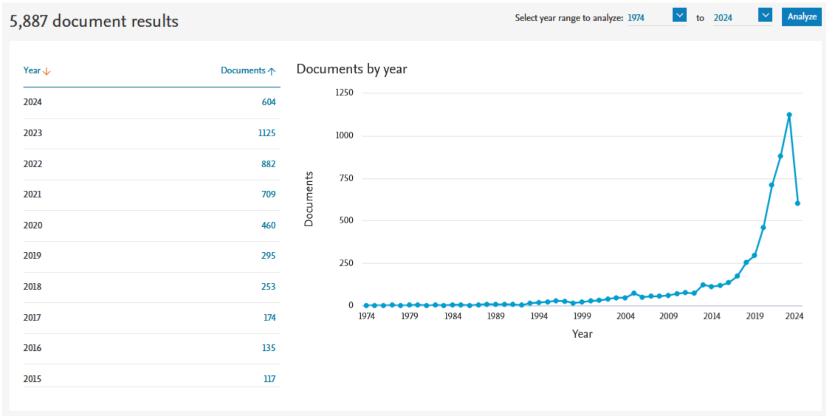


Fig. 1. Previous studies on “PET waste” management based on the Scopus database, taken on May 2024.

Table 1. Plastic waste treatment research data.

No.	Title	Note	Ref.
1	Utilization of recycled plastic waste from polyethylene terephthalate (PET) as an additive in the manufacture of nanocomposites, cement mortar, and asphalt	The results of this study showed that PET waste can be used as an additional material in the manufacture of nanocomposites, cement mortar, and asphalt. In cement mortar production, the addition of PET waste can significantly improve the smooth flow by increasing the percentage of the amount of PET plastic waste used.	[17]
2	Utilization of plastic waste into fuel oil	In this study, sampling was carried out using the grab-sample method, with the condition that per experiment 1 kg sample. Thus, the total sample with three times replication was 3 kg of PET plastic waste. According to the research results, the pyrolysis device can produce 201.67 mL of fuel oil at an optimal temperature of 270ºC. In the pyrolysis process, temperature and time are very important. In addition, solid residues from polyethylene terephthalate plastics can be processed into paraffin, which is the raw material for candle making.	[18]
3	Processing PET (polyethylene terepthalathe) type plastic waste using pyrolysis method into alternative fuel	The method used is a simple pyrolysis technique. The results of this study showed that the most effective pyrolysis temperature of PET is >250°C. The oil yield from the parallel flow testing is more abundant than counter flow testing. The largest volume of oil is produced in the range of 260ºC to 350°C. Condenser 1 produces more oil compared to condenser 2. The oil produced by Condenser 2 is purer because it contains less heavy hydrocarbon matter.	[19]
4	Processing plastic waste into fuel oil to overcome plastic waste in Bontang City	The processing of thermoplastic polyester-type plastic waste into fuel oil using a slow pyrolysis method with simple equipment shows high effectiveness. From 1.4 kg of plastic waste, 350 mL of fuel oil was obtained at a temperature of 225 °C with a residence time of 4 hours.	[20]

		However, if the temperature is raised to 400-600°C, the volume of oil produced will likely increase.	
5	Utilization of LDPE and PET plastic waste into oil fuel by pyrolysis process	The results showed that pyrolysis of Low-Density Polyethylene (LDPE) plastic waste produced 525 mL of oil from 1 kg of LDPE plastic waste, while pyrolysis of PET plastic waste produced 368.47 mL of oil from 1 kg of PET plastic waste. The quality test of fuel oil from LDPE plastic waste shows a density value of 0.7673 kg/L, close to the density of kerosene which ranges from 0.78-0.81 kg/L. The viscosity value is 0.7923 cP, included in the kerosene viscosity range which ranges from 0.294-3.34 cP. Flashpoints cannot be compared due to the limitations of test equipment that cannot measure above -5, and firepoints do not have a standard. The calorific value of LDPE oil is 44.0533 kg/L, close to the calorific value of diesel oil which is 44.8 kg/L. The quality test of fuel oil from PET plastic waste shows a density value of 0.7976 kg/L, which corresponds to the density of kerosene ranging from 0.78-0.81 kg/L. The viscosity value is 1.2217 cP, included in the kerosene viscosity range which ranges from 0.294-3.34 cP. Flashpoints are also incomparable due to limitations of test equipment that cannot measure above -5, and hotspots do not have a standard. The calorific value of PET oil is 42.6224 kg/L, close to the calorific value of kerosene which is 43 kg/L.	[21]
6	Utilization of plastic waste through process pyrolysis as an alternative fuel	From the pyrolysis process of PP and PET-type plastics, the fuel content of PP-type plastic was higher, which was 91.56% of 1 kg of raw materials, while the PET type produced 35% of 1 kg of raw materials. *milpa* has a higher flame temperature of 242.67°C and a longer flame time of 1.44 s, while premium has a flame temperature of 238.33°C and a flame time of 0.48 s. The engine speed produced by premium is higher at 659.3 rpm compared to *milpa* which produces 613.6 rpm. Thus, *milpa* has the potential to be developed as an alternative fuel equivalent to premium.	[22]

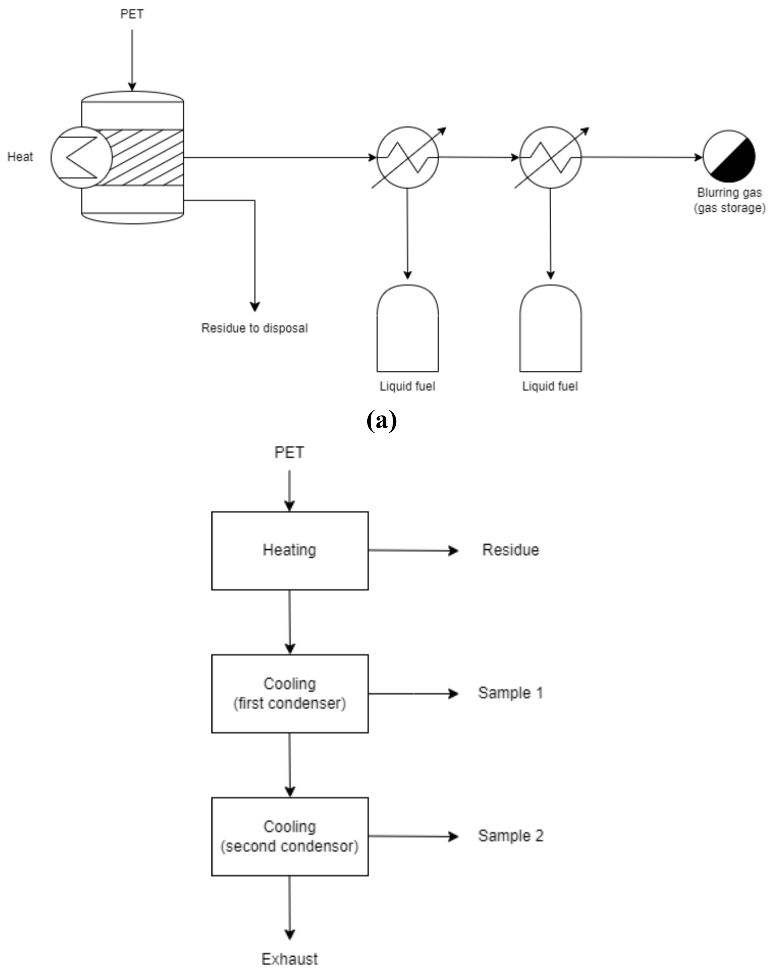
The advantage of this pyrolysis technology research is that it is relatively flexible and can be used at various scales, including in remote locations and rural areas. This research can be a solution in hard-to-reach areas with traditional waste treatment infrastructure. This research had an impact on the management of PET plastic waste by knowing the compounds contained in PET plastic waste. Finally, this study can be a problem solver to solve current issues in the sustainable development goals (SDGs).

2. Method

The equipment for preparing the pyrolysis process was done by setting three pieces of cans (length x width x height = 12 cm x 8 cm x 5 cm), 1 aluminium pipe with a diameter of 8 mm, 2 pieces of aluminium pipe with a diameter of 12 mm, 1 set of thermocouples, gas stoves, digital scales (10 kg), 2 vial bottles, 2 pieces of the basin, and a silicone hose (diameter = 1 m). The materials were 400 g of PET plastic waste, 3 kg of LPG gas, plasticine, water, and dextone glue. The pyrolysis reactor was connected to two used cans using two pieces of iron pipes (cans and iron pipes are heat conductors). Then, the series condenser tool set was assembled by placing

2 containers (as condensers) filled with water at the bottom of the second and third cans. Next, the sample container bottle is connected to the third can.

The reactor was placed on the stove. The PET plastic waste sample (400 g) was put into the reactor (working volume of about 80%). Next, the pyrolysis reactor was a closed system (i.e. air from outside the reactor can't enter the system, and gas from the reaction cannot come out except to the designated outlet that was connected to the condenser). The stove was used until the PET plastic waste melted. We periodically checked the temperature using a thermocouple. Then, the results were observed until the PET plastic melt vapor from the reactor moved to the condenser and finally accommodated in the oil container bottle. Figures 2(a) and 1(b) are the equipment design and flowchart of the PET pyrolysis procedure. Samples were then collected and characterized using FTIR (FTIR Shimadzu-8400). Detailed information regarding FTIR is explained elsewhere [23-25].

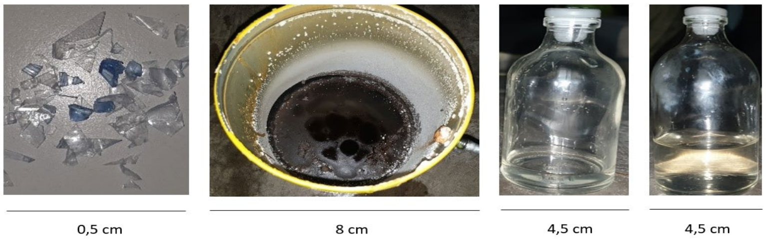


(b)

Fig. 2. (a) Design of PET plastic waste pyrolysis process tool, (b) Flowchart of PET plastic pyrolysis.

3. Results and Discussion

Pyrolysis was carried out for 105 minutes. We checked the temperature every 15 minutes. The pyrolysis products were liquids produced from both condensers. In the first condenser, 3.90-mL colourless and distinctively smelling liquid was obtained. In the second condenser, 30-mL liquid was obtained in the form of a colourless liquid with a distinctive smell. In the reactor, the black and solid residue was obtained. Figure 3(a) is the form of PET plastic waste before the pyrolysis process. Figure 3(b) shows the results of the pyrolysis process of PET plastic waste in the reactor. Figure 3(c) is the result of the pyrolysis of PET plastic in the first condenser. Figure 3(d) is the result of the pyrolysis of PET plastic waste in the second condenser.



(a) (b) (c) (d)

Fig. 3. (a) Waste PET Ore (b)The result of the pyrolysis process of PET plastic waste in the reactor section, (c) The pyrolysis result of the first condenser, and (d) The pyrolysis result of the second condenser.

Based on the product obtained, there are several differences between the first and the second condenser. The second condenser has a lower temperature, allowing the liquid to crystallize and collect more in it. Conversely, the first condenser has a less effective design or a higher temperature resulting in the liquid evaporating faster and not collecting as much as the second condenser. The scale of comparison between the results of the first condenser with the second condenser is 2:15. Pyrolysis products are characterized using the Shimadzu-8400 Fourier Transform Infrared Spectroscopy (FTIR) instrument to see the functional groups contained in the first and the second condenser. Figure 4 illustrates the comparison of the FTIR spectra in samples from condensers 1 and 2, PET, and benzoic acid.

Figure 4 shows four comparative FTIR spectra, including PET spectrum; sample from condensers 1 and 2, and benzoic acid. The wavenumber scale of the spectrum was set at 500-4000 cm^{-1} and the transmittance was 20-100%. The peaks provide information in the form of functional groups on PET, sample 1, sample 2, PET, and benzoic acid. The FTIR spectrum of PET shows significant peaks for PET characteristics, such as the 712 cm^{-1} wavelengths indicating the interaction of polar ester groups and benzene rings. In the FTIR spectrum of samples from condensers 1 and 2, there is a wide OH group at 3350-3450 cm^{-1}, indicating that the compound contains water. In the spectrum of benzoic acid, there is an aromatic ring group or C-C bond of the acid group at 900-1100 cm^{-1}. Table 2 shows the comparison between PET, sample 1, sample 2, and benzoic acid FTIR peaks.

Table 2 shows the FTIR spectrum. The first sample obtained 4 peaks. The first peak with a wavenumber of 678.97 cm^{-1} describes the presence of a C-H stretching functional group (alkene), while the second peak with a wavenumber of 1635.69 cm^{-1} indicates a C=O functional group. The 2067.76 cm^{-1} wavenumber at the third peak indicates the presence of a C-H alkane functional group, and the fourth peak with a wavenumber of 3448.84 cm^{-1} detects a functional group which is the O-H hydrogen bond (H_2O). One of the characteristics of hydrogen bonding in the FTIR spectrum is a widening peak that has a wavenumber of more than 3000 cm^{-1}. This indicates that the sample still contains a lot of water. Therefore, this sample cannot be further tested using the GC-MS instrument.

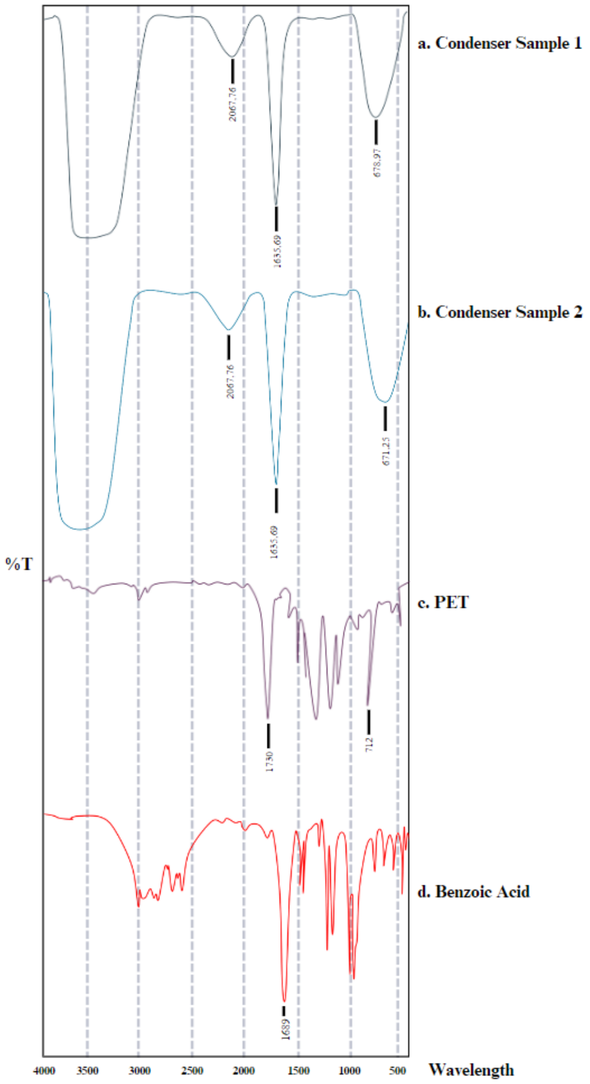


Fig. 4. Comparison of IR spectra on PET, condenser sample 1, condenser sample 2, and benzoic acid.

Table 2 shows FTIR spectra for the sample from condenser 2, obtaining 5 peaks with different wavenumbers. At the first peak, the wavenumber 401.21 cm^{-1}

describes the presence of C-C vibrations. The second peak with wavenumber 671.25 cm^{-1} describes the presence of a C-H stretching functional group (alkene). The third peak with a wavenumber of 1635.69 cm^{-1}, indicates a C=O functional group. The wavenumber 2067.76 cm^{-1} at the fourth peak indicates the presence of a C-H alkane functional group. For the fifth peak with a wavenumber of 3394.83 cm^{-1}, it is identified as the O-H (HO) functional group. One of the characteristics of hydrogen bonding in the FTIR spectrum is a peak that has a wavenumber of more than 3000 cm^{-1} and a widening peak. This means that the sample still contains a lot of water, this sample cannot be tested further using the GC-MS instrument because the sample has water content (H_2O).

Table 2. Comparison of FTIR of PET, Sample 1, Sample 2, and benzoic acid.

No.	Wavelength (cm^{-1}) FTIR				Description
	PET	Sample 1	Sample 2	Benzoic Acid	
1	3432	3448.84	3394.83	2500-3000	Hydrogen Bonding O-H (H_2O)
2	3054	-	-	-	C-H Stretch
3	2969 and 2908	-	-	-	C-H stretching sp^3
4	2350	-	-	-	CO_2 axial
5	-	2067.76	2067.76	-	C-H alkane
6	1730	1635.69	1635.69	1680-1750	C=O stretching (carboxylic acids)
7	1577 and 1504	-	-	-	C=C aromatic
8	1453, 1410, and 1342	-	-	-	Stretching the deformation of the C-O group of the O-H group and bending and shaking the vibration of the ethylene glycol segment mode
9	-	-	-	1300	OH
10	1240 and 1124	-	-	-	Terephthalate group ($OOCC_6H_4$ -COO)
11	1096 and 1050	-	-	-	Methylene group and vibration of C-O ester bonds
12	972, 872, and 848	-	-	900-110	Aromatic ring or carbon-oxygen bond of the acid group
13	1960 and 795	-	-	-	Vibration of two nearby H-aromatics in a substituted-p compound and aromatic tape
14	712	-	-	-	Interaction of polar ester groups and benzene rings
15	-	678.97	671.25	-	C-H alkene
16	-	-	401.21	-	Vibration C-C

The FTIR for PET in Table 2 shows the presence of O-H groups at wavelengths 3432 cm^{-1}, C-H groups at wavelengths 3054 cm^{-1}, C-H sp^3 groups at wavelengths 2969 and 2908 cm^{-1}, axial CO_2 groups at wavelengths 2350 cm^{-1}, C=O stretching groups (carboxylic acids) at wavelengths 1730 cm^{-1}, aromatic C=C groups at wavelengths 1577 and 1504 cm^{-1}, stretching the deformation of the C-O group of the O-H group and bending and shaking the vibration mode of the ethylene glycol segment at wavelengths 1453, 1410, and 1342 cm^{-1}, the terephthalate group ($OOCC_6H_4$-COO) at wavelengths 1240 and 1124 cm^{-1}, the methylene group and the vibration of the C-O ester bond at wavelengths 1096 and 1050 cm^{-1}, the aromatic ring group at wavelength 972, 872, and 848 cm^{-1}, vibration of two adjacent H aromatics in a p-substituted compound and aromatic band at wavelengths 1960 and 795 cm^{-1}, and interaction of polar ester groups and benzene rings at wavelengths 712 cm^{-1} [26].

From the FTIR spectrum of benzoic acid in Table 2, there are 4 cluster peaks. The first peak with wavenumbers 2500-3300 cm^{-1} describes the presence of hydrogen bond functional groups O-H (H_2O). The second peak with wavenumbers 1680-1750 cm^{-1} indicates that at the peak there are functional groups C=O. The wavenumber 1300 cm^{-1} at the third peak indicates the presence of a functional group OH. For the fourth peak with wavenumbers 900-1100 cm^{-1}, the functional group was identified as the aromatic ring or carbon-oxygen bond of the acid group.

Based on the comparison results in Table 2, the spectrum of PET plastics has 12 peaks with different wavenumbers. In the FTIR for PET, the pyrolysis results have 4 and 5 peaks. When viewing the wavenumber, the pyrolysis PET does not have wavenumbers 2969 and 2908 cm^{-1} which are C-H stretching groups. In FTIR the results of PET pyrolysis, it is formed C-H alkanes at wavenumber 2067.76 cm^{-1} even though the pure PET has no C-H alkane functional groups but has C-H stretching sp^3 groups which are alkane groups.

The formation of C-H alkene groups in FTIR from PET pyrolysis results in a wavelength of 670 cm^{-1}, but pure PET has no C-H alkene functional groups. This has the possibility of thermal decomposition at high temperatures without the presence of oxygen, breaking chemical bonds in the polymer chain. In addition, there is the same group from the four data above at wavelengths 3000-3500 cm^{-1}, which is an OH group and there is a C=O group at wavelengths 1600-1750 cm^{-1}.

Figure 5 illustrates the reaction mechanism of the pyrolysis process from PET plastic ore waste raw materials. The reaction mechanism of the pyrolysis process begins with polymer degradation. Polymer degradation is an irreversible reaction process involving various chemical reactions. This process changes the properties of the polymer and eventually results in the breakdown of long chains of polymer molecules into small molecules.

Degradation starts from the surface of the polymeric material and slowly penetrates its molecular structure. This process depends on the structure of the polymer and other factors such as heat, light, radiant ions, and mechanical, biological, and enzymatic actions [27]. The general reaction of degradation of PET polymers is as follows (reaction (1)).

$$(C_{10}H_8O_4)n \rightarrow C_2H_6O_2 + C_6H_4(COOH)_2 \quad (1)$$

PET →Ethylene Glycol + Terephthalic acid

Furthermore, an oxidation reaction occurs in the hydrocarbon chain, if the reaction takes place perfectly, it will produce carbon dioxide gas and water molecules. Here is the general reaction equation for the oxidation of terephthalic acid to benzoic acid and the hydrolysis reaction of Benzoyl Oxide to Benzoic Acid (reactions (2) and (3)). Finally, this study can be a problem solver to solve current issues in the SDGs.

$$C_6H_4(COOH)_2 + O_2 \rightarrow 6H_4(CO)_2O + H_2O \qquad (2)$$

Terephthalic acid + Oxygen →Benzol Oxide + Water

$$6H_4(CO)_2O + H_2O \rightarrow C_6H5COOH + H_2O \qquad (3)$$

Benzol Oxide + Water →Benzoic Acid + Water

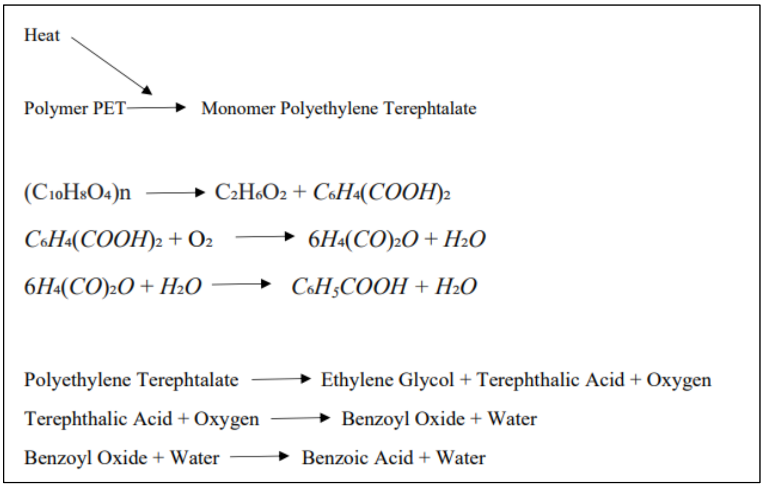


Fig. 5. Pyrolysis reaction mechanism of waste PET ore.

Finally, this study adds new information from our previous studies [28-31]. This study also can be a problem solver to solve current issues in the SDGs. This also adds new information regarding SDGs as reported elsewhere [32-36].

4. Conclusion

Based on the results of research on processing PET pyrolysis, oil, residue, and gas were obtained. The results of FTIR analysis of PET pyrolysis samples show that samples resulting from pyrolysis of PET may contain benzoic acid compounds with the side product of water. This pyrolysis process showed the occurrence of degradation and oxidation reactions by breaking the hydrocarbon chain into short chains. This causes terephthalic acid to oxidize to produce benzoic acid and water. This research had an impact on the management of PET plastic waste by understanding the compounds contained in PET plastic after pyrolysis. Finally, this study can be a problem solver to solve current issues in the SDGs.

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