

What Phenomena Happen During Pyrolysis of Plastic? FTIR AND GC-MS Analysis of Pyrolyzed Low Linear Density Polyethylene (LLDPE) Polymer Particles Completed with Bibliometric Research Trend and Pyrolysis Chemical Reaction Mechanism

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ARTICLE INFO	ABSTRACT
Article history: Received 2 October 2023 Received in revised form 13 January 2024 Accepted 22 April 2024 Available online 22 May 2024	This research aims to determine the compounds produced from the pyrolyzed Low Linear Density Polyethylene (LLDPE) using Fourier Transform Infrared (FTIR) and Gas Chromatography-Mass Spectroscopy (GC MS). We used pyrolysis of 985 g of 2-mm diameter LLDPE plastic ore at a temperature between 192-194°C for 135 min using a batch reactor (length x width x height = 35 cm x 24 cm x 44 cm) equipped with an outlet connected to two condensers (24°C). To ensure the chemical results, the reactor was connected in series to condensers 1 and 2. The results obtained in condenser 1 were 3 mL of 2-phase liquid with a strong odor. The upper fluid is yellow, and the lower fluid is brown. In condenser 2 (connected directly to condenser 1), 4 mL of yellow single-phase liquid was produced with a strong odor. The liquid obtained from the pyrolysis process was then analyzed by FTIR and GC-MS. FTIR results on both samples showed that the samples contained the functional groups O-H, CH ₂ , and C=O. The GC-MS results on condenser sample 2 showed that the pyrolysis product contained acetone compounds. This confirmed that pyrolysis caused chemical structural changes in LDPE
Keywords:	chain bond-breaking reactions, producing many smaller chemical compounds.
Fuel; LLDPE; Plastic waste; Renewable energy	Additionally, some oxidation happens, which is due to the oxygen content in the reactor. This study provides new insights into the LLDPE pyrolysis mechanism and the physical and chemical properties of the liquid resulting from the pyrolysis process.

1. Introduction

Linear Low-Density Polyethylene (LLDPE) is a type of polyethylene plastic with a linear molecular structure and low-density level. LLDPE is a type of synthetic plastic that is non-biodegradable or cannot be degraded by microorganisms. Thus, it causes environmental problems [1]. LLDPE plastic

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production in Indonesia has experienced quite significant development, driven by increasing demand from various industrial sectors. LLDPE is usually used in food containers, plastic wraps, and food packaging in supermarkets. However, waste from LLDPE is still directly dumped, causing issues in the environment. Specific data regarding the amount of LLDPE waste produced in Indonesia varies, but generally LLDPE plastic contributes around 17-20% of the total plastic waste produced [2].

Currently, various methods can be used to proceed with plastic waste. Some people proposed the use of alternative plastic, such as bioplastic [3-5]. Other researchers give ideas for re-using, recycling plastic [6], or even give regulation [7]. Some methods for solving plastic issues are mechanical shredding methods for transforming plastic into small pieces to make it easy for further processes [8-9]. Others are the pyrolysis methods for decomposing plastic into simple fractions with high heating [10-11], and processing methods using chemical reactions to modify the structure of plastic and change it into a valuable product [12].

Research on how to solve LLDPE issues has been well-documented and well-attracted attention for researchers (see Figure 1 from bibliometric analysis in Scopus database), showing increasing number of research time by time. Bibliometric analysis is one of the excellent methods to understand current research trend, as reported elsewhere [13-20].



Fig. 1. Scopus database for research trend for "LLDPE waste" taken on May 2024. Detailed information for the bibliometric analysis is explained elsewhere [21]

Table 1 shows several previous studies regarding LLDPE plastic pyrolysis. Pyrolysis was chosen because it can produce renewable fuels and other valuable products that can be useful for everyday life [22-28]. Based on the above methods, we focused on understanding the pyrolysis method to process LLDPE plastic. This is because there has been no research discussing LLDPE pyrolysis analysis from the perspective of FTIR and GC-MS analysis [29].

Based on our previous studies on polymer utilization [30-31], this research aims to determine the compounds produced from the LLDPE plastic pyrolysis process using Fourier Transform Infrared Spectroscopy (FTIR) and Gas Chromatography-Mass Spectroscopy (GC MS). Identification using FTIR is useful for finding out what functional groups are present in each sample and knowing what happens during the combustion process, and identification using GC MS is used to find out what compounds are contained in the resulting sample. In short, we pyrolyzed LLDPE particles. Pyrolysis is a thermal degradation process of waste (such as plastic waste). It requires heat without oxygen, which can transform organic materials into charcoal, gas, and oil. The plastic pyrolysis process

produces gases in addition to solids and oil. The gas produced in the pyrolysis process can be burned as fuel or simply left alone [32]. The present pyrolysis was carried out without oxygen, in which we proceeded with 985 g of LLDPE plastic ore with a diameter of 2 mm at a temperature of between 192-194°C. The plastic pyrolysis process has three stages of decomposition. The first stage was the random cutting of the polymer chain into shorter ones. Then, the second stage was where cuts occur at the end of the molecular chain. And the final stage was the separation of the polymer chains which results in the formation of smaller molecules. The results from the reactor are connected in series with condensers 1 and 2. After the sample was pyrolyzed, we analyzed using FTIR and GC-MS. The research results provide new insights into the pyrolysis mechanism of LLDPE and the physical and chemical properties of the resulting fuel.

Table 1

No	Title	Author(s)	Note	Ref.
1.	Pyrolysis Characterization of a	Capote et	This paper contained the pyrolysis characterization of	[33]
	Linear Low-Density Polyethylene	al.	LLDPE to measure material and reaction parameters.	
2.	Process Simulation Modeling of	Hafeez <i>et</i>	This paper described the pyrolysis process in an FBR	[34]
	the Linear Low-Density	al.	reactor. Further studies showed that the hydrocarbon	
	Polyethylene Catalytic Pyrolysis in		(HC) range of diesel fuel (C10-C19) was between 40	
	a Fluidized Bed Reactor		and 63% in the pyrolysis oil yield obtained	
3.	Identification of Pyrolysis	Seungdo	This paper described how to estimate Arrhenius	[35]
	Reaction Model of Linear Low-	dan	parameters as well as LLDPE pyrolysis reaction	
	Density Polyethylene (LLDPE)	Kavitha	models from isothermal kinetic data.	
4.	Study of the fuel properties of	Al-Salem <i>et</i>	This research focused on the use of low linear and	[36]
	extracted oils obtained from low	al.	low-density virgin-grade polyethylene for fuel	
	and linear low density		production pyrolysis routes. The oil in this study had	
	polyethylene pyrolysis		a low aromatic content with the flash point measured	
			for the oil sample (80°C) following market standards	
			for diesel fuel. However, its pour point was slightly	
			higher (10°C) so it is still suitable for use as diesel fuel.	
5.	Comparative study of continuous-	Fan <i>et al</i> .	This study compared continuous and batch-stirred	[37]
	stirred and batch microwave		microwave pyrolysis. Hydrocarbons from continuous	
	pyrolysis of linear low-density		pyrolysis showed a narrower distribution of carbon	
	polyethylene in the		atoms. Up to 98.0% of gasoline-grade hydrocarbons	
	presence/absence of HZSM-5		were obtained from the continuous system.	

The novelty of this research was to understand optimum the pyrolysis process without using a catalyst and the phenomena happening during the process. We used a set of tools made simply from materials available in the commercial market, informing that the process can be done homemade. This research can bring ideas to get better results and become an alternative way for processing plastic waste.

2. Methodology

The tools used in this research were a gas stove, LPG gas (combustion source), thermocouple, 8mm aluminum pipe, 12-mm aluminum pipe, can square reactor (length x width x height = 35 cm x 24 cm x 44 cm), 11-cm diameter round can, 14-cm diameter water basin, vials, glue gun, plasticine, Shimadzu 8400 FTIR instrument, and GC-MS instrument. The material used in this research was 985 g of LLDPE plastic ore.

Figure 2(a) shows the Process Flow Diagram for the LLDPE plastic pyrolysis process which begins by inserting LLDPE plastic ore into the reactor. Then the stove that uses LPG fuel is turned on to heat

the reactor. Burning LLDPE plastic ore produces gas and residue in the form of wax which is at the bottom of the reactor. Gas from burning LLDPE will flow to two condensers connected in series, with condenser 1 in front of condenser 2. In the condenser, the gas will be condensed into liquid oil. The resulting oil liquid will be stored in a container.

Figure 2(b) shows the flowchart of the pyrolysis process. We placed 985 g of LLDPE plastic particles (pellets) into the reactor and closed it to make sure closed system. Next, we carried out the pyrolysis process with a total time of 2 hours 15 minutes. Every 15 minutes, we checked the temperature in the reactor using a thermocouple. We got results in the form of liquid in each condenser. In condenser one, a yellowish liquid with two phases is obtained. The upper phase is dark brown, and the lower phase is light yellow. In condenser two, a liquid with one phase is yellow. Next, FTIR and GC-MS analysis was carried out for the resulting liquid. Detailed information for the FTIR analysis is explained elsewhere [38-40]. Also, for GC MS is explained elsewhere [41-43].



3. Results

3.1 Physical Condition of the Pyrolysis Process

In this study, pure LLDPE plastic pellets were used for the pyrolysis process. The plastic pellets used were 935 g (working volume 80% into the reactor), which were burned in a reactor for 135 minutes. The temperature was checked periodically every 15 minutes. During the combustion process, combustion takes place constantly at a temperature of 190°C and finally decreases to 174.5°C. When the pyrolysis process begins, the long chains of polyethylene will be cut into shorter chains. Plastic pellets, which initially form a white solid, will eventually melt and become liquid as more and more polymer chains are broken. In the first 15 minutes, the temperature in the reactor was still very low, which means that most of the LLDPE plastic was still solid and not much gas was produced. LLDPE plastic melts at a temperature of 105-115°C. Thus, when reaching the 45th minute,

LLDPE plastic pellets will have melted, and more gas will be produced. In the research carried out, the peak of heating occurred at 90 minutes with a temperature of 194.5°C. The temperature in the reactor tends to rise significantly after the plastic starts to melt, which indicates that heating is taking place effectively and quite a lot of gas is being produced. The heating process was only carried out for 135 minutes after the temperature decrease in the reactor. To maximize the gas produced and condensed fuel, heating can be stopped when the temperature in the reactor is constant. After the pyrolysis is stopped, the liquid is obtained in condenser one and condenser two. This liquid is a condensed combustion gas. Because the reactor was opened immediately after the heating was stopped, there was still a lot of combustion gas in the reactor and had not yet condensed. This results in less liquid being obtained.

3.2 Pyrolysis Product Physical Results

Figure 3 shows the changes in LLDPE before and after the pyrolysis process. Figure 3(a) shows LLDPE before the pyrolysis process. LLDPE ore is white granules measuring 2 mm. Figure 3(b) shows the liquid resulting from LLDPE pyrolysis in condenser 1 (sample 1). Sample liquid 1 has two phases because the pyrolysis process produces a complex mixture of various organic compounds that have different properties. The two phases produced consisted of a heavy oil fraction and a light oil fraction. Figure 3(c) shows the liquid resulting from pyrolysis in condenser 2 (sample 2) which is a single-phase liquid. This is due to slower cooling; Thus, the liquid resulting from pyrolysis tends to remain in one phase in this condition. Sample 1 contained two phases that had not separated completely. Thus, there were more solid particles or other phases that can cause turbidity. Sample 2 had gone through a better phase separation process. Thus, the liquid obtained is clearer. Figure 3(d) shows that the pyrolysis process leaves residue in the form of wax on the reactor can. This is because LLDPE contains long carbon chains which tend to form hydrocarbon compounds. During the pyrolysis process, these plastic molecules were broken down into smaller molecules, including molecules that had a wax-like structure.





3.3 FTIR and GC-MS identification

The liquid results obtained in condensers 1 and 2 were then analyzed using an FTIR instrument to determine the functional groups present in each liquid (see Figure 4). GC-MS analysis was also carried

out on the liquid in condenser 2 to determine for sure the volatile compounds produced from pyrolysis carried out using LLDPE plastic (see Figure 5).

Figure 4 shows the results of FTIR identification of samples produced with pure LLDPE (named LLDPE), as well as samples from condensers 1 and 2 (named samples 1 and 2, respectively). Sample 1 had 4 peaks, in which the results of each peak showed several wavelengths whose values were close to the wavelengths of the raw material. This indicated that the sample in condenser 1 was likely to have polyethylene and it had only experienced bond breaking and had not been transformed into other compounds. The pyrolysis process involved breaking chemical bonds in polyethylene plastic molecules and changes in the molecular size distribution due to high heat. In sample 2, 6 peaks were produced. All resulting wavelengths are no longer similar to the raw material wavelengths. This indicates that the sample in condenser 2 experienced a pyrolysis reaction. It occurred and produced other compounds where the results can be confirmed by GC-MS (see Figure 4). Detailed information for the peak comparison is presented in Table 2.

In the FTIR spectrum of raw LLDPE material, a peak was produced at a wavelength of 2915 cm⁻¹, indicating the presence of C-H bonds in the polymer carbon chain which is the region for aliphatic C-H. At a wavelength of 2849 cm⁻¹, LLDPE contains a symmetric CH2 functional group. LLDPE as a carbon polymer also shows a peak in the 1469 cm⁻¹ area which indicates CH2 bonds in the carbon chain. Then, at a wavelength of 729-719 cm⁻¹, it is indicated a split of CH2 rocking deformation [44]. This indicates the existence of certain states in the molecule that cause resonance coupling.

Based on the FTIR spectrum of sample 1, 6 peaks with different wavelengths were obtained. The first peak with a wavelength of 3464.27 cm⁻¹ indicates the presence of an O-H group, namely hydrogen bonds. This is caused by the formation of water in the pyrolysis process. The water formed in the pyrolysis process is caused by the presence of oxygen in the reactor. The second peak with a wavelength of 2924.18 cm⁻¹ indicates the presence of the C-H alkane functional group. The third peak with a wavelength of 1643.41 cm⁻¹ indicates the presence of a conjugated C=O ketone group. The fourth peak with a wavelength of 1458.23 cm⁻¹ indicates the presence of C-C vibrations. The sixth peak with a wavelength of 725.26 cm⁻¹ indicates the presence of C-H bonds. There are C-H bonds and also C-H alkanes which probably come from the structure of polyethylene itself. The results on this condenser show that there are still CH2 groups, indicating a break in the polyethylene polymer chain. There are also ketone groups which indicate that the broken polymer chain has undergone oxidation.

Based on the FTIR spectrum of sample 2, 4 peaks with different wavelengths were obtained. The first peak with a wavelength of 3363.97 cm⁻¹ indicates the presence of the O-H group, namely the hydrogen group. The presence of the O-H group can be caused by the formation of water in the pyrolysis process. The water formed in the pyrolysis process is caused by the presence of oxygen in the reactor. The second peak with a wavelength of 2067.76 cm⁻¹ indicates the presence of the C-H alkane group. The third peak with a wavelength of 1635.69 cm⁻¹ indicates the presence of a conjugated C=O ketone group. The fourth peak shows a wavelength of 663.53 cm⁻¹ which indicates the presence of an OH Out-of-plane bend group. In this spectrum, there are C-H alkane groups originating from polyethylene molecules that experience chain termination. There are also ketone groups which indicate that the broken polymer chain has undergone oxidation.

Figure 4 shows the results of the GC-MS chromatogram of sample 2. Based on the chromatogram, the most dominant compound peak was obtained with a percent area value of 100%. This compound is acetone with a retention time of 1.442 minutes. The acetone compound with the largest area percent is a volatile compound that has a ketone group. Acetone has a low boiling point. Thus, it can be easily evaporated and passed through a gas chromatography column. The high volatility allows ketones to be separated from other components in the sample. Acetone produces a molecular ion

with a value of m/z 58 and other characteristic fragment ions such as m/z 43 (CH₃CO⁺), m/z 15 (CH3), and m/z 14 (CH2). The resulting fragmentation pattern closely matches the typical fragmentation pattern of acetone. Acetone can be formed because of pyrolysis due to the presence of traces of oxygen in the reactor which causes the formation of carbonyl groups (C=O) in the broken polymer chains.



Fig. 4. Combined IR spectrum of LLDPE and pyrolysis samples

Table 2



Fig.	5.	GC-MS	chromatogram	of Sampl	e	2
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No		Wavelength (cm	Functional groups	
	Peak LLDPE	Peak Sample 1	Peak Sample 2	
1.	-	3464.27	3363.97	O-H Hydrogen bonds
2.	2915	2924.18	-	CH2 Asymmetric
3.	-	-	2067. 76	C-H Alkane
4.	-	1643.41	1635. 69	C=O Conjugated ketones
5.	1378	1458.23	-	CH3 Umbrella mode
6.	-	902.72	-	C-C Vibration
7.	729-719	725.26	-	Split CH2 Rocking deformation
8.	-	-	663.53	OH Out-of-plane bend

3.4 Proposal Mechanism for Chemical Reactions in Pyrolysis of LLDPE

Figure 6 shows the reaction mechanism that occurs during the combustion process and the reaction mechanism for cutting acetone fragments. At the high temperatures produced by combustion, the covalent bonds holding together the polyethylene polymer chains can decompose. This causes the breaking of the polymer chains into smaller fragments. Breaking polymer bonds produces free radicals, such as alkyl radicals. These radicals can be reactive and can participate in further reactions. Termination is the final step in a series of free radical reactions that occur, where two active free radicals meet and form a covalent bond, stopping the polymer chain elongation process. Under pyrolysis conditions, smaller fragments of the polymer chain can interact and undergo complex reactions, including hydrogenation and oxidation, ultimately producing acetone as one of the final products.

Figure 5 also shows the mechanism of the polyethylene fragment-cutting reaction. In the analysis performed using GC MS, CH_2 with m/z 14 was detected first which is part of polyethylene and has been cut off from the long polyethylene chain. Next is m/z 15, which represents CH_3 .

In addition, fragmentation occurs in the typical pattern produced. The chemical can initially ionize into the original molecular ion (M), which is $CH_3COCH_3^+$. The original molecular ions can then undergo further fragmentation, one of which becomes CH_3CO ions and CH_3 ions. This fragmentation occurs because the bond between carbon and oxygen in the acetyl group (CH_3CO) is relatively weak. Thus, it is easy to split. In addition, compounds can undergo additional ionization, where the CH_3CO^+ ion can act as a precursor for further fragmentation, resulting in additional fragmentation including CH_3 and CH_2 ions.



Fig. 6. Pyrolysis Reaction Mechanism

4. Conclusions

The research was focused on FTIR and GC MS Analysis of LLDPE pyrolysis results, which was to determine the compounds produced from the LLDPE pyrolysis process. The analysis was also supported by the observation of the physical appearance of the pyrolysis product after condensation. Analysis for the possible chemical reaction was also added. The FTIR results show that the pyrolysis products include alkane, alkene, and ketone functional groups. The GC-MS results show that the pyrolysis product contains acetone. This shows that pyrolysis causes structural changes in LLDPE. This study provides new insights into the LLDPE pyrolysis mechanism and the physical and chemical properties of the liquid resulting from the pyrolysis process.

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