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Research Paper

Catalytic Pyrolysis of Plastic Waste using Red Mud and Limestone: Pyrolytic Oil Production and Ignition Characteristics

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Abstract

This study aims to provide new insights into utilizing local waste materials to enhance the quality of pyrolytic plastic products. This study investigated the catalytic pyrolysis of polypropylene (PP) and low-density polyethylene (LDPE) using 10 wt.% red mud and 10 wt.% limestone catalysts in a batch reactor. The process was conducted at an operating temperature of 350 °C with retention times of 30, 60, and 90 minutes. The effects of adding red mud and limestone catalysts on the yields of liquid, solid, and gas pyrolysis products were analyzed. The pyrolytic oil was further evaluated using droplet evaporation measurements, equipped with a K-type thermocouple and a CCD camera to monitor droplet evolution within an atmospheric chamber. The addition of catalysts enhanced the liquid product yield while reducing the solid yield. The catalytic pyrolysis successfully facilitated the isomerization of plastic polymers, breaking the carbon chains of PP with 10 wt.% red mud. Olefin content increased by up to 7.3% for both 10 wt.% red mud and 10 wt.% limestone. Furthermore, the evaporation rate constant of the catalytic pyrolysis oils improved by up to 8.3%.

Keywords: Catalytic pyrolysis; LDPE; Polypropylene; Red mud; Limestone, Single droplet

1. Introduction

The increasing global population has led to two major problems: the rise in solid waste [1] and the growing energy demand [2]. According to the United Nations Environment Programme, 11.2 billion tons of solid waste are collected worldwide each year. Solid waste contains approximately 13% plastic [3]. Several properties of plastic, such as durability, pressure resistance, chemical inertness, flexibility, versatility, low production cost, and better thermal stability, make plastic the material of choice in various applications. Plastic waste is one type of solid organic polymer waste, rich in carbon and hydrogen, with common types being polyethylene and polypropylene [4], [5]. Compared to traditional plastic waste processing technologies, pyrolysis offers significant advantages [6]–[10]. Pyrolysis is the process of depolymerizing and reforming plastic waste through thermochemical action to produce hydrogen-rich gas [11]. This technology enables the harmless and resource-based treatment of plastic waste, with remarkable advantages in safety, environmental protection, high efficiency, and energy savings [12]. Pyrolysis is a promising thermolysis technique to recover valuable oil and light hydrocarbons (HC) with high yields from solid plastic waste [13]–[15]. Through pyrolysis, long-chain (heavier) hydrocarbon molecules are broken down into smaller (lighter) molecules, producing volatile hydrocarbons and carbon residues that can be condensed into liquid fuel [16], [17]. Pyrolysis can be conducted thermally or catalytically under different experimental conditions, yielding solid char, liquid oil, and non-condensable gas as end products [13]. Parameters such as temperature, reactor type, catalyst, pressure, residence time, and gas flow rate play crucial roles in conducting the pyrolysis reaction of plastic waste [12], [18].

Several studies report that various organic compounds, such as aromatic, alicyclic, and aliphatic hydrocarbons, are found in pyrolytic oil obtained from plastic pyrolysis [19], [20]. The liquid fuel produced by pyrolysis has physical and chemical properties similar to fossil-derived liquid fuels, as plastic itself is made from naphtha, a byproduct of fossil fuel or natural gas refining [21]. Catalytic pyrolysis tends to favor the formation of diesel (C_{12} – C_{24}) [22]. The pyrolysis temperature plays a significant role in determining the type of fuel produced; higher temperatures increase the carbon atom content in the pyrolysis products [23]–[25]. Liquid fuel from pyrolysis is expected to serve as an alternative to conventional fuels, which are becoming increasingly scarce and difficult to obtain [26]–[28]. In recent years, catalytic pyrolysis of plastic to produce hydrogen-rich gas has garnered attention from researchers [11], [29]. Catalysts play an important role in reforming plastic to produce hydrogen by helping to break long chains into shorter ones and breaking chemical bonds to promote the formation of small-molecule gas products [30], [31]. Therefore, efficient and stable catalysts are essential, though there are significant challenges associated with the catalysts themselves. Pyrolytic oil produced from the pyrolysis process generally has high viscosity and acidity, making it unsuitable for direct use as fuel [23], [32], [33]. Catalysts are needed to improve the quality of the pyrolytic oil. One useful catalyst for cracking heavy (long-chain) compounds into lighter oxygenates (short-chain) and reducing acidic compounds in pyrolytic oil is calcium oxide (CaO) [34], [35].

This study investigated the catalytic pyrolysis of PP and LDPE using natural minerals such as red mud and limestone to analyse the characteristics of pyrolysis by-products. Red mud and limestone have a stable metal oxide content at high temperatures so they can accelerate the reaction rate, thereby increasing the production of heavy oil. Additionally, the Ignition properties of pyrolytic oil were evaluated and compared between neat plastic and plastic with the addition of natural mineral catalysts. The primary objective of this study was to develop an innovative approach for enhancing the quality of pyrolytic oil, increasing liquid fuel production, converting solid wax into activated char, and providing a comprehensive analysis of Ignition characteristics. These characteristics included droplet evolution, evaporation rate, droplet lifetime, and ignition delay time, with and without the addition of natural mineral catalysts.

2. Material and Methods

2.1. *Production of Plastic Pyrolytic Oil*

Waste Low-Density Polyethylene (LDPE) and Polypropylene (PP) were sourced from a local waste collection center, while red mud and limestone, used as catalysts, were obtained from local resources. PP can be produced as food packaging containers which are microwaveable as PP has a high melting point (~160–180 °C) and heat deflection point. On the other hand, LDPE is available as plastic bags and packaging films; however, recycled LDPE has yet to find suitable applications due to its low mechanical properties [36]. Waste commodity plastics, LDPE have a high hydrogen to carbon ratio and molecular chain structures suitable for liquefaction [37]. The plastic waste collected for pyrolysis was first prepared by cutting it into small pieces approximately 1-1.5 cm^2 in size. These uniformly sized plastic pieces were thoroughly washed with water to remove dust or foreign particles and then oven-dried to remove any residual moisture. Once prepared, 500 grams of plastic waste was loaded into a fixed-bed reactor. The pyrolysis process was conducted at a constant temperature of 350°C, monitored and controlled using a digital temperature controller. This is based on research from Ilmi, et.al. the study compared engine performance and emissions using mixtures of plastic pyrolysis oil (PPO), biodiesel from waste cooking oil (WCO), and diesel, with temperature variations between 350 °C and 550 °C [38]. The retention time within the reactor was varied at 30, 60, and 90 minutes to evaluate its effect on pyrolytic oil yield. During the process, the plastic vapors generated in the reactor were directed to a double-walled condenser, this is based on research from Goffer, et al on extracting the fuel phase from pyrolysis oil generated via the lesser-explored Azadirachta indica biomass. Dichloromethane and n-hexane were the solvents employed at four different volume percentages (10%, 20%, 30% and 40%) as solvents relative to raw bio-oil [39]. Cold water at approximately 10 °C circulated through the condenser to condense the vapors into liquid form. The condensed liquid, i.e., the pyrolytic oil, was collected in a chemical storage container. The catalytic pyrolysis was performed by adding 10wt.% of red mud in the PP and 10wt.% of limestone in the LDPE, this is based on the recommendation from research Zhaoying Li, et al. The study revealed that with the incorporation of 10 wt% Ni/ZSM-5 catalyst, the relative content of H_2 from the catalytic pyrolysis of waste tires can be significantly improved by about 41.3 % [40].

2.2. *Catalyst Characterization*

Scanning Electron Microscopy (SEM) combined with Energy Dispersive X-ray Spectroscopy (EDX) was employed to characterize the catalysts used in this study. This technique offers valuable insights into both the surface morphology and elemental composition of the catalysts, which are essential for understanding their catalytic properties and performance in pyrolysis reactions. To complement the morphological analysis, EDX was used to identify and quantify the elemental composition of the catalyst materials. As the electron beam interacts with the sample, it generates characteristic X-rays that correspond to the elements present in the material. By analyzing these X-rays, EDX provides a detailed elemental map of the catalyst, helping to confirm the presence of key elements like iron, aluminum, calcium, and other components that contribute to the catalytic performance. This was done in previous research by testing nanocarbon catalysts [41].This combined SEM-EDX approach ensures a comprehensive understanding of both the physical and chemical properties of the catalysts, which is crucial for optimizing their use in pyrolytic processes [42].

2.3. *Droplet Experimental Method*

Figure 1 illustrates the experimental setup used for droplet testing in this study. A droplet, approximately 1 mm in diameter, was suspended at the tip of a thermocouple and injected using a standard insulin syringe (1 mL) with 100 increments, each representing 10 μL. A heater was positioned 3 mm away from the droplet to heat it until evaporation and Ignition occurred. The fixed distance between the droplet and the heater ensured sufficient heat transfer to exceed the fuel's flashpoint. A K-type thermocouple with a 0.1 mm diameter was employed, and the heater consisted of an electric coil measuring 40 mm in length and having a resistance of 1.02 Ohms. The experimental apparatus was previously detailed in an earlier study [41], [43]–[45]. A data logger (Advantech, USB-4178) was used to record the droplet's temperature at a sampling rate of 1 kHz. Droplet and flame behavior were captured using a Nikon D3300 camera operating at 60 frames per second. The entire setup was enclosed in an acrylic chamber to minimize airflow interference. Before each new measurement, the thermocouple was thoroughly cleaned to remove any particles from its surface, and the chamber temperature was maintained at room temperature. Videos of the droplets were processed to generate still images using Image J software.

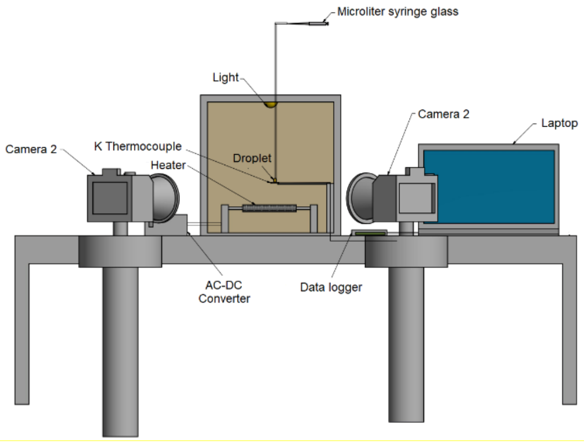


Figure 1. Droplet evaporation set up

2.4. *Pyrolytic Oil Characterization*

The chemical composition of the pyrolytic fuels was analyzed using a GC-MS system (GCMS-QP2010SE, Shimadzu, Kyoto, Japan). This system was equipped with an HT-5 column measuring 30 mm in length, 0.25 mm in diameter, and 0.1 μm in thickness. Helium, with a high purity of 99.99%, served as the carrier gas at a total flow rate of 1.69 mL/min. For each analysis, a 1 μL sample was injected into the system with a split ratio of 100:1. To improve the identification of complex compounds, the temperature of the chromatographic column was programmed to increase in steps: (i) held at 50 °C for 1 minute, (ii) raised to 120°C at 2 °C/min and held for 2 minutes,

(iii) increased to 200 °C at 5°C/min and held for 1 minute, and (iv) further heated from 200 °C to 285 °C at 8 °C/min for 10 minutes. The composition percentages were determined using the peak normalization and similarity method, and the peaks were identified by comparing the mass spectra with those in the GC-MS library.

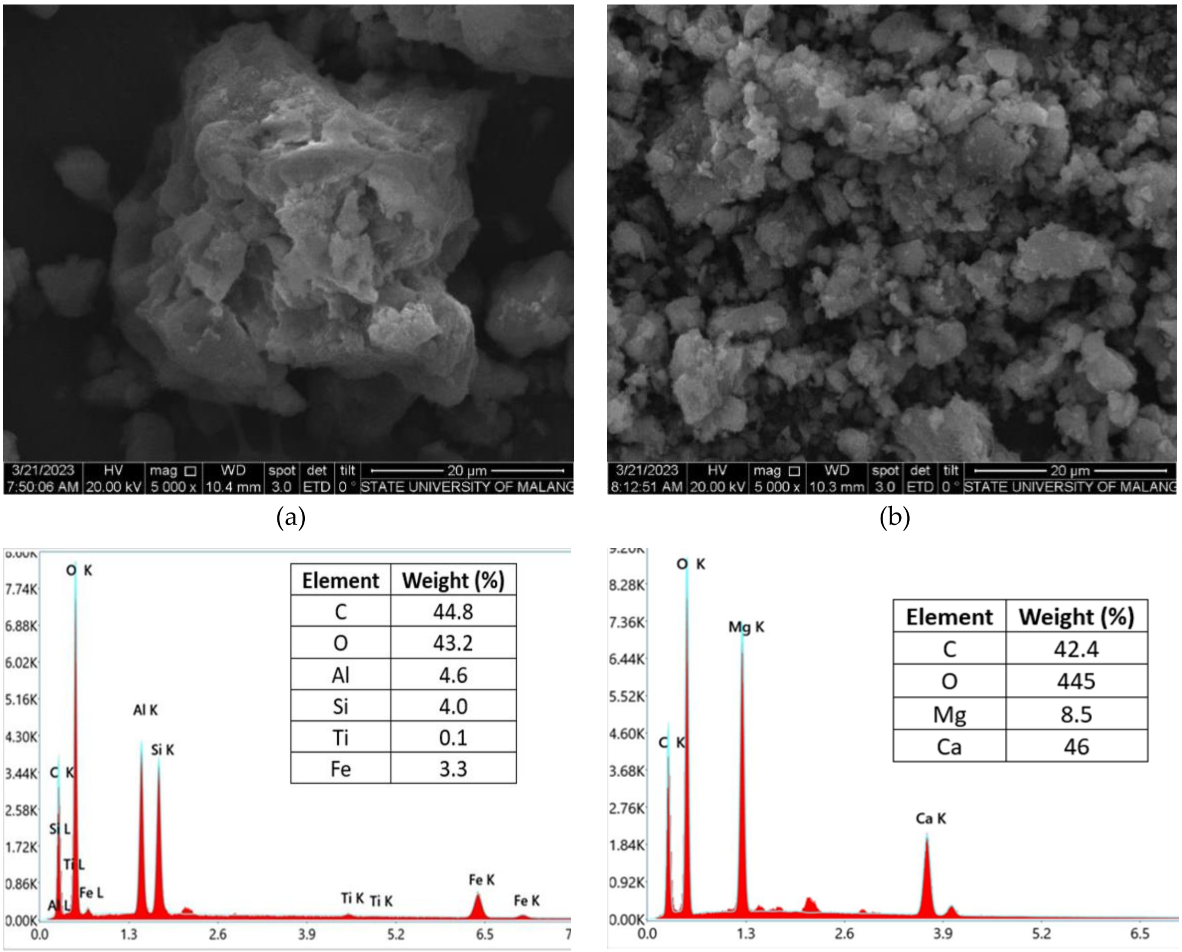
3. Results and Discussion

3.1. *Catalyst Characterization*

Figure 2 shows the SEM-EDX mapping of red mud and limestone catalysts, highlighting their distinct morphological and physical properties at 5,000 magnifications. Additionally, the mineral compositions of the two catalysts are notably different. Red mud contains higher concentrations of metals, including alumina, silica, titanium, and iron, which may contribute to its catalytic performance in pyrolytic oil production [46]. In contrast, limestone contains alkali metals such as magnesium and calcium. Metal oxide catalysts have been reported to enhance co-pyrolysis, potentially leading to higher liquid production, as noted by previous study [47], [48] These two distinct natural catalysts have attracted attention for their impact on the pyrolysis of plastic waste, particularly LDPE and PP (effect of catalyst on product yield, effect of catalyst on chemical compositions, and effect catalyst on Ignition characteristics).

3.2. *Effect of Catalyst on Product Yiels*

Figure 3a shows the liquid yield from plastic pyrolysis with and without natural catalysts at reaction times of 30, 60, and 90 minutes. Overall, the liquid yield increased with longer reaction times, consistent with previous study [49]. LDPE and PP exhibited different liquid yields. LDPE had a lower liquid conversion across all time points, up to 50% less than PP. When 10% red mud was added to PP, liquid production increased by



(a) (b)

Element	Weight (%)
C	44.8
O	43.2
Al	4.6
Si	4.0
Ti	0.1
Fe	3.3

Element	Weight (%)
C	42.4
O	445
Mg	8.5
Ca	46

(c) (d)

Figure 2. (a) SEM images of red mud catalyst, (b) limestone catalyst, (c) EDX of red mud catalyst, and (d) EDX of limestone catalyst

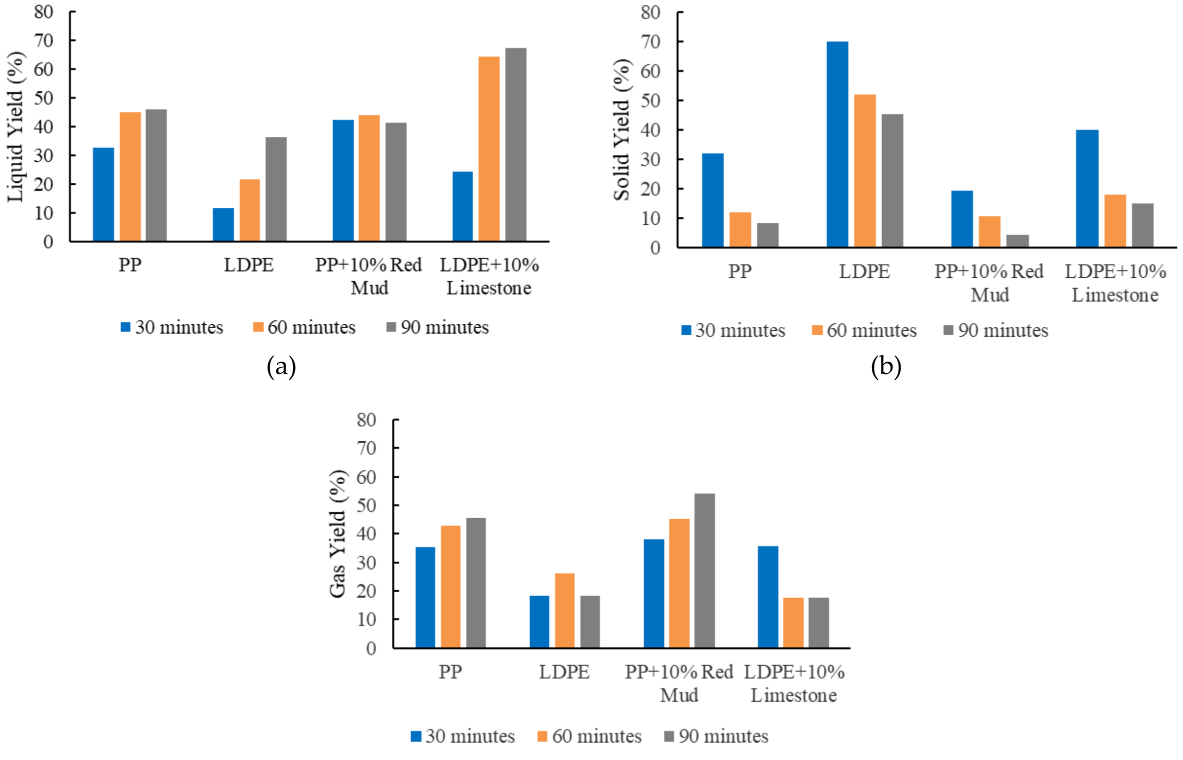
23% at the 30-minute mark, though the yields remained similar at later time points. A substantial increase in liquid yield was observed when 10% limestone was added to LDPE. At the 30-minute reaction time, the liquid yield was double that of neat LDPE. A similar trend was observed at 60 and 90 minutes. **Figure 3b** shows the solid product from plastic pyrolysis with and without natural catalysts at the same reaction times. Overall, the solid yield decreased with longer reaction times, as the raw materials were more fully decomposed, and pyrolysis gases condensed into liquid [50]. As seen in **Figure 3b**, the trends for liquid and solid yields are inversely related. Neat LDPE had the highest solid conversion rate, while PP with 10% red mud produced the least solid yield. Adding 10% limestone to LDPE reduced the solid yield but significantly increased the liquid yield. **Table 1** summarizes the solid product yields for all variants. For LDPE, the solid product was wax, but with the addition of 10% limestone, it became char. All other tested variants produced char as the solid product. Notably, the presence of char indicates better conversion compared to wax, as wax is a long-chain polymer. The conversion of LDPE with 10% limestone to char suggests more effective decomposition, with the char potentially usable as solid fuel. **Figure 3c** shows the gas yield from plastic pyrolysis with and without natural catalysts. Overall, the addition of 10% natural catalysts increased gas production for both LDPE and PP. This is because the catalytic oxidation effectively decomposed the long polymer chains of the plastic, leading to increased gas production [51], [52].

Table 1. Solid product of plastic pyrolytic oils

Components	Solid product
PP	Char
PP+10%Red Mud	Char
LDPE	Wax
LDPE+10%Limestone	Char

3.3. *Effects of Catalyst on Chemical Compositions*

Figure 4 presents the GC-MS data of pyrolytic oil produced from neat PP and PP mixed with 10% red mud. Isomerization occurred when 10% red mud was added to PP, causing the carbon chains to break into smaller fragments. At a retention time of around 18 minutes, neat PP produced $C_{14}H_{28}$, while PP with 10% red mud led to the breakdown of the alkane into $C_{11}H_{22}$. This chain



(a)

(b)

(c)

Figure 3. Product distribution of catalytic pyrolysis: (a) Liquid yield, (b) Solid yield, and (c) Gas yield

breaking during catalytic pyrolysis is attributed to catalytic oxidation during the decomposition process, which fragments the long carbon chains into shorter ones. Red mud contains aluminum (Al) and silicon (Si), which can bond with oxygen and carbon, thereby accelerating the decomposition of the polymer and the formation of liquid oil. Additionally, at a retention time of 30 minutes, PP with 10% red mud broke the hydrocarbon alkane $C_{16}H_{34}$ into $C_{13}H_{26}$. Smaller carbon chains in the liquid fuel make it more like petroleum products. Therefore, catalytic pyrolysis of PP with red mud is a promising method for producing liquid fuels with chemical compositions closer to those of petroleum products [53]. **Figure 5** presents the GC-MS data of pyrolytic oil derived from LDPE and LDPE with 10% limestone. The hydrocarbon chains of LDPE with 10% limestone were reported to be similar to those of neat LDPE.

Figure 6 shows the chemical composition of the pyrolytic oil. The olefin percentage in PP with 10% red mud increased by 7.25% compared to neat PP, while both alkanes and aromatics showed smaller percentages in the modified fuel. This result confirms that adding red mud to PP during thermal decomposition enhances isomerization, resulting in a higher olefin content [54]. Similarly, adding 10% limestone to LDPE increased the olefin percentage by approximately 6.73%, which is slightly lower than the increase observed in PP with 10% red mud. Although the GC-MS profiles of neat LDPE and LDPE with 10% limestone were similar, the higher olefin percentage indicates that isomerization also occurred in the presence of limestone, albeit to a lesser extent than with red mud in PP. Notably, the alkane concentration in LDPE with 10% limestone decreased slightly by up to 4%, and the aromatics concentration was reported as negligible.

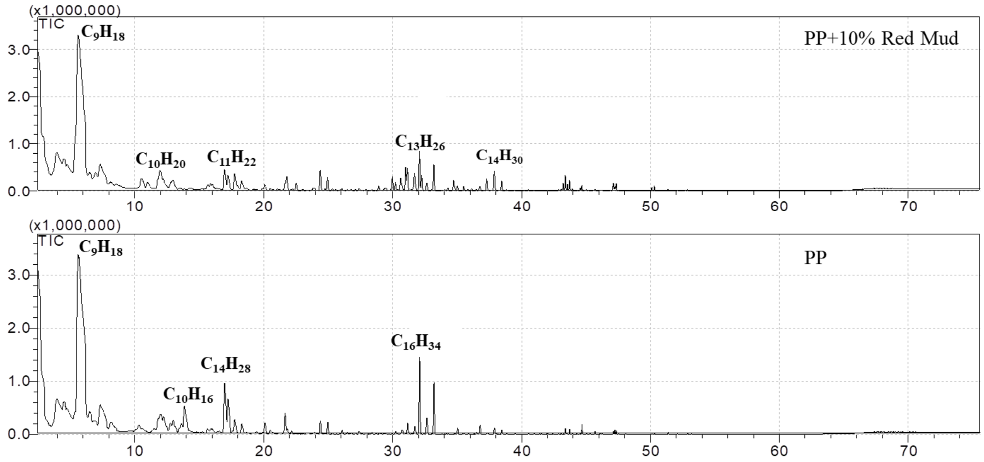


Figure 4. GC-MS image of PP and PP+10% red mud

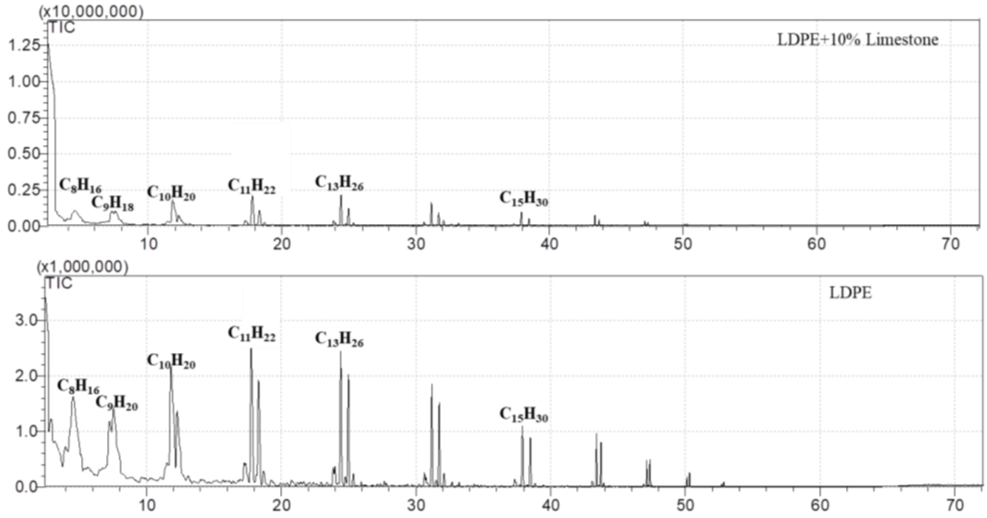


Figure 5. GC-MS image of PP and PP+10% limestone

Figure 7 illustrates the carbon range identified in the tested fuels. This carbon range is critical for determining whether the pyrolytic fuel is closer to gasoline or diesel [55]. Neat PP and LDPE were predominantly composed of carbon chains in the C_7-C_{10} range, accounting for more than 50%, which confirms their similarity to gasoline products. Interestingly, the use of natural mineral catalysts increased the proportion of the C_{11}-C_{12} carbon range in both PP and LDPE. Specifically, PP with 10% red mud showed a 10.7% increase in the C_{11}-C_{12} range, while LDPE with 10% limestone demonstrated an 8% increase.

3.4. *Effect of Catalyst on Ignition Characteristics*

Figure 8 shows the droplet evolution of pyrolytic oil derived from neat PP and LDPE (a), LDPE with 10% limestone (b), and PP with 10% red mud (c). The droplet evolution characteristics of all the pyrolytic fuels were reported to be similar, indicating that catalytic pyrolysis does not reduce the evaporation quality of the plastic-derived pyrolytic oils. **Figure 9** presents the evaporation rate (a), droplet lifetime (b), and ignition delay time (c) of the pyrolytic oils. The addition of 10% red mud to PP increased the evaporation rate by up to 8.37%, while the addition of 10% limestone to LDPE increased the evaporation rate significantly, by as much as 21.27%. This improvement is attributed to the successful isomerization facilitated by the catalysts, which break the lengthy polymer chains and accelerate the evaporation rates of the fuels [56], [57]. A slightly longer droplet lifetime was observed for PP with 10% red mud compared to neat PP, with an increase of 6.5%. Conversely, the addition of 10% limestone to LDPE significantly reduced the droplet lifetime by up to 7%. In terms of ignition delay properties, the addition of 10% red mud shortened the ignition delay by up to 2.8%, while the addition of 10% limestone slightly prolonged the ignition delay. These findings demonstrate that the isomerization process facilitated by natural mineral catalysts improves the Ignition quality of plastic-derived pyrolytic oils.

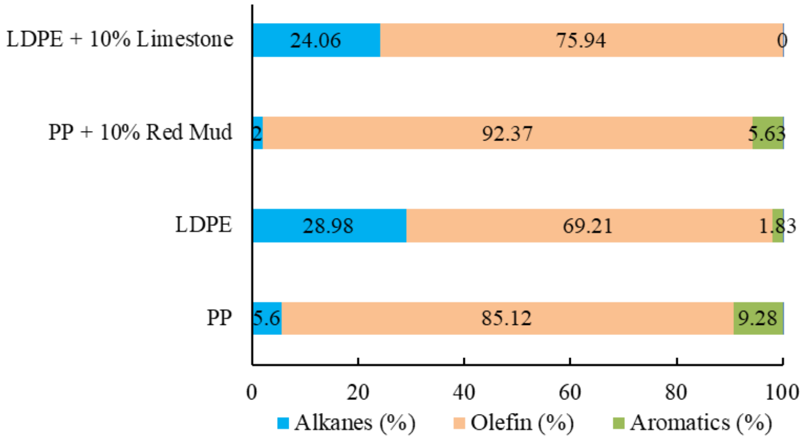


Figure 6. Chemical composition of the pyrolytic oil

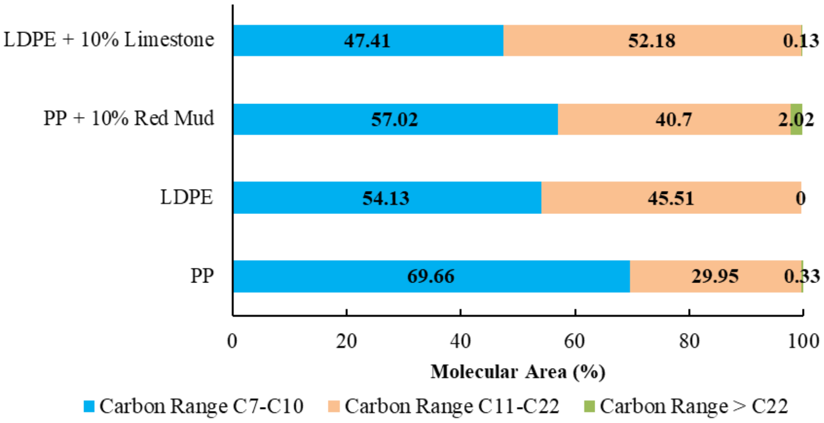
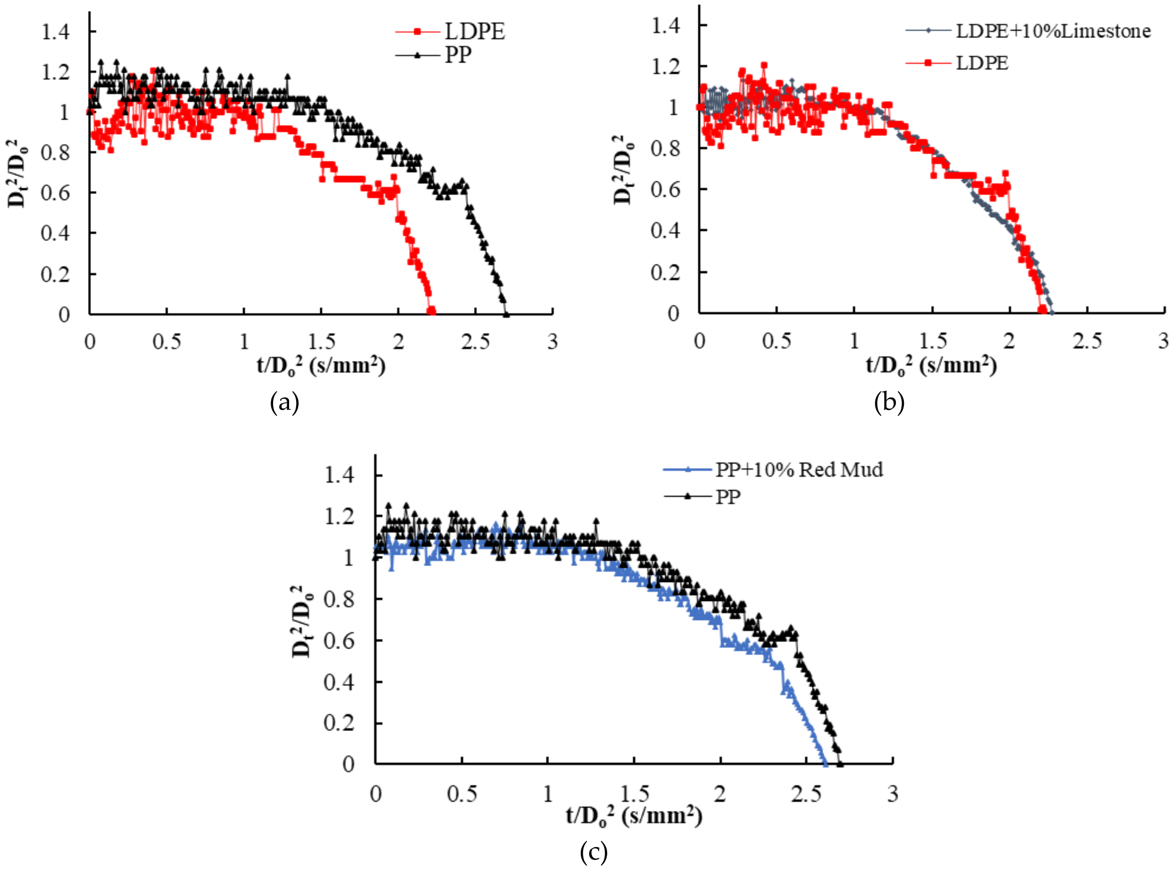


Figure 7. Carbon distribution of pyrolytic oil with and without catalyst

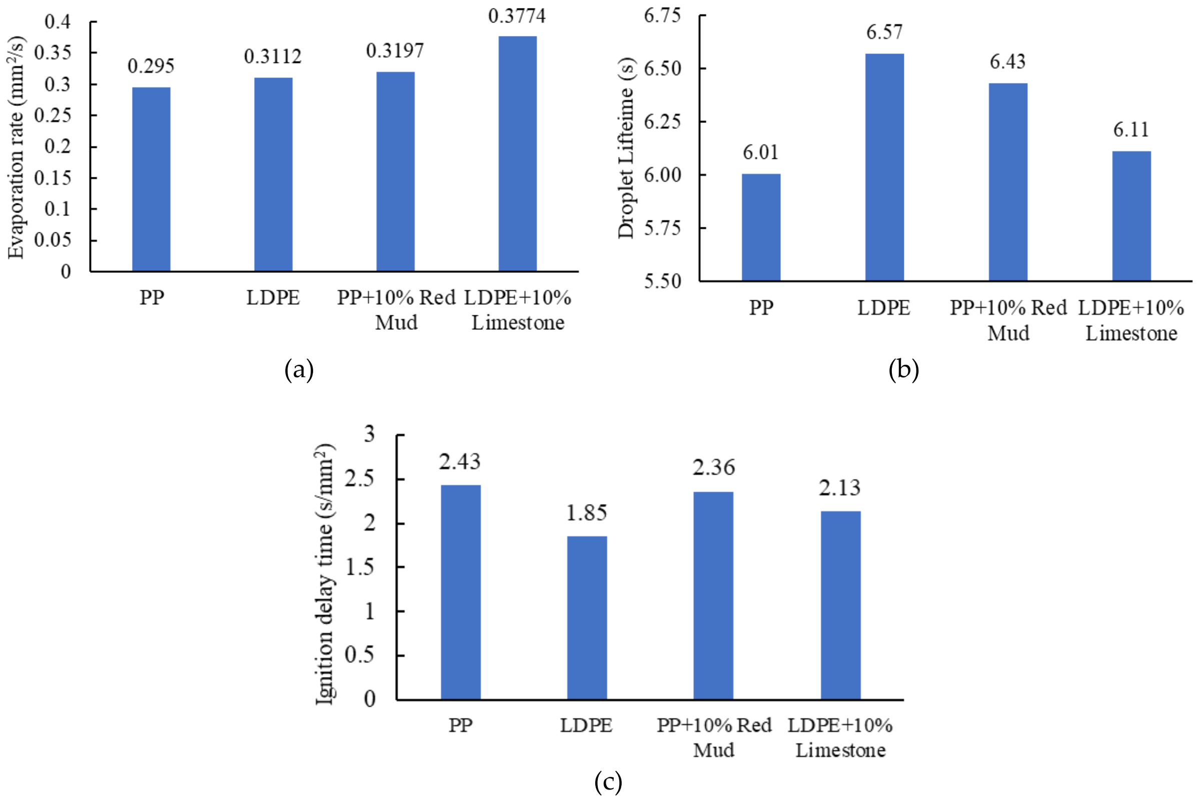


(a)

(b)

(c)

Figure 8. Droplet evolution of pyrolytic oil with and without catalyst



(a)

(b)

(c)

Figure 9. Evaporation (a), droplet lifetime (b), and ignition delay time (c) of pyrolytic oils with and without catalyst

4. Conclusion

This study examined the catalytic pyrolysis of PP and LDPE using natural mineral catalysts, specifically red mud and limestone, focusing on pyrolytic oil production and Ignition properties. The catalytic properties of red mud stem from its metal oxides, such as alumina and silica, while limestone contains magnesium and calcium. The quantity of pyrolytic oil increased with longer reaction times, whereas the solid product yield decreased. The addition of natural catalysts significantly reduced the solid yield while increasing the liquid yield. For instance, 10% red mud promoted the breakdown of carbon chains into smaller fragments, resulting in a 7.2% increase in olefin compounds compared to neat PP. Similarly, the addition of 10% limestone to LDPE enhanced the olefin content by up to 6.73%. The droplet evolution profiles of catalytic pyrolysis fuels were found to be similar to those without catalysts. However, the addition of 10% red mud to PP increased the evaporation rate by up to 8.37% compared to neat PP, while 10% limestone in LDPE significantly improved the evaporation rate by up to 21.27%.

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Author's Declaration

Authors' contributions and responsibilities

The authors made substantial contributions to the conception and design of the study. The authors took responsibility for data analysis, interpretation and discussion of results. The authors read and approved the final manuscript.

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Availability of data and materials

All data are available from the authors.

Competing interests

The authors declare no competing interest.

Additional information

No additional information from the authors.

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