

# Pyrolysis of Polyethylene Terephthalate Granules in Presence of Lewis Brønsted Acid Sites Catalysts

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## Research Article

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# Abstract

Among municipal polymer wastes, Polyethylene Terephthalate (PET) is a unique agent because of its widespread usage and broad range of gaseous products in its catalytic cracking process. Pyrolysis of polyethylene terephthalate (PET) in a semi-batch reactor was investigated using a Lewis Brønsted acid sites catalysts. Experiments were performed under isothermal condition to determine reaction kinetic parameters, product compositions, catalyst/PET mass ratio and temperature effect on the conversion. The products of reaction consist of gas, solid, and liquid phase with a maximum liquid product of 6% at 350 °C. The optimal catalyst/PET mass ratio and temperature were determined. Additionally, the reaction order and activation energy for the reaction were detected.

## 1. Introduction

Primary, secondary (or mechanical), and tertiary (or chemical) recycling are the major approaches for plastic recycling. The plastic materials can be reused as second hand products or modified products in primary and secondary recycling methods (Scheirs 1998; Sorum et al. 2001; Öztürk and Güçlü 2004; Nikles and Farahat 2005; 2012). In contrast, in chemical recycling methods, plastic wastes are converted into their constituent monomers or basic hydrocarbon feedstock (Al-Salem et al. 2009; Sinha et al. 2010; Welle 2011). Among the chemical recycling approaches, catalytic cracking pyrolysis of waste plastics is one of the most applicable methods, during which plastics are heated in the absence of oxygen and broken down into gaseous hydrocarbons, gasoline, or diesel, i.e., fuels (Demirbas 2004; Scheirs and Kaminsky 2006; Karayannidis and Achilias 2007; Jankauskaite et al. 2008).

The plastic type alone affects the yield of the pyrolysis process, molecular weight distribution and the quality of liquid products gained in the catalytic degradation process of waste plastics (Kumar and Rao 2003). In our previous studies in clean technologies and environmental policy, we have reported catalytic degradation of single and mixed polymer granules such as polystyrene, high density and low density polyethylene mixtures, which include various types of urban waste plastic disposals (Roozbehani et al. 2014, 2015, 2016; Sakaki et al. 2014; Imani Moqadam et al. 2015). Among all materials discussed so far, Polyethylene Terephthalate (PET) is rather a unique agent. That is due to its widespread usage as plastic bottle as well its low liquid production and wide range of gas products in its catalytic pyrolysis (Ding et al. 1997; Achilias and Karayannidis 2004). So far, catalytic degradation of PET has been extensively studied by other researchers (Aguado and Serrano 1999; Chiu and Cheng 1999; Park et al. 1999, 2019; Sakata et al. 1999; Awaja and Pavel 2005; Saha and Ghoshal 2005; Bartolome et al. 2012; Santos et al. 2018). For instance, the impact of heating rate on non-isothermal degradation and kinetic parameters in an inert atmosphere was studied (Jenekhe et al. 1983; Sorum et al. 2001; D. Spaseska 2010; Niksiar et al. 2015). It was revealed that the activation energy and pre-exponential factor are extremely affected by the rate of heating while the reaction order remains constant (Niksiar et al. 2015). It was also shown while the thermal degradation is affected by the particle size, it is strongly influenced by temperature in a range of 400 °C and 500 °C (Kpere-daibo 2009). Copper (II) chloride is known as one of the most active agents among catalysts used in PET catalytic cracking reactions (Chiu and Cheng 1999; Faravelli et al. 2001). It

reduces the carbonaceous residues and increases the percentage weight loss of PET 3.5 times in comparison to thermal degradation at similar reaction conditions. Taking the weight loss of catalyst in course of reaction into consideration, copper (II) chloride was considered to be an effectual catalyst (Awaja and Pavel 2005; Báez et al. 2013). While catalytic cracking of PET using copper (II) chloride yielded a shortened cracking time, showed a similar temperature influence on the polymer weight loss as compared to thermal degradation process with a optimal catalyst/ PET weight ratio of 0.1 (Villain et al. 1994; Gladden and Mitchell 2011). The maximum of weight loss was reported 90.8% and 80.5 in presence and absence of catalyst, respectively (Chiu and Cheng 1999).

Thermal degradation in a nitrogen atmosphere at 200–700 °C was examined in a tubular furnace under isothermal conditions by Dzieńcioł et al (Dzieńcioł and Trzecznyński 1998). The main volatile substances evolved from PET in the thermal degradation in a nitrogen atmosphere were CO, CO<sub>2</sub>, acetaldehyde, acetophenone, aliphatic hydrocarbons (C<sub>1</sub>–C<sub>4</sub>), and benzene (Dzieńcioł and Trzecznyński 2000). Within the temperature range 200–300 °C, only acetaldehyde and formaldehyde were detected and at higher temperatures, the mixtures of volatile decomposition products of PET became more complex. The concentrations of carbon oxides, aromatic and aliphatic hydrocarbons increases by increasing temperature, where the maximum yields of acetaldehyde and vinyl benzoate were observed at 600 °C (Dzieńcioł and Trzecznyński 2000). In another work, kinetics of PET soft drink's bottles wastes was investigated in a dynamic condition and at various heating rates in nitrogen atmosphere (Villain et al. 1994). It was found that PET pyrolysis results in a 70 to 80% polymer weight loss in the temperature range of 107 to 242 °C. The n<sup>th</sup> order model technique provided a more precise prediction of experimental data than previously applied techniques yielding an activation energy of 322.3 and 338.98 kJ/mole for Coca-Cola and Pepsi samples, and the order of reaction by of 1.72 (Saha and Ghoshal 2005).

In this work the conversion of PET using a Lewis Brønsted acid sites catalyst is studied. Catalytic measurement was conducted to convert PET into lighter compounds followed by a kinetic model determined from the experimental data. Different conversion percentages were assessed in various temperatures on gas, liquid and waste productions. The effect of temperatures during reaction was also assessed. The other aim of this study is to estimate the reaction model of PET thermal catalytic cracking. Since a few detailed studies on the kinetics of PET pyrolysis by thermo-gravimetric (TG) analysis have been, this work present results by employing a kinetic model.

## 2. Material And Method

### 2.1. Catalyst

The catalyst used in the measurements contains Lewis Brønsted acid sites to improve the catalytic activity and product selectivity in the catalytic degradation of PET. This is because the Brønsted acid sites act as proton addition and the Lewis acid sites are in charge of hydride abstraction. Thus, the polymer degradation occurs at lower temperatures compared to non-catalytic pyrolysis. The Lewis (electron acceptor) and Brønsted (proton donor) in the catalyst controls acidity due to its crystalline structure. The

acid sites were generated by Al species in the catalyst consisting of silica and alumina. The ratio of silica to Alumina (Al/Si) in the catalyst powder has also significantly affects the cracking of polymer chains. High acid site density enhances the cracking reaction of hydrocarbons. Note that the activated acidic sites of the catalyst are sensitive to metal impurities in the feed.

The catalytic and isothermal decompositions were carried out for a mixture of PET in bottle grade. General characteristics of bottle grade were acquired for the samples. A maximum DEG content of 2.0 Wt.% was reported. The melting point was around 250° C, the water content of the samples was 0.3 Wt.% with a maximum carboxyl end group level of 32. The amount of acetaldehyde was also determined to be 1 ppm at its highest level.

The catalyst consists of the following components: (a) zeolite (molecular sieve), (b) palatine, (c) active-matrix component, (d) inactive-matrix component, and (e) binder. The last two properties regulate the activity by dilution of the active components and to obtain the desired particle strength and morphology. Some characteristics of the catalyst are reported in Table 1.

Table 1  
The main properties of the catalyst used in the experiments.

Property	Unit	Minimum	Maximum
Al <sub>2</sub> O <sub>3</sub>	wt. %	38.5	48.2
Pt	wt. %	0.001	0.001
total surface area	m <sup>2</sup> /gr	38	42
Attrition Index	wt. %	1.5	5.3

## 2.2. Experimental

The catalytic degradation experiments were carried on the pure PET granules in a Pyrex reactor under isothermal conditions at a temperature range of 330 °C to 430 °C with a step-wise increment of 10 °C and with the catalyst fractions of 10–40% at atmospheric pressure. The scheme of the setup used in the measurements is shown in Fig. 1.

A semi-batch reactor and Thermo-gravimetric analysis (TGA) equipment was used to carry out polymer degradation reaction. TGA is implemented to determine the rate of degradation in catalytic and thermal processes and determine the relationship between samples mass and temperature. The air in the system was purged with an N<sub>2</sub> stream before each startup. The catalytic experiments were conducted in two steps: first, different catalyst fractions were examined. Regardless of the rate of weight loss, the percentage of conversion was determined at each temperature, and second, the optimum catalyst fraction was determined. The product consists of three-phases (solid, liquid, and gas), in which a remarkable portion is in the gas phase. A condenser was used to cool the vapor product formed in the

reactor. The presence of solid in the output caused a blockage in some experiments, especially when the reaction temperature is high. In addition, due to the presence of solids at the outlet of the setup, the quality of the liquid product was low and measurement of the weight loss versus time was difficult. Analysis of the gas product is important due to its component. As the catalytic degradation occurs at atmospheric pressure, collection and sampling of the products in the constant-volume balloon was on some occasions challenging. Therefore, gas collector and different-volume balloons were used to collect the gaseous product for further analyses.

## **2.3. Gas chromatography analysis**

The products derived from the degradation of PET were analyzed to determine the main consisting components. The products were analyzed using a capillary column gas chromatography with flame ionization detection (GC/FID) ((50 m x 25  $\mu$ m ID, 0.5  $\mu$ m film of silica gel film, AGILENT, US 10420012, GC equipped with an HP-5 capillary column). Helium was supplied as carrier gas at the flow rate of 15.8 ml/min. The injector's temperature was set to a temperature and pressure of 250 °C and 10 psi, respectively. The capillary column separated the liquid components based on their volatility/boiling point. A specific mixture containing alkanes was used to calibrate and determine the retention time in the chromatogram in order to be split into intervals according to the boiling points of the normal alkanes of the calibration mixture. Species were quantitatively determined by a standard method by reliable detection of relative retention time of the components in a gas chromatography/ mass spectrometry (GC/MS) device once a component.

## **3. Result And Discussion**

The parameters that influenced the conversion are discussed in this section. First, the effect of catalyst/polymer mass ratio is described. Afterward, the effect of temperature on the catalytic cracking process is discussed (Sect. 3.1.2).

### **3.1. Thermal and catalytic degradation analysis of PET**

The effect of catalyst weight fraction on the conversion was investigated at different temperatures. Catalyst weight fraction range of 10 to 50% was chosen with a stepwise increment of 10%. The conversion rate yielded from the catalyst ratios are given for different temperatures in Table 2. It can be seen that by an increment in catalysis quantity the percentage of weight loss of the reactant decreases. Generally, there is no liquid product in the thermal degradation of PET in the previous studies. However, some differences were observed regarding the product distribution in the current study which is discussed in Sect. 3.3. The effect of catalyst weight fraction on the weight loss of polymer is given in Fig. 2. Generally, the increase in polymer/catalyst ratio leads to a higher yield of gaseous hydrocarbons and the formation of coke.

Table 2  
Effect of temperature and catalyst weight fraction on conversion.

Catalyst weight fraction	Conversion (%)					
	330 °C	350 °C	370 °C	390 °C	410 °C	430 °C
10						
20	20	33	56	67	75	84
30	17	29	53	64	69	82
40	12	25	48	60	66	79
50	10	19	43	58	63	77

Increasing the catalyst percentage, the amount of coke increases, and the color of the solid product changes from yellow to a colorless mode. PET particles are surrounded by the catalyst, this causes a decrease in the amount of solid and liquid products, but the gas production was increased. The best conversion rate was achieved at a temperature of 430 °C with a catalyst/polymer ratio of 1:10.

## 3.2. Temperature

The lowest conversion rate was obtained at catalyst to polymer mass ratio of 40% and at 330 °C (see Fig. 3). At a catalyst/polymer ratio of 1:10, only 20 wt. % of the sample was degraded at 330 °C, but when the temperature raised to 430 °C, the weight loss percentage increased to 84%. The color of the carbonaceous solid residue tends to be darker, as temperature increased in various catalyst mass ratios.

By increasing temperature, the conversion increases at a certain catalyst loading. Thus, the reaction time was determined by the reaction temperature. Maximum PET weight loss occurred at the beginning of the reaction. According to Table 3, the degradation time rapidly decreases at higher temperatures (about 45 min at 430 °C). This fact is illustrated in Fig. 4, where the weight loss is plotted versus total processing time.

Table 3  
Effect of time on conversion rate (catalyst mass fraction of 10%).

Temperature (°C)	330	350	370	390	410	430
Time (min)	193	150	95	85	54	45
Conversion (%)	20	33	56	67	75	84

## 3.3. Products yield

The yield of liquid, gas, and solid products versus temperature in the range of 330–430 °C with step-wise increment of 20 °C are given in Table 4. The main hydrocarbon products of pyrolysis are the alkane gases

such as methane, ethane, and higher concentrations of the alkenes and ethylene. Due to the breakage of the chemical bonds during the pyrolysis, more gaseous products and consequently more aliphatic gases such as CH<sub>4</sub>, C<sub>2</sub>H<sub>6</sub>, and C<sub>2</sub>H<sub>4</sub> is formed as displayed in Fig. 5c. One of the notable findings in this work is the presence of a large volume of ethylene in the products which increases by increasing temperature. The presence of a large volume of ethylene in gas is another interesting aspect regarding the products. Additionally, unlike the other PET analyses in the literature, in which no formation of liquid products is reported, a slight amount of liquid at 330 °C and 350 °C was formed in this study. The maximum liquid in products was reported to be 6% at temperature of 350 °C and a catalyst to polymer ratio of 10%.

In general, the decomposition of PET results in the production of solid and gas products. The solid products include a small amount of fine pale-yellow particles mostly consisting of benzoic acid and terephthalic acid were found at the outlet of the reactor and some carbonaceous residue in the reactor. The solid phase product increases by increasing temperature. The formation of the solid phase is due to the breakage of more molecular bonds in higher temperatures. The main component of this solid product was terephthalic acid (TPA). TPA degrades to benzene, carbon dioxide and benzoic acid at high temperatures. Figure 5b illustrates the increment of solid product in higher temperatures. The main components of the solid product are terephthalic acid and benzoic acid. As shown in Figs. 5d and 5e, the yield of benzoic acid, carbon dioxides, and benzene increase as temperature rises.

Table 4  
Product yield versus temperature.

Temperature °C	Solid	Liquid	Gas
330	7	3	10
350	10	6	16
370	16	2	35
390	20	0	46
410	22	0	53
430	23	0	61

Terephthalate is the primary product of the pyrolysis of polyethylene. By an increase in temperature the ratio of gas to solid increases. At 350 °C, the ratio of gas to solid is 1.42 and at 430 °C is 2.65. The volatile substances formed in the pyrolysis are given in Table 5 specifically.

Table 5  
Volatile products of the catalytic degradation of  
PET.

Gas composition	350 °C	370 °C	390 °C
CO	0	1	4
CO <sub>2</sub>	10.5	13	16
CH <sub>4</sub>	10	10	13
C <sub>2</sub> H <sub>6</sub>	4.5	6	7.5
Benzoic acid	18	17.5	15
C <sub>2</sub> H <sub>4</sub>	32	34	41
Benzene	1	1	2
C <sub>2</sub> H <sub>4</sub> O	2.5	1.5	0.5
Other	21.5	16	1

### 3.4. Kinetic parameters

It is known that the thermal and catalytic decomposition process of PET atmospheric pressure can be represented by the reaction scheme:  $A_1(s) \rightarrow A_2(s \text{ or } l) \rightarrow A_3(g)$ , where  $A_1$  is the solid polymer with the structure and  $A_2$  mainly consists terephthalic acid and benzoic acid. Subsequently, the rapid production of low molecular weight volatile fragments with consequent weight loss is expected. The volatiles ( $A_3$ ) are mostly COOH, CO, CO<sub>2</sub>, C<sub>2</sub>H<sub>4</sub>, and CH<sub>4</sub>. Since few detailed studies on the kinetics of PET pyrolysis by thermo-gravimetric (TG) analysis have been reported throughout the literature, this work presents results of this investigation by employing kinetic model (Sorum et al. 2001; Sánchez-Jiménez et al. 2009; Al-Salem and Lettieri 2010).

Isothermal thermo-gravimetric analysis (TGA) was used to investigate the mass change of a sample as a function of temperature or time because the reaction process can be followed over a wide temperature range. The kinetic of PET decomposition is considered heterogeneous as more than one phase during reaction exists. The rate of reaction of a heterogeneous reaction can be described as follow:

$$\frac{dm}{dt} = k(t) f[m(t)] \quad (1)$$

Where,  $\frac{dm}{dt}$  rate of consumption of the reactant

$k(t)$  = reaction rate constant, assumed to be only a function of temperature,  $f[m(t)]$

According to Eq. (1), for a heterogeneous system  $k$  is usually defined using Arrhenius Law. Arrhenius Law is given as:

$$k = Ae^{(-E/RT)} \quad (2)$$

Where  $A$  is pre-exponential factor,  $E$  is the activation energy ( $\text{kJ mol}^{-1}$ ),  $R$  represents the universal gas constant ( $\text{kJ mol}^{-1} \text{K}^{-1}$ ), and  $T$  stands for temperature (K).

$f[m(t)]$  has various forms depending upon the reaction system. The most commonly presumed form for the reaction is

$$f[m(t)] = (m)^n \quad (3)$$

where  $n$  is the order of the reaction. By substituting equations (2) and (3) in Eq. (1), the general form of the kinetic equation is given as

$$\frac{dm}{dt} = kAe^{(-E/RT)}(m)^n \quad (4)$$

Here, kinetic parameters in two modes were calculated ( $n \neq 1$  and  $n = 1$ ). According to Eq. (4), for each temperature a specific  $n$ ,  $E$ , and  $A$  exists. There are three unknowns parameters, so determination of the order of reaction ( $n$ ), for all temperature is possible. In addition, kinetic parameters change with temperature. For determination of  $E$  and  $A$  should be plotted  $\ln(k)$  versus  $(1/T)$ . Logarithmic model of Eq. (4) is as follows:

$$\ln\left(\frac{dm}{dt}\right) = \ln(A) - \frac{E}{RT} + n \ln(m) \quad (5)$$

The values of the order of reaction for other temperatures (330 °C, 350 °C, 390 °C, and 410 °C) are calculated by  $n^{\text{th}}$  order kinetic model in Table 6.

Table 6  
Kinetics parameters derived for other temperatures.

Temperature (K)	n
603	1.04
623	1.27
643	1.49
663	1.01
683	1.49

### 3.4.1. Kinetic parameters (second mode, $n = 1$ )

we consider the order of the reaction equal to be one. Thus, Eq. (4) can be rewritten as follow when  $n = 1$ :

$$\frac{dm}{dt} = A e^{(-E/RT)} m \quad (6)$$

Manipulating this equation leads to

$$\ln \left( \frac{m}{m_0} \right) = -k(t - t_0) \quad (7)$$

The  $n^{\text{th}}$  order model technique  $n \neq 1$ , yields a higher values of  $E$  and  $\ln(A)$  compared to “first order” (see Table 7). Kinetic parameters change with temperature in different manner, even though one can assume that they are constant.

Table 7  
Kinetics parameters derived from experiments.

State	E (kJ. mol <sup>-1</sup> )	A (min <sup>-1</sup> )
n = 1	323.87	48×10 <sup>21</sup>
n ≠ 1	109	1013141.08

In case of  $n \neq 1$   $n^{\text{th}}$  order model technique, values of  $E$  and  $A$  are 109 (kJ mol<sup>-1</sup>), and 1013141.08 (min<sup>-1</sup>) respectively. In this study, order of reaction changed from 1.01 to 1.49. Also, in “first order” technique  $E$  and  $A$  are reported to be 323.87 (kJ mol<sup>-1</sup>) and 48×10<sup>21</sup> (min<sup>-1</sup>) respectively. Considering  $n^{\text{th}}$  order model technique, it was found that when  $n \neq 1$  is more suitable to describe the catalytic pyrolysis of the PET as no assumption or reaction degree is required unlike the first order technique.

## 4. Conclusion

The results showed the effect of temperature and catalyst/PET on the PET degradation using Lewis Brønsted acid sites catalyst. Increasing temperature reaction increased the conversion rate, where the products of the reaction form gas, liquid, and solid phases in contrary to other polymers that the product of the degradation mostly consist of liquid, less gas and no solid. The efficiency of degradation process decreased with increasing catalyst/PET mass ratio, since at higher catalyst ratios, the PET molecules surrounded by catalyst leading to lower reaction speed and insufficient heat transfer to PET molecules.

The analysis of gas sample at higher temperature showed that CO, CO<sub>2</sub> and aliphatic hydrocarbons increased and terephthalic acid degraded to benzene and benzoic acid.

## Declarations

**Conflicts of interest/Competing interests:** NO

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**Consent for publication/participate:** All authors agree on publishing the results

**Authors' contributions:** All the authors are fully aware of their contribution in this research work. They have read, edited the final version of the manuscript submitted to CTEP.

**Code availability:** NO

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## Figures

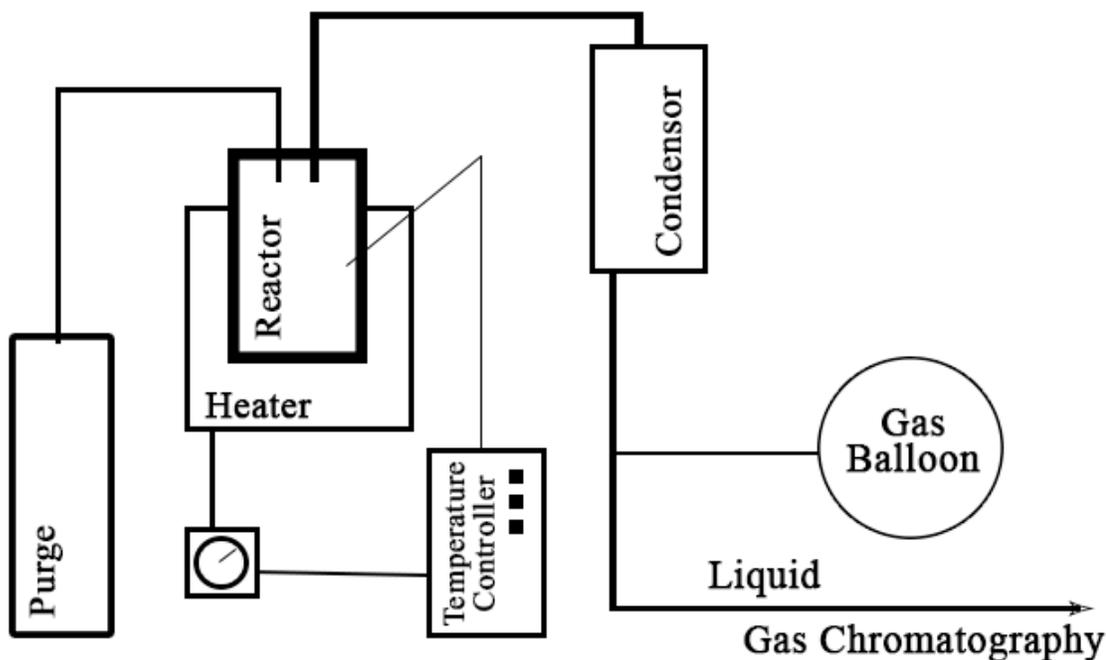


Figure 1

Scheme of apparatus used for the thermal and catalytic degradation studies of materials in a nitrogen atmosphere.

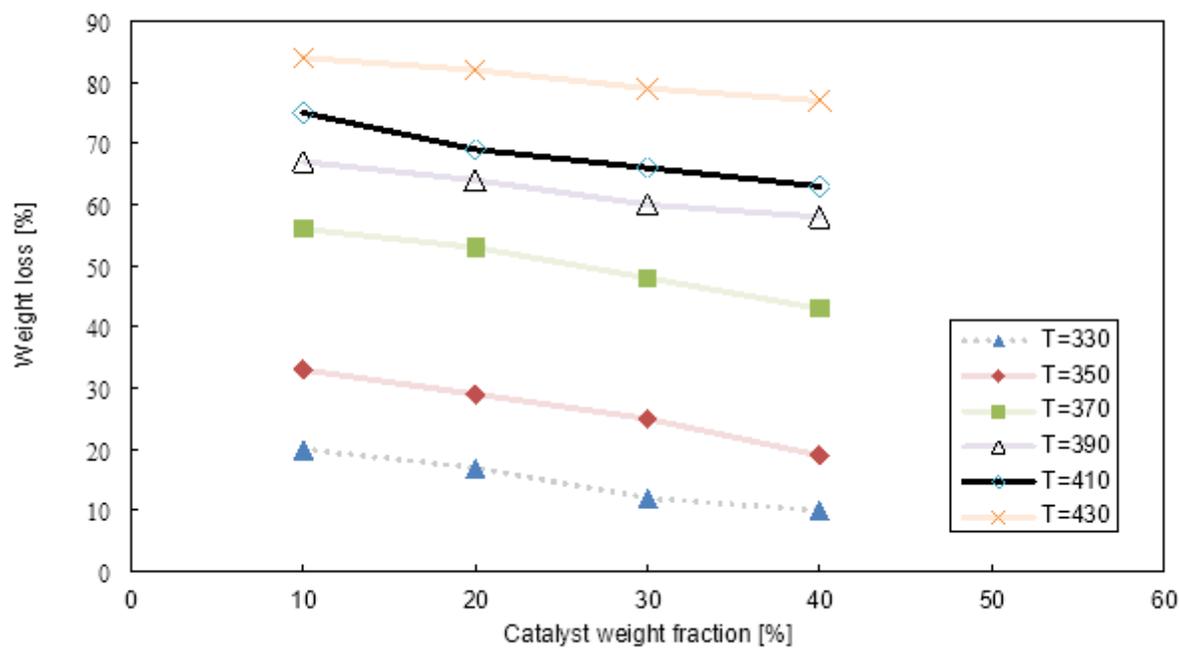


Figure 2

The catalyst weight fraction effect on the conversion.

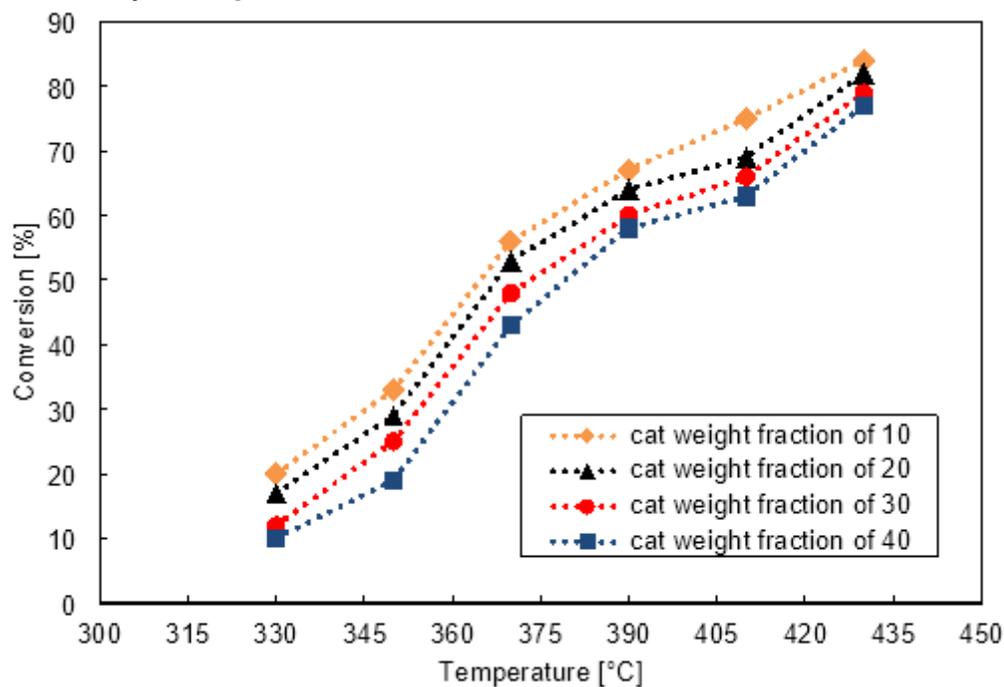


Figure 3

Effect of temperature on conversion rate in different catalyst weight fractions.

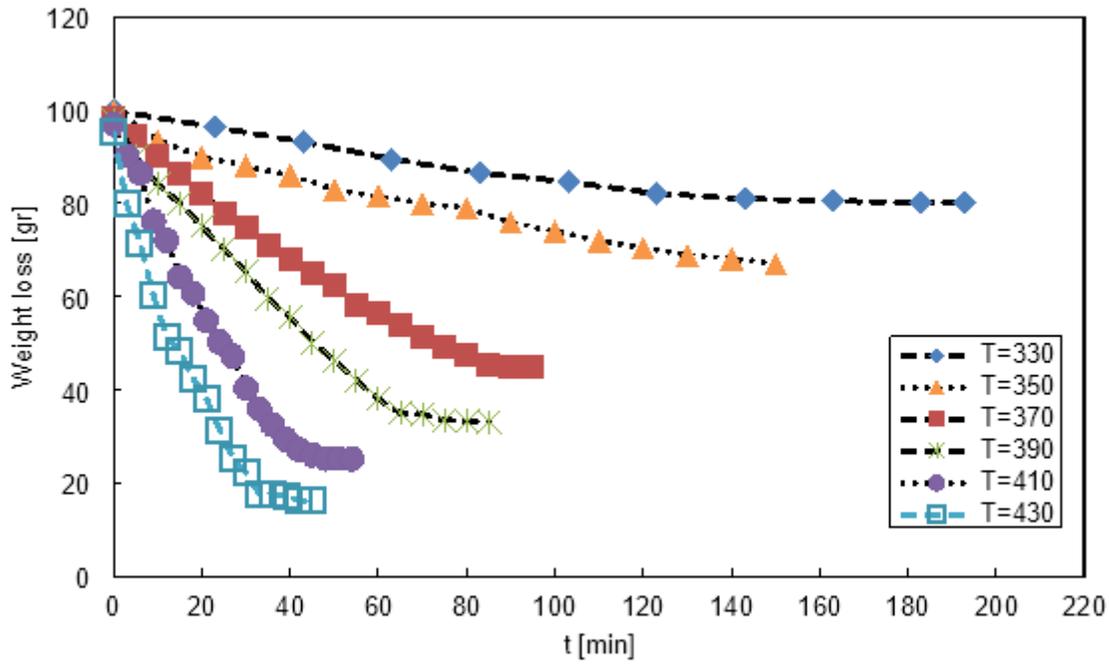
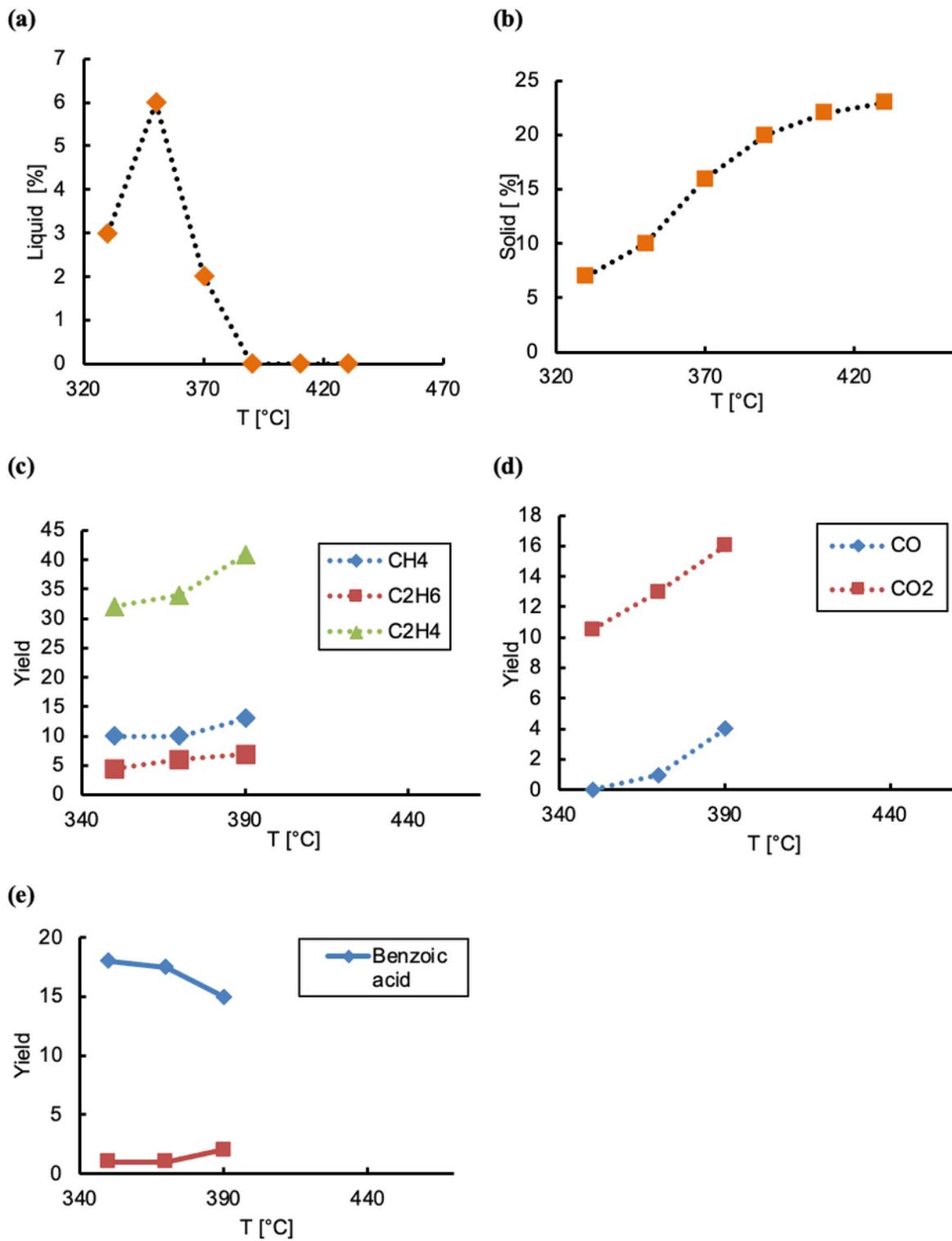


Figure 4

Weight loss of PET at different temperatures.



**Figure 5**

(a) Yield of liquid product, (b) solid product, (c) aliphatic gases, (d) aromatic gases, and (e) carbon oxides at various temperatures.

## Supplementary Files

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