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Catalytic pyrolysis of waste polyethylene terephthalate granules using a Lewis-Brønsted acid sites catalyst

Aref Shahi[1] · Behrooz Roozbehani[1] · Mojtaba Mirdrikvand[2]

Abstract

Among municipal polymer wastes, polyethylene terephthalate (PET) is unique due to its widespread use in the plastics industry. The wide range of gaseous products produced during the catalytic cracking of PET makes it one of the most difficult polymer products to recycle. The aim of this study is to achieve more liquid products in pyrolysis of PET by introducing a Lewis-Brønsted acid sites catalyst. The pyrolysis of polyethylene terephthalate (PET) was studied in a semi-batch reactor using the Lewis-Brønsted acid sites catalyst. The experiments were carried out under isothermal condition to determine the kinetic parameters, product distribution, catalyst/PET mass ratio, and the effect of temperature on the conversion rate. The reaction products consisted of a gas, solid and liquid phase with a maximum liquid product of 6% at 350 °C. The optimum catalyst/PET mass ratio and temperature were also determined. In addition, the reaction order and activation energy for the reaction were determined. Since few detailed studies on the kinetics of PET pyrolysis by thermogravimetric analysis have been reported in the literature, this work presents the results of this investigation using the kinetic model.

Graphic abstract
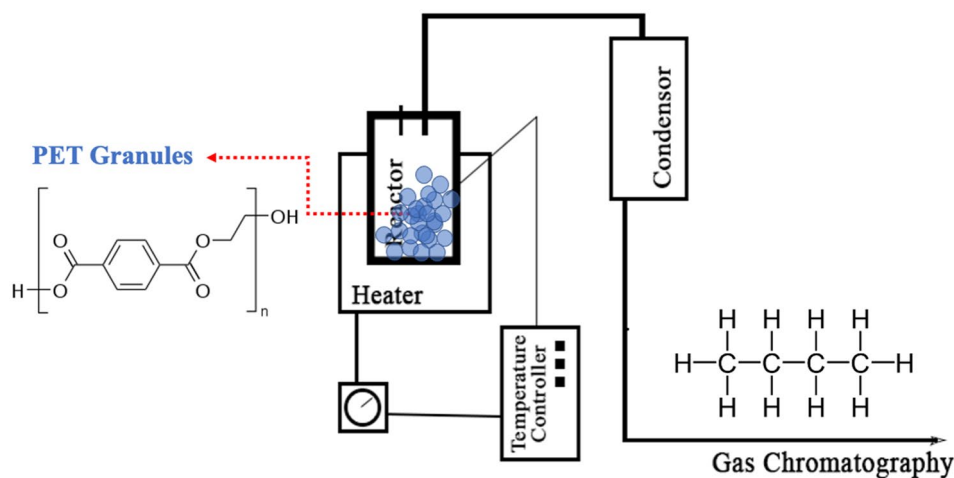
✉ Behrooz Roozbehani
  roozbehani@put.ac.ir

[1] Research Center of Chemical Engineering, Petroleum University of Technology, Abadan, Iran

[2] Chemical Process Engineering, Production Engineering Department, University of Bremen, Bremen, Germany

Introduction

Primary, secondary (or mechanical) and tertiary (or chemical) recycling are the main approaches for plastic recycling. In the primary and secondary recycling processes, plastics can be reused as second-hand products or modified products

(Scheirs 1998; Sorum et al. 2001; Öztürk and Güçlü 2004; Nikles and Farahat 2005; Niksiar et al. 2015). In contrast, chemical recycling processes convert plastic waste into its constituent monomers or hydrocarbon base materials (Al-Salem et al. 2009; Sinha et al. 2010; Welle 2011). Among chemical recycling approaches, catalytic cracking pyrolysis of waste plastics is one of the most applicable methods, in 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 AchiliasDS, , 2007; Jankauskaite and Lygaitis 2008).

The type of plastic alone influences the yield of the pyrolysis process, the molecular weight distribution, and the quality of the liquid products obtained during the catalytic degradation of waste plastics (Kumar and Rao 2003). In our previous studies in published in Clean Technologies and Environmental Policy, we have reported on the catalytic degradation of single and mixed polymer pellets such as polystyrene, blends of high-density and low-density polyethylene, containing various types of urban plastic wastes (Roozbehani et al. 2014, 2015, 2016; Sakaki et al. 2014; Imani Moqadam et al. 2015). Among all the materials discussed so far, polyethylene terephthalate (PET) is rather unique. This is due to its widespread use as a plastic bottle. Also, the catalytic pyrolysis of PET typically results in a wide range of gas products without significant liquid formation (Ding et al. 1997; Achilias and Karayannidis 2004). So far, the 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 example, the effect of heating rate on non-isothermal degradation and kinetic parameters was studied in an inert atmosphere (Jenekhe et al. 1983; Sorum et al. 2001; Spaseska 2010; Niksiar et al. 2015). It was found that the activation energy and the pre-exponential factor are extremely affected by the heating rate, while the reaction order remains constant (Niksiar et al. 2015). It was also shown that although thermal degradation is affected by particle size, it is strongly dependent on temperature in the range of 400 °C and 500 °C (Kpere-daibo 2009). Copper(II) chloride is known as one of the most active agents among the catalysts used in the catalytic cracking of PET (Chiu and Cheng 1999; Faravelli et al. 2001). It reduces the carbonaceous residues and increases the percentage weight loss of PET by 3.5 times compared to thermal degradation under similar reaction conditions. Taking into account the weight loss of the catalyst during the reaction, copper(II) chloride was found to be an effective catalyst (Awaja and Pavel 2005; Báez et al. 2013). Catalytic cracking of PET using copper(II) chloride not only resulted in a shortened cracking time, but also showed a similar temperature effect on polymer weight loss as the thermal degradation process with an optimum catalyst/PET weight ratio of 0.1 (Villain et al. 1994; Gladden and Mitchell 2011). The maximum weight loss was found to be 90.8% in the presence and 80.5% in the absence of the catalyst (Chiu and Cheng 1999).

Thermal degradation in a nitrogen atmosphere at 200–700 °C was also studied in a tube furnace under isothermal conditions by Dzie et al. (Dzięcioł and Trzeszczyński 1998). The main volatiles produced during the thermal decomposition of PET were CO, CO_2, acetaldehyde, acetophenone, aliphatic hydrocarbons (C_1–C_4) and benzene (Dzięcioł and Trzeszczyński 2000). In the temperature range of 200–300 °C, only acetaldehyde and formaldehyde were detected; at higher temperatures, the mixtures of volatile decomposition products of PET became more complex. The concentrations of carbon oxides, aromatic and aliphatic hydrocarbons were shown to increase with increasing temperature, with maximum yields of acetaldehyde and vinyl benzoate observed at 600 °C (Dzięcioł and Trzeszczyński 2000). In another work, the kinetics of PET soft drink bottle waste was studied under dynamic conditions and at different heating rates in nitrogen atmosphere (Villain et al. 1994). PET pyrolysis was found to result in a 70–80% weight loss of the polymer in the temperature range of 107 to 242 °C. The nth-order modeling technique provided a more accurate prediction of the experimental data than previously applied techniques, yielding an activation energy of 322.3 and 338.98 kJ/mol for Coca-Cola and Pepsi samples, respectively, and a reaction order of 1.72 (Saha and Ghoshal 2005).

In this work, the conversion of PET using a Lewis-Brønsted acid sites catalyst is investigated to improve the selectivity and activity of the catalytic degradation. Catalytic measurements were performed for the conversion of PET to lighter compounds, followed by a kinetic model determined from the experimental data. Different conversion percentages were evaluated at various temperatures for gas, liquid, and waste production. The effect of temperatures during the reaction was also evaluated. The other objective of this study is to estimate the reaction model of thermal catalytic cracking of PET by using a kinetic model. A suitable reaction model is suggested for the catalytic pyrolysis of PET.

Material and method

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 are responsible for proton addition. Lewis acid sites are responsible for hydride abstraction. Therefore, polymer degradation occurs at lower

temperatures compared to non-catalytic pyrolysis. The Lewis (electron acceptor) and Brønsted (proton donor) in the catalyst control the acidity due to its crystalline structure. The acid sites were generated by Al species in the catalyst, which consists of silica and alumina. The ratio of silica to alumina (Al/Si) in the catalyst powder significantly affects the cracking of polymer chains. A high density of acid sites improves the cracking of hydrocarbons. Note that the activated acid sites of the catalyst are sensitive to metal impurities in the feedstock.

Catalytic and isothermal decompositions were performed for a mixture of PET in bottle grade. General properties of the bottle grade were recorded for the samples. A maximum diethylene glycol (DEG) content of 2.0 wt% was determined. The melting point was approximately 250 °C. The water content of the samples was 0.3 wt% with a maximum carboxyl end group content of 32. The amount of acetaldehyde was also determined with a maximum value of 1 ppm.

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 to obtain a desired particle strength and morphology. Some characteristics of the catalyst are reported in Table 1.

Experimental

The catalytic degradation experiments were carried out with the pure PET pellets in a Pyrex reactor under isothermal conditions in a temperature range from 330 °C to 430 °C with a stepwise increase of 10 °C and with the catalyst proportions from 10 to 50% at atmospheric pressure. The schematic of the setup used in the measurements is shown in Fig. 1.

A semi-batch reactor and a thermogravimetric analysis (TGA) instrument were used to perform the polymer degradation reaction. TGA was used to determine the rate of degradation in catalytic and thermal processes and to determine the relationship between sample mass and temperature. The air in the system was purged with a stream of N_2 before performing the experiments. The catalytic experiments were performed in two steps: First, different catalyst fractions were investigated. Regardless of the rate of weight loss, the percentage of conversion at each temperature was
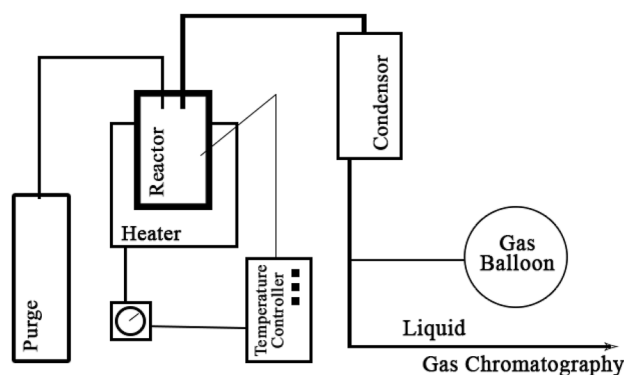

Fig. 1 Scheme of apparatus used for the thermal and catalytic degradation studies of materials in a nitrogen atmosphere

determined, and second, the optimum catalyst fraction was determined. The product consists of three phases, solid, liquid and gas. However, the gaseous phase is the dominant phase of the products. Thus, a condenser was used to cool the vapor product formed in the reactor. The presence of solids in the outlet caused clogging in some experiments, especially at high reaction temperatures. In addition, due to the presence of solids at the exit of the setup, the quality of the liquid product was low and measuring the weight loss over time was difficult. Since the catalytic degradation was conducted at atmospheric pressure, collecting and sampling the products in the constant volume balloon were rather difficult. Therefore, gas collectors and balloons with different volumes were used to collect the gaseous product for further analysis.

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 × 25 μm ID, 0.5 μm film of silica gel film, AGILENT, US 10,420,012, 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 quantitively 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.

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

Property	Unit	Minimum	Maximum
Al_2O_3	wt.%	38.5	48.2
Pt	wt.%	0.001	0.001
Total surface area	m²/gr	38	42
Attrition index	wt.%	1.5	5.3

Result and discussion

The parameters that influenced the conversion are discussed in this section. First, the effect of catalyst/polymer mass ratio is described. Then, the effect of temperature on the catalytic cracking process is discussed see section 'Temperature'.

Thermal and catalytic degradation analysis of PET

The influence of the catalyst weight fraction on the conversion was investigated at different temperatures. The catalyst weight fraction was selected in the range of 10 to 50% with a stepwise increase of 10%. The obtained conversion rates are given in Table 2 for different temperatures. It can be seen that as the amount of catalyst increases, the percentage weight loss of the reactant decreases. In general, no liquid product is formed during the thermal degradation of PET. However, some differences were observed with respect to product distribution. These observations are discussed in see section 'Products yield'. The effect of catalyst weight fraction on polymer weight loss is shown in Fig. 2. In general, an increase in the polymer/catalyst ratio leads to an increase in the yield of gaseous hydrocarbons and the formation of coke.

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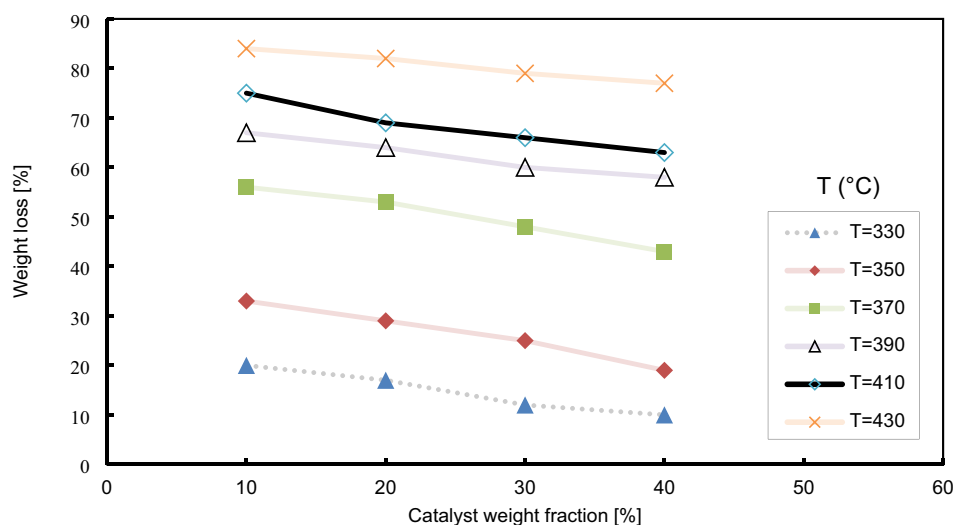
Temperature

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

By increasing the temperature, the conversion increases for a given catalyst loading. Thus, the reaction time was determined by the reaction temperature. The maximum PET weight loss occurred at the beginning of the reaction. According to Table 3, the degradation time decreases rapidly at higher temperatures (about 45 min at 430 °C). This fact is

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

Fig. 2 The catalyst weight fraction effect on the conversion

illustrated in Fig. 4, where the weight loss is plotted against the total processing time.

Products yield

The yields of liquid, gaseous and solid products as a function of temperature in the range of 330–430 °C with gradual increase 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 alkenes and ethylene. The breaking of chemical bonds during pyrolysis results in more gaseous products and thus more aliphatic gases such as CH_4, C_2H_6 and C_2H_4, as shown in Fig. 5c. One of the remarkable findings in this work is the presence of a

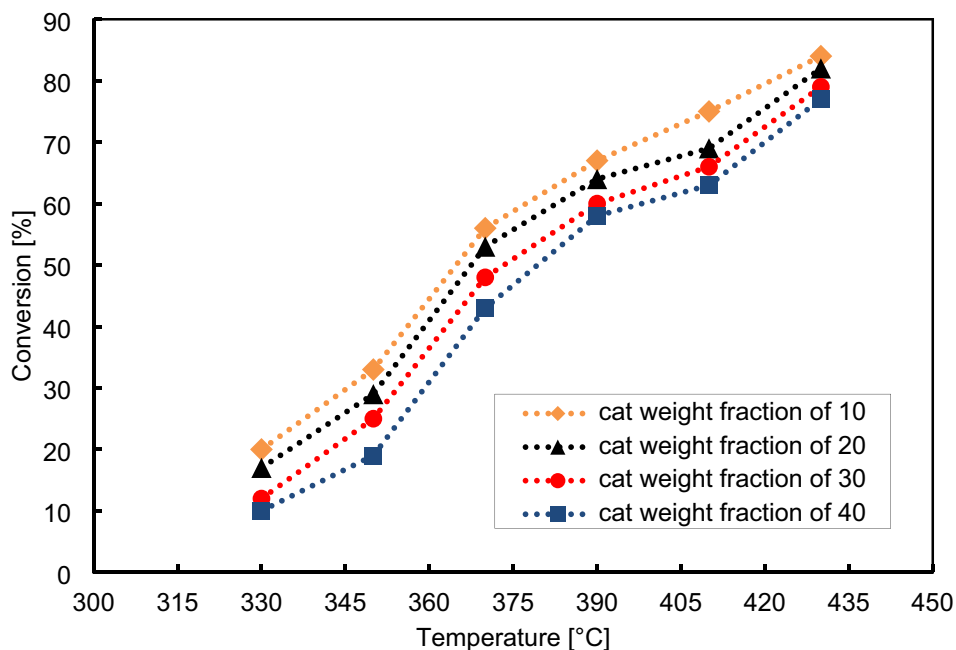**Fig. 3** Effect of temperature on conversion rate in different catalyst weight fractions

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

Fig. 4 Weight loss of PET at different temperatures
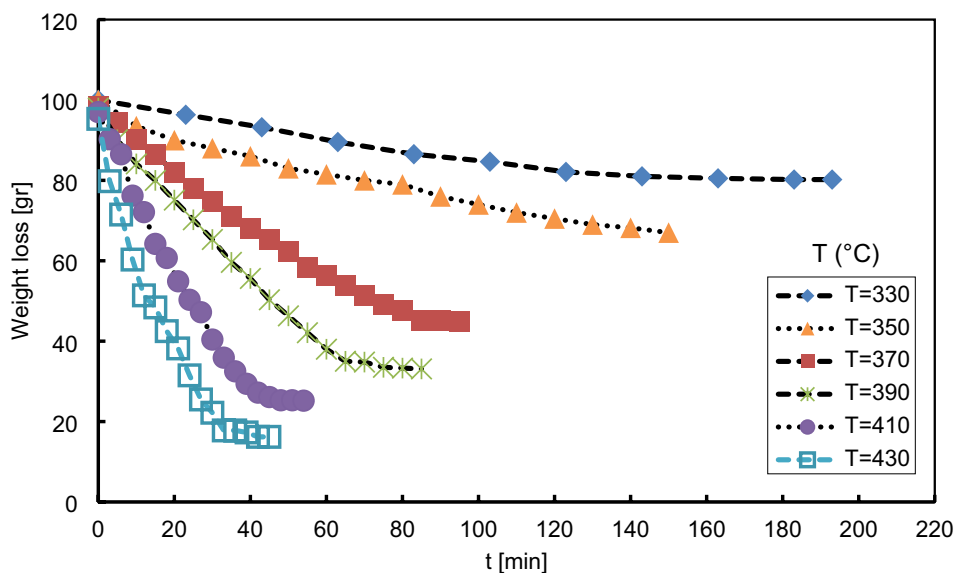
Table 4 Product yield versus temperature

T (°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

large amount of ethylene in the products, which increases with increasing temperature. The presence of a large volume of ethylene in gas is another interesting aspect related to the products. Moreover, unlike the other PET analyses

in the literature, where no formation of liquid products is reported, in this study, a small amount of liquid was formed at 330 °C and 350 °C (Fig. 5a). The maximum liquid in the products was reported to be 6% at a temperature of 350 °C and a catalyst to polymer ratio of 10%.

In general, the decomposition of PET leads to the production of solid and gaseous products. The solid products include a small amount of fine pale yellow particles consisting mainly of benzoic acid and terephthalic acid found at the outlet of the reactor, and some carbonaceous residues in the reactor. The solid phase product increases with increasing temperature. The formation of the solid phase is due to the breaking of more molecular bonds at higher temperatures. The main component of this solid phase product was terephthalic acid (TPA). TPA

Fig. 5 **a** Yield of liquid product, **b** solid product, **c** aliphatic gases, **d** aromatic gases, and **e** carbon oxides at various temperatures
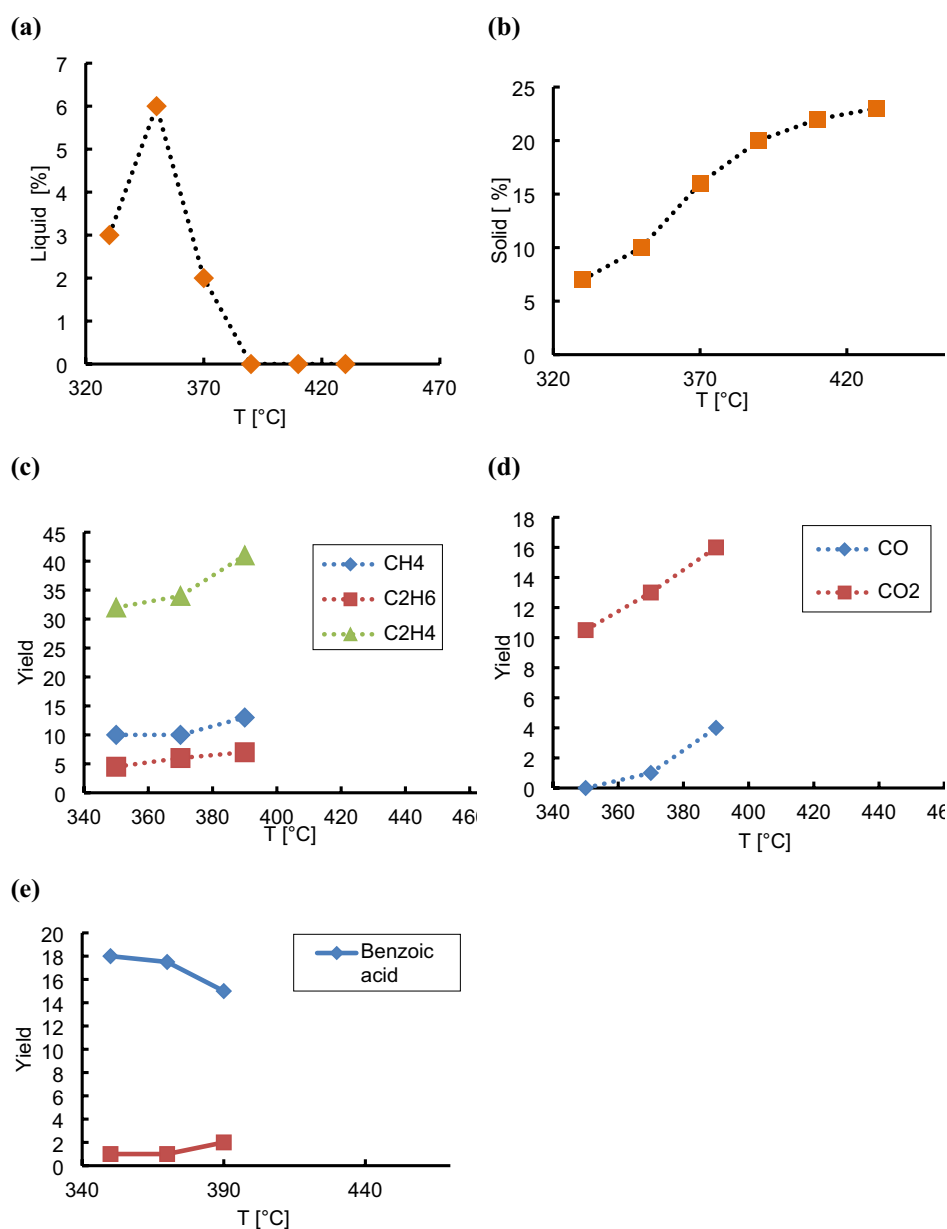
Table 5 The formed volatiles in the catalytic degradation of PET

Gas composition	350 °C	370 °C	390 °C
CO	0	1	4
CO_2	10.5	13	16
CH_4	10	10	13
C_2H_6	4.5	6	7.5
Benzoic acid	18	17.5	15
C_2H_4	32	34	41
Benzene	1	1	2
C_2H_4O	2.5	1.5	0.5
Other	21.5	16	1

decomposes at high temperatures to benzene, carbon dioxide, and benzoic acid. Figure 5b illustrates the increase of the solid product at higher temperatures. The main components of the solid product are terephthalic acid and benzoic acid. As shown in Figs. 5d, e, the yield of benzoic acid, carbon dioxide and benzene increases with increasing temperature.

Terephthalate is the main product of the pyrolysis of polyethylene. By increasing the temperature, the ratio of gas to solid increases. At 350 °C, the gas to solid ratio is 1.42, and at 430 °C, it is 2.65. The volatiles formed during pyrolysis are specifically listed in Table 5.

Kinetic parameters

It is known that the thermal and catalytic decomposition process of PET atmospheric pressure can be represented by the reaction scheme: $A_{1(s)} \rightarrow A_{2(s \, or \, l)} \rightarrow A_{3 \, (g)}$, where A_1 is the solid polymer with the structure, and A_2 consists mainly of terephthalic acid and benzoic acid. As a result, the rapid production of volatile fragments with low molecular weight and consequent weight loss is expected. The volatile components (A_3) are mainly COOH, CO, CO_2, C2H$_4$ and CH_4. Since few detailed studies on the kinetics of PET pyrolysis by thermogravimetric (TG) analysis have been reported in the literature, this work presents the results of this investigation using the kinetic model (Sorum et al. 2001; Sánchez-Jiménez et al. 2009; Al-Salem and Lettieri 2010).

Isothermal thermogravimetric analysis (TGA) has been used to study the mass change of a sample as a function of temperature or time, since the reaction process can be followed over a wide temperature range. The kinetics of PET decomposition is considered heterogeneous because more than one phase exists during the reaction. The reaction rate of a heterogeneous reaction can be described as follows:

$$\frac{-\mathrm{d}m}{\mathrm{d}t} = k(t)f[m(t)] \tag{1}$$

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:

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

where A is pre-exponential factor, E is the activation energy (kJ mol^{-1}), R represents the universal gas constant (kJ mol^{-1} 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

$$f[m(t)] = (m)^n \tag{3}$$

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

$$\frac{-\mathrm{d}m}{\mathrm{d}t} = Ae^{(-E/RT)}(m)^n \tag{4}$$

Here, the kinetic parameters were calculated in two modes ($n \neq 1$ and $n = 1$). According to Eq. (4), for each temperature, there is a certain n, E, and A. There are three unknown parameters, so the determination of the reaction order (n) is possible for all temperatures. Moreover, the kinetic parameters change with temperature. To determine E and A, Ln (k) should be plotted against (1/T). The logarithmic model of Eq. (4) is as follows:

$$\ln(\frac{-\mathrm{d}m}{\mathrm{d}t}) = [\ln(A) - \frac{E}{RT}] + n\ln(m) \tag{5}$$

The values of the order of reaction for other temperatures (330 °C, 350 °C, 390 °C, and 410 °C) are calculated by nth order kinetic model in Table 6.

Kinetic parameters (second mode, $n = 1$)

If a reaction order of is considered for the catalytic degradation of PET, Eq. (4) can be rewritten as follows when $n = 1$:

$$\frac{-dm}{dt} = Ae^{(-E/RT)}m \tag{6}$$

Manipulating this equation leads to

Table 6 Kinetics parameters derived for other temperatures

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

Table 7 Kinetics parameters derived from experiments

State	E (kJ mol^{-1})	A (min^{-1})
$n=1$	323.87	48×10^{21}
$n\neq1$	109	1,013,141.08

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

The nth-order model technique $n\neq1$ gives higher values for E and $\mathrm{Ln}(A)$ compared to "first order" (see Table 7). The kinetic parameters change with temperature in different ways, even if one can assume that they are constant.

In the case of the $n\neq1$ nth-order model technique, the values of E and A are 109 (kJ mol^{-1}) and 1,013,141.08 (min^{-1}), respectively. In this study, the order of the reaction changed from 1.01 to 1.49. Also, in the "first-order" technique, E and A are 323.87 (kJ mol^{-1}) and 48×1021 (min^{-1}), respectively. When considering the nth-order model technique, it was found that $n\neq1$ is better suited to describe the catalytic pyrolysis of PET because unlike the first-order technique, no assumptions or degrees of reaction are required.

Conclusion

The results showed the effect of temperature and catalyst/PET on degradation of PET using a Lewis-Brønsted acids side catalyst. With increasing temperature, the conversion rate increased forming gas, liquid and solid phase products. The efficiency of the degradation process decreased with increasing catalyst/PET mass ratio, because at higher catalyst ratios, the PET molecules were surrounded by the catalyst, resulting in lower reaction rate and insufficient heat transfer to the PET molecules. Analysis of the gas sample at higher temperature showed that CO, CO_2 and aliphatic hydrocarbons increased and terephthalic acid was degraded to benzene and benzoic acid. Finally, the most suitable kinetic model was found to be the n$^{\text{th}}$-order model for the catalytic reactions.

Declarations

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