KINETIC INVESTIGATION OF LOW-DENSITY POLYETHYLENE PYROLYSIS: EFFECTS OF HEATING RATE

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This study investigates the kinetic behavior and thermal degradation mechanisms of low-density polyethylene (LDPE) plastic waste during pyrolysis at 500 °C. Using thermogravimetric analysis performed in a modified muffle oven, the pyrolysis process was examined under non-oxidative atmosphere to determine mass variation, in-sample temperature profile, and kinetic triplet, across heating rates of 10 °C/min and 15 °C/min. The activation energy, pre-exponential factors and kinetic models were estimated by using model-fitting techniques, such as Coats-Redfern and master plots for the most common kinetic mechanisms. These findings provide a comprehensive understanding of the LDPE pyrolysis kinetics, offering insights for optimizing thermal conversion pathways.

Keywords: plastic waste, LDPE, pyrolysis, thermogravimetric analysis, kinetic study, activation energy

1. Introduction

Over 413.8 Mt of plastic were produced in 2023 worldwide. Low density polyethylene (LDPE) being the second most demanded, representing 14% of the total plastic production below the production of polypropylene (PP) (19%), followed by polyvinyl chloride (PVC) (12.8%), high-density polyethylene (HDPE) (12.2%), polyethylene terephthalate (PET) (6.2%), polyurethane (PUR) (5.3%), polystyrene (PS) (5.2%), and other (15.7%) [1]. The main source of LDPE waste comes from post-consumer packaging sector, where only 26.9% of the total collected post-consumer waste is being recycled.

Pyrolysis is an effective method of chemical recycling of plastic waste, converting it into valuable fuels and chemicals, such as pyrolysis oil, waxes, gases and aromatic compounds [2], [3], [4], [5]. It is the process of thermal cracking long polymer chains into smaller molecules in a non-oxidative atmosphere at

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temperatures above 400 °C. Due to the proximate composition of plastic materials, which is preponderant made of volatile matter (95-98%), this offers a great opportunity of generating fuel quality liquids, up to 70-80 wt.% [6].

Pyrolysis of LDPE has been used extensively to obtain high amounts of liquid products. For example, Aguado et al. 2007, has investigated the thermal pyrolysis of LDPE at 425 °C, 450 °C, 470 °C with a heating rate of 10 °C/min and obtained between up to 74% liquid fraction, preponderantly in the diesel (C13-C20) ranges. While in a study by Akgün et al. 2021, the highest liquid yield (approx. 86%) was obtained at 800 °C with 5 °C/min heating rate. Generally, the oil produced contains hydrocarbons in gasoline (C7-C12) and diesel (C13-C20) ranges [9], [10].

Kinetic studies provide insights into reaction mechanisms, activation energy, enabling the prediction of product distributions under various operating conditions, optimizing the process and designing more efficient reactors [11]. There are numerous kinetic studies of LDPE pyrolysis in literature using model-fitting and model-free methods. Dubdub et al. 2020 investigated the kinetic LDPE using both methods and obtained values for the activation energy between 193 and 195 $kJ \cdot mol^{-1}$, while Xie et al. 2023 obtained values of 271 $kJ \cdot mol^{-1}$ for the activation energy, but in different conditions. Aboulkas et al. 2010, have also studied the kinetic parameters of LDPE and for the model-free methods they obtained values between of 228-247 $kJ \cdot mol^{-1}$. Moreover, by applying the Coats-Redfern and Criado method, they were able to estimate the mechanism of LDPE pyrolysis follow a contracting sphere (R_2) model.

In this study, the LDPE waste pyrolysis and TGA was performed in a muffle furnace at 500 °C, thus overcoming the limitations of the TGA instruments. Furthermore, the kinetic study was performed with model-fitting methos (Coats-Redfern and master plots) at different heating rates (10 °C/min and 15 °C/min). Various common reaction mechanisms were investigated and through careful analysis, the appropriate kinetic triplet and reaction model has been selected.

2. Materials and Methods

2.1. Materials procurement and pretreatment

LDPE film. The LDPE used in this study is from packaging waste, obtained from industrial applications. The waste foil is not pure LDPE, but an enhanced material with different additives to meet mechanical requirements of industrial packaging applications. The LDPE waste was cleaned and reduced in size in an electrical mill.

LDPE cylinder. To measure the temperature inside the sample during processing the LDPE foil was transformed into one solid cylindrical block by low temperature partial melting in calcination oven (Nabertherm muffle electric furnace type

L9/11/SW). The LDPE powder was placed in a refracted steel cylindrical crucible, pressed, and heated at 140°C. To ensure the formation of a dense, air-free LDPE cylinder, successive layers of powder were added and compressed during the heating process. The resulting cylinder measured 25 mm in height, 31 mm in diameter, and an approximate 18.18 ± 0.2 g mass.

2.2. Pyrolysis experimental set-up

Pyrolysis Reactor. The pyrolysis of LDPE was conducted in the same oven at 500 °C and two different heating rates: 10 °C/min, respectively 15 °C/min. The LDPE cylinder was placed inside the furnace in a refracted steel crucible with a lid, restricting the air access to the sample. The pyrolysis was conducted until no mass variation was recorded during the experiment, approximately 48 min for the lower heating rate, and 37 min respectively for the higher heating rate. At least three experiments were performed for each heating rate, until minimum standard deviation was achieved and the results averaged.

Thermogravimetric analysis. For the thermogravimetric analysis of LDPE waste, the in-sample temperature and mass variation were recorded in real time for the full durations of the pyrolysis experiments using a custom-made Arduino based data acquisition system. The temperature was measured using a type K thermocouple with 1 mm diameter was placed in the middle of the LDPE cylinder. The crucible containing the/ LDPE sample was placed support connected to a KERN KB2000-2N balance which recorded the mass variation in real time. Fig. 1. depicts the simplified experimental design.

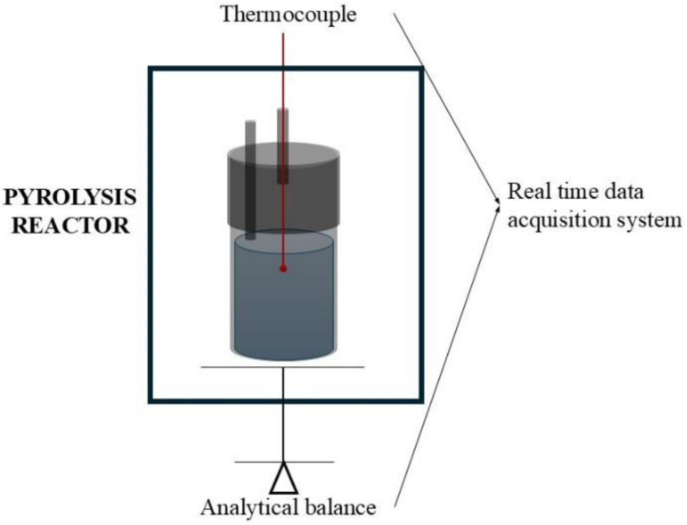

Fig. 1. Simplified schematic representation of the experimental set-up2.

2.3. Kinetic study

The kinetic analysis is implemented to determine the rate and mechanism in which the sample is decomposed. The computations for the kinetic study began with normalizing the output from the thermogravimetric study. The dimensionless conversion rate, α, is defined in this regard using Eq. (1).

$$\alpha = \frac{m_i - m_{(t)}}{m_i - m_f} \tag{1}$$

where m_i (g) represents the initial mass of the sample, $m_{(t)}$ (g) the mass at the moment t and m_f (g) is the final mass.

The classic isothermal single-step solid-state reaction, described by the Arrhenius expression was used to model the process:

$$\frac{d\alpha}{dt} = k(T)f(\alpha) = A\, e^{\frac{-E_a}{RT}} f(\alpha) \tag{2}$$

where $k(T)$ is the rate constant, further expressed by the Arrhenius equation, A (min^{-1}) is the pre-exponential factor, E_a (kJ/mol) is the apparent activation energy of the process and $f(\alpha)$ is the reaction model function which describes the conversion pathway. For example, a n^th order reaction **Error! Reference source not found.**:

$$f(\alpha) = (1-\alpha)^n \tag{3}$$

For non-isothermal conditions, the heating rate plays a crucial role in determining the kinetics of the process, Eq. (2) becoming:

$$\beta \frac{d\alpha}{dt} = A\, e^{\frac{-E_a}{RT}} f(\alpha) \tag{4}$$

where β $(K\ min^{-1})$ is the heating rate of the process and can be expressed as Eq. (5):

$$\beta = \frac{dT}{dt} = \frac{dT}{d\alpha} \times \frac{d\alpha}{dt} \tag{5}$$

Substituting in β Eq. (4) with the new expression Eq. (5), after rearranging we obtain:

$$\frac{d\alpha}{dT} = \frac{A}{\beta}\, e^{\frac{-E_a}{RT}} f(\alpha) \tag{6}$$

$$\frac{d\alpha}{f(\alpha)} = \frac{A}{\beta}\, e^{\frac{-E_a}{RT}} dT \tag{7}$$

The integrated form of the kinetic equation of Eq. (7) is as follows:

$$g(\alpha) = \int_0^\alpha \frac{d\alpha}{f(\alpha)} = \frac{A}{\beta}\int_0^T e^{\frac{-E_a}{RT}}\, dT \qquad (8)$$

The purpose of this kinetic study is to obtain the values of the kinetic triplet $(A$, E_a and $n)$ which describes the pyrolysis process for the studied sample. Therefore, the kinetic parameters rely on eq. (2) and can be determined through two main pathways, model-fitting or model-free methods [15]. For most model-free methods, the reaction model is assumed to be a first order, and the kinetic triplet is calculated as a function of the conversion rate [12], [16]. On the other hand, the model-fitting methods assume a reaction model for the kinetic parameters to be calculated [17]. For the purposes of this paper, the kinetic study was performed by model-fitting approaches, using Coats-Redfern and master plots techniques. For both techniques, the reaction models proposed for investigation are presented in Table 1.

Table 1

Solid-state reaction models used in kinetic analysis of LDPE pyrolysis

Reaction mechanism			Integer form, f(α)	Differential form, g(α)
Reaction Order	First Order (Mampel)	F1	$1-\alpha$	$-\ln(1-\alpha)$
	Second order	F2	$(1-\alpha)^2$	$[(1-\alpha)^{-1}]-1$
	Third order	F3	$(1-\alpha)^3$	$\{[(1-\alpha)^{-2}]-1\}/2$
Diffusion Models	One Dimension	D1	$1/(2\alpha)$	α^2
	Two Dimensions	D2	$(-\ln(1-\alpha))^{-1}$	$(1-\alpha)\ln(1-\alpha)+\alpha$
	Three Dimensions (Jander)	D3	$3[1-(1-\alpha)^{2/3}]/(2(1-(1-\alpha)^{1/3})$	$[1-(1-\alpha)^{1/3}]^2$
Nucleation models (Avrami-Erofeev)	Two Dimensions	A2	$2(1-\alpha)[-\ln(1-\alpha)]^{1/2}$	$[-\ln(1-\alpha)]^{1/2}$
	Three Dimensions	A3	$3(1-\alpha)[-\ln(1-\alpha)]^{1/3}$	$[-\ln(1-\alpha)]^{1/3}$
	Four Dimensions	A4	$4(1-\alpha)[-\ln(1-\alpha)]^{1/4}$	$[-\ln(1-\alpha)]^{1/4}$
Geometrical contraction models	One Dimension	R1	1	α
	Sphere (Area)	R2	$2(1-\alpha)^{1/2}$	$1-(1-\alpha)^{1/2}$
	Cylinder (Volume)	R3	$3(1-\alpha)^{1/3}$	$1-(1-\alpha)^{1/3}$
Nucleation Models	Two-power	P2	$2\alpha^{1/2}$	$\alpha^{1/2}$
	Three-power	P3	$3\alpha^{2/3}$	$\alpha^{1/3}$
	Four-power	P4	$4\alpha^{3/4}$	$\alpha^{1/4}$
Autocatalytic reactions	Autocatalytic reaction	B1	$\alpha(1-\alpha)$	$\ln[\alpha/(1-\alpha)]$

Therefore, through model-fitting, a specific reaction model is investigated and the kinetic parameters associated with it are derived. The process involves fitting the proposed reaction models to the experimental data obtained [17].

2.3.1. Coats-Redfern method

The Coats-Redfern method is widely used for the calculation of reaction order, pre-exponential factor and activation energy [12], [16], [17]. It's an integral model-fitting with the final equation [17], [18]:

$$\ln\left(\frac{g(\alpha)}{T^2}\right) = \ln\left(\frac{AR}{\beta E_a}\right) - \frac{E_a}{RT} \qquad (9)$$

By plotting $\ln\,(g(\alpha)/T^2\,)$ against $1000/\mathrm{T}$, a straight line correlation at a constant heating rate β will be obtained with the slope E_a/R and intercept $\ln\,(AR/\beta E_a)$. Determining the slope and intercept of the correlation, the activation energy and the pre-exponential factor can be calculated using:

$$E_a = slop\ \times R\ (\frac{kJ}{mol}) \qquad (10)$$

$$A = \frac{\beta E_a}{\mathrm{R}} \times e^{intercept}\ (\mathrm{s}^{-1}) \qquad (11)$$

2.3.2. Master Plots

Master plot methods are using either the integral or the differential forms of reaction mechanisms, as presented in Table 1. In this study, the integral form was chosen to analyze the experimental data. Master plots compare the fitting of the experimental results to the theoretical master plots for different kinetic models. Therefore, the most appropriate kinetic model is chosen based on the overlay of the theoretical and experimental results.

Eq. (12) represents the master plot method, which compares various theoretical reaction models (left side of the equation) with the experimental data (right side of equation).

$$\frac{\mathrm{Z}(\alpha)}{\mathrm{Z}(0.5)} = \frac{\mathrm{f}(\alpha)}{f(0.5)} = \frac{T_\alpha}{T_{0.5}} \times \frac{\left(\frac{d\alpha}{dt}\right)_\alpha}{\left(\frac{d\alpha}{dt}\right)_{0.5}} \qquad (12)$$

where $\mathrm{f}(\alpha)/f(0.5)$ is the integral form of the reaction model; $T_\alpha/T_{0.5}$ are the temperatures (K) at conversion rates α, and $\alpha = 0.5$; $(d\alpha/dt)_\alpha$ and $(d\alpha/dt)_{0.5}$ are the reaction rates at conversion rate α, and $\alpha = 0.5$, respectively. The $\alpha = 0.5$ serves as a reference point because it normalizes all the theoretical plots to a value of 1 [19].

3. Results and Discussion

3.1. LDPE waste transformation during pyrolysis process

The results of the mass variation and the first derivative of the TG analysis (DTG) for the pyrolysis of LDPE at 500 °C for both heating rates are presented in Fig. 2. It can be observed that no significant differences are displayed, both heating rates following the same degradation behavior. The experimental data of LDPE waste shows that the main thermal decomposition occurs in the 450-500 °C range, which is validated by the literature data on this type of plastic [12], [20], [21]. For the 10 °C/min heating rate, the maximum degradation occurs at approx. 486 °C, while for the 15 °C/min heating rate at approx. 500 °C.

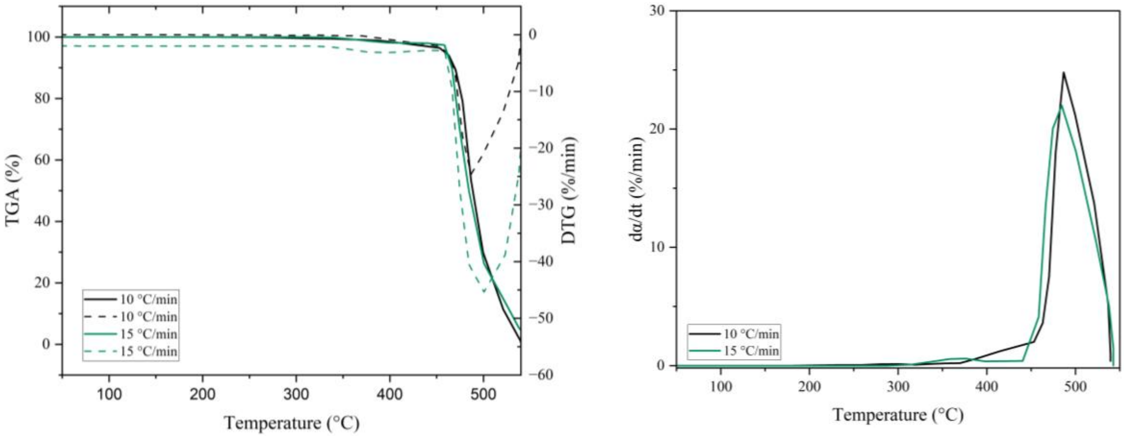

Fig. 2. Mass loss of LDPE sample together with the DTG results (left), and conversion rate of the two heating rates (right)

By analyzing the first derivative of the DTG curves, we can determine the onset temperature (T_{onset}), the offset temperature (T_{offset}) and the peak degradation temperature (T_{peak}). T_{onset} is an important parameter that describes the thermal stability of the material, being the temperature at which the LDPE begins to degrade [22]. T_{offset} indicates the temperature at which the degradation of LDPE is completed, no significant changes in mass occurring. T_{peak} represents the temperature at which the maximum mass loss occurs. The results of this analysis are presented in Fig. 3.

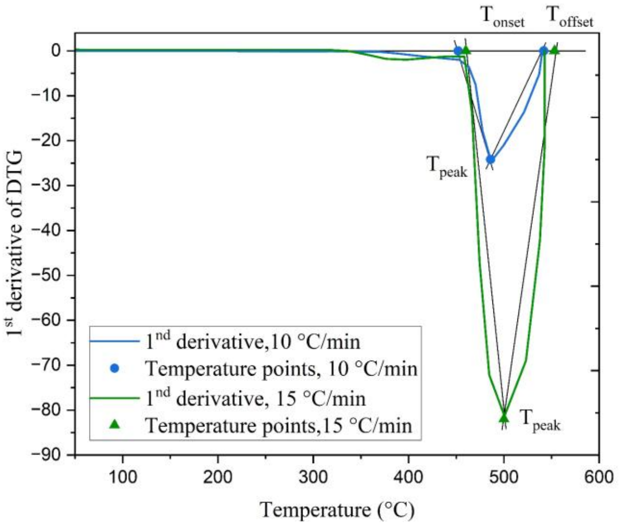

Fig. 3. The onset, offset and peak degradation temperatures determined from the first derivative of the DTG against temperature

Analyzing the values presented in Table 2, it can be observed an increase in T_{onset} from 452 °C at 10 °C/min to 460 °C at 15 °C/min, which suggest that for the initialization of thermal degradation of LDPE requires higher temperatures as the heating rate is increased. T_{offset} also temperature increases from 541 °C at 10 °C/min to 553 °C at 15 °C/min. T_{peak} increases from 486 °C at 10 °C/min to 500 °C at 15 °C/min. In a study done by Santos 2018 , the authors obtained similar results for the T_{onset} 452-479 °C for heating rates between 10-50 °C/min. Therefore, these results demonstrate the direct proportionality of these key temperatures with the increase of heating rates, which highlights the importance of the heating rate in the thermal degradation of LDPE.

Table 2

The onset, offset and peak degradation temperatures values for both heating rates

Heating rate (°C/min)	T_{onset} (°C)	T_{offset} (°C)	T_{peak} (°C)
10	452	541	486
15	460	553	500

3.2. Kinetic Modeling of LDPE waste

3.2.1 Coats-Redfern method

In this study, Coats-Redfern method is applied to determine the range for the E_a, and A for both heating rates. Table 3 presents the kinetic results for the Coats-Redfern method applied to different reaction mechanism. Overall, all reactions mechanisms can be used to plot ln $(g(\alpha)/T^2)$ against $1000/T$ with a high correlation factor, which ca be observed in Fig. 4.

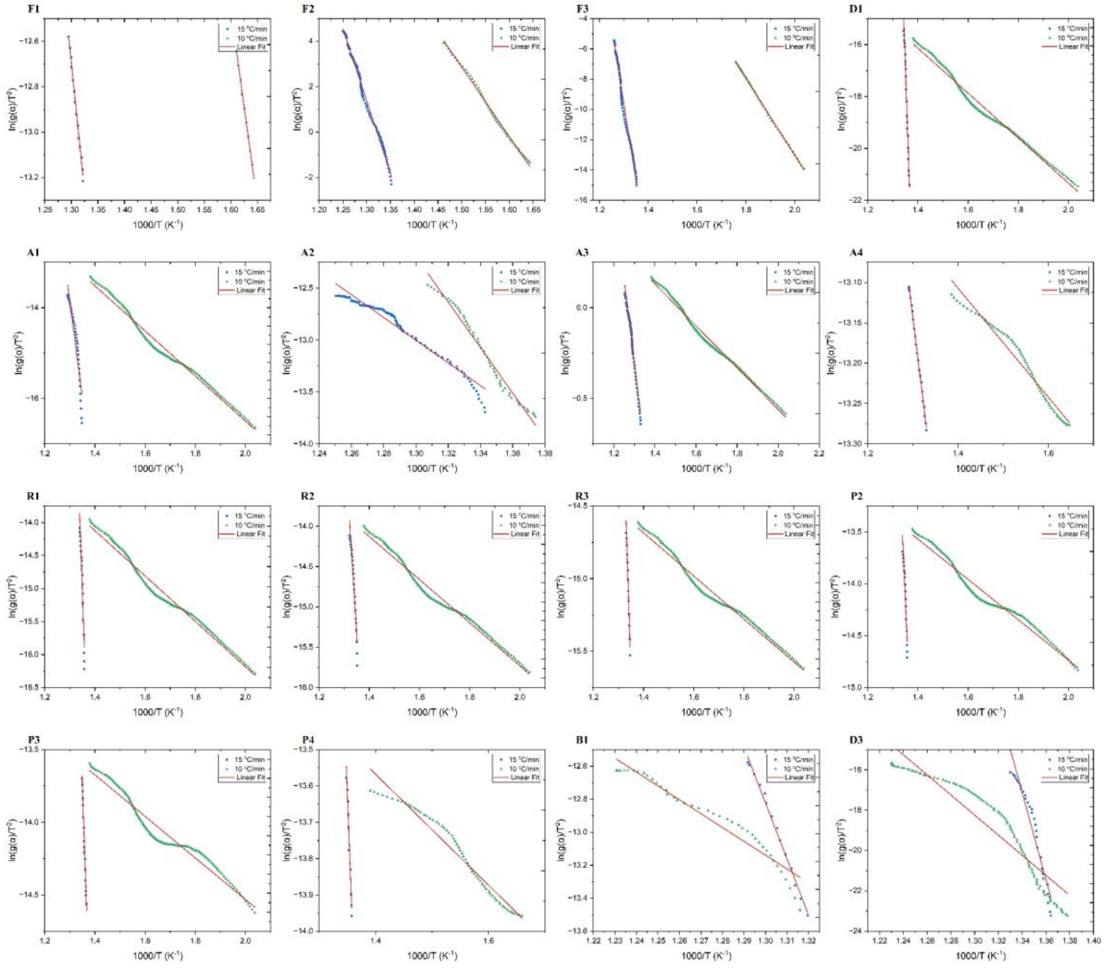

Fig. 4. The onset, offset and peak degradation temperatures determined from the first derivative of the DTG against temperature

These results are compared with the expected values that are available in literature and with the common reaction types that are possible for the pyrolysis of LDPE. Abnormalities are removed from the results. Therefore, diffusion mechanisms (D1, D3), are excluded from the results as their E_a are too high (2342.41 kJ/mol, respectively 1791 kJ/mol) for the 15 °C/min heating rate. In the same way, the A3, A4, P2, P3, P4 reaction models compute a low E_a, below 28 kJ/mol, and therefore excluded for the 10 °C/min heating rate. After a preliminary exclusion, the E_a ranges from 46-208 kJ/mol. Considering the very high variation of A for different reactions mechanisms, it can be noted that the most accurate E_a lies in 112-208 kJ/mol range for the 10 °C/min heating rate. This variation also suggests that this method cannot be applied individually for the determination of the reaction mechanisms [24]. Analogically, for the 15 °C/min heating, the values for E_a that are supported by the literature are between 189-228 kJ/mol. Wong et al. obtained similar values for the Ea (166.78 kJ/mol), while Dubdub et al. 2020 found that it ranges from 105- 260 kJ/mol.

Reaction mechanisms such as F1, F2, and F3 show consistently high R^2 values (>0.99) for both heating rates, suggesting that these mechanisms provide a better fit for describing the thermal degradation kinetics of LDPE.

Table 3

Kinetic parameters for different reaction mechanisms of LDPE pyrolysis

	10 °C/min			15 °C/min		
Reaction mechanism	R^2	E_a, kJ/mol	A, s^{-1}	R^2	E_a, kJ/mol	A, s^{-1}
F1	0.9988	112.82	8.30E-01	0.9939	189.02	1.19E+11
F2	0.9962	141.43	7.11E+08	0.9910	228.45	4.89E+40
F3	0.9995	95.15	1.26E-01	0.9797	243.65	3.21E+57
D1	0.9859	102.96	1.91E+02	0.9734	2342.41	5.61E+162
D2	0.9843	103.23	1.02E+02	0.8995	349.88	5.76E+21
D3	0.8888	437.16	4.73E+26	0.9333	1791.60	5.00E+122
A2	0.9806	208.32	2.12E+12	0.9486	91.11	9.54E+03
A3	0.9912	28.73	1.70E+04	0.9735	75.83	2.36E+08
A4	0.9623	7.49	6.99E-04	0.9960	37.80	8.21E-01
R1	0.9824	46.74	2.25E-01	0.9153	890.80	5.06E+60
R2	0.9823	46.83	1.15E-01	0.9362	408.77	1.80E+26
R3	0.9822	46.86	7.72E-02	0.9763	450.05	1.15E+29
P2	0.9713	18.46	4.36E-03	0.9133	457.15	1.73E+30
P3	0.9525	9.03	7.78E-04	0.9792	415.961	2.82E+27
P4	0.9582	7.61	7.28E-04	0.9681	342.415	1.75E+22
B1	0.9367	205.90	9.12E+11	0.9927	281.838	3.15E+17

3.2.2. Master Plot method

Master plot method was used to determine the reaction mechanism of the LDPE pyrolysis process. By plotting the experimental results $T_\alpha/T_{0.5} \times (d\alpha/dt)_\alpha/(d\alpha/dt)_{0.5}$ in a $f(\alpha)/f(0.5)$ vs. α, it can be observed the mechanism on which it fits best. The values for α were selected from 0.01 to 0.99 with a 0.01 step. The result of this plot is presented in Fig. 5.

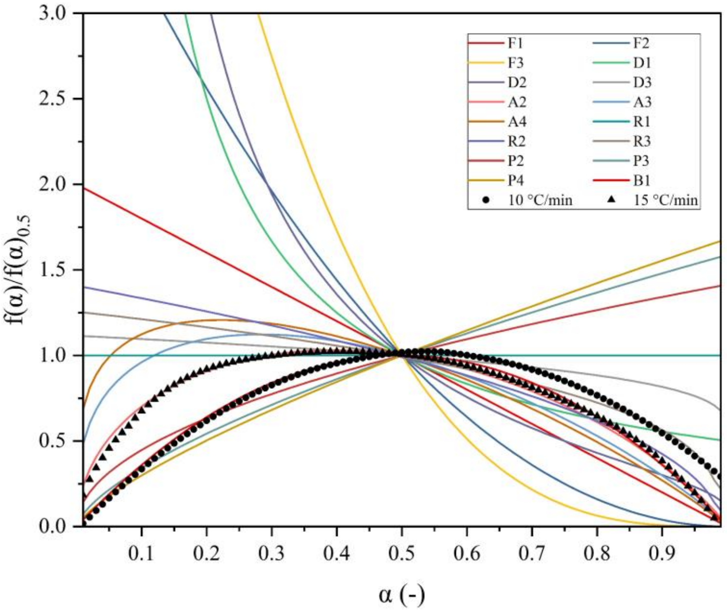

Fig. 5. Fitting of the experimental curves for both heating rates with the theoretical master plot

The correlation factor was calculated for each proposed reaction mechanism and centralized in Table 4. The R^2 ranges from 0.083 (R3) to 0.926 (B1) for the 10 °C/min heating rate and 0.0002 (F3) to 0.99 (A2) for the 15 °C/min heating rate. This variation indicates that most of the proposed reaction mechanisms have poor performance compared to the experimental results. Therefore, for the 10 °C/min heating rate it suggests that the mechanism that better fits the thermal degradation is an autocatalytic process (B1), while the higher heating rate 15 °C/min follows a two-dimension nucleation model (A2).

Table 4

Correlation factor (R^2) of the experimental dataset against the theoretical master plot

Reaction mechanism	10 °C/min	15 °C/min	Reaction mechanism	10 °C/min	15 °C/min
F1	0.0740	0.1427	A4	0.0485	0.6106
F2	0.2518	0.0198	R1	-	-
F3	0.3944	0.0002	R2	0.0247	0.1907
D1	0.3180	0.1035	R3	0.0083	0.2400
D2	0.3164	0.1008	P2	0.1906	0.0402
D3	0.0012	0.3355	P3	0.1442	0.0708
A2	0.5376	0.9922	P4	0.1238	0.0878
A3	0.1647	0.7881	B1	0.9257	0.8271

4. Conclusions

In this study, the kinetic parameters were determined for the pyrolysis of low-density polyethylene. The process temperature selected was 500 °C and performed at two heating rates: 10 °C/min, respectively 15 °C/min. Unlike most thermogravimetric studies on LDPE, the mass variation was performed in a custom-made muffle furnace connected to a real-time mass and in-sample monitoring system. The kinetic study was performed using two model-fitting methods for different purposes. The Coats-Redfern method was applied to the TGA data to obtain an appropriate range of activation energy and pre-exponential factor. The master plot technique was then used to identify the most fitting reaction model for the pyrolysis of LDPE at the two heating rates.

Firstly, the Coats-Redfern results show that there is evident difference between the two heating rates, and the activation energy is between 112-208 kJ/mol range for the 10 °C/min heating rate, and for the 15 °C/min heating is between 189-228 kJ/mol. These values are supported by the relevant literature.

Secondly, the master plot technique was applied to identify the reaction model that better fits the experimental data. Therefore, by analyzing the correlation factor it was determined that the LDPE degradation at 10 °C/min follows an autocatalytic reaction (B1) and at 15 °C/min a two-dimension nucleation model (A2). While these results correspond to the higher correlation factor, the literature indicates that they are not common degradation pathways for LDPE pyrolysis. This highlights the importance of the specific process parameters, as well as the difference between a close-to-real experiment versus the data obtained from specialized TGA instruments.

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