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Evaluating Model-Free Methods in the Catalytic Pyrolysis of HDPE

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Abstract - This study investigates the catalytic pyrolysis of high-density polyethylene (HDPE) using the nanocatalyst HZSM-5, focusing on the comparison of different model-free kinetic methods. Thermogravimetric analysis (TGA) data at various heating rates and catalyst-to-polymer ratios were analyzed using the Sarink (STK), DAEM, and advanced isoconversional (AIC) methods. The activation energy for the catalytic thermal decomposition of HDPE was determined using each method, and the results were compared to assess the accuracy and consistency of the models. The study found that all three methods predict a substantial decrease in activation energy with the addition of HZSM-5 catalyst, indicating its effectiveness in promoting HDPE pyrolysis. The AIC method, known for its improved accuracy, predicted a decrease in activation energy ranging from 79 to 105 kJ mol^{-1}. These findings were consistent with previous studies utilizing traditional isoconversional methods such as Friedman, Flynn–Wall–Ozawa, and Kissinger–Akahira–Sunose. The consistent trends and values observed across the three models suggest their applicability and reliability in describing the kinetic parameters of this catalytic process. This research provides valuable insights into the selection and application of model-free kinetic methods for the analysis of polymer pyrolysis, contributing to the development of sustainable polymer disposal strategies.

***Keywords*:** HDPE Pyrolysis; HZSM-5 Catalyst; Model-Free Kinetics; Isoconversional Methods

1. Introduction

High-density polyethylene (HDPE) is a thermoplastic polymer produced from the polymerization of ethylene [1]. Its properties are influenced by the length of the polymer chain, also known as the molecular weight [2]. HDPE is favored for its high strength-to-density ratio, durability, and chemical resistance. It is widely used in various applications, including packaging, piping, construction, and consumer products. However, being a petroleum-based plastic, HDPE production can negatively impact the environment [3].

The use of nanomaterials as catalysts, known as nanocatalysis, has gained significant attention due to their unique properties such as high surface area and enhanced reactivity. HZSM-5, a microporous aluminosilicate zeolite with pore sizes in the nanometer range, is a synthetic catalyst with a unique three-dimensional structure, making it highly selective for various reactions [4]. Its specific pore size allows for the selective transformation of smaller molecules while restricting larger ones, preventing unwanted side reactions [5]. HZSM-5 is a valuable catalyst due to its high activity, acidity, shape-selective catalysis, and thermal stability [6]. Its nanoscale features contribute to its excellent catalytic performance, making it a promising candidate for various nanocatalytic applications. It is extensively used in various industries, particularly in petroleum refining processes [7]. Additionally, HZSM-5 finds applications in biodiesel production [8], pharmaceutical synthesis [9], and environmental applications [10]. As advancements continue, HZSM-5 is expected to play an even more prominent role in sustainable and efficient chemical processes.

Pyrolysis has been investigated in literature as a route for thermal disposal of HDPE using thermogravimetric (TGA) technique in combination with kinetic analysis to better understand the reaction mechanism. To evaluate the kinetic parameters of the thermal decomposition of HDPE, model-free or isoconversional methods (e.g., Friedman [11], Flynn–Wall–Qzawa (FWO) [12], [13], Kissinger–Akahira–Sunose (KAS) [14], Sarink (STK) [15], and DAEM [16], [17]) and model-fitting or non-isoconversional methods (e.g., Coats–Redfern [18], Criado [19], and Arrhenius) can be utilized. Previous studies have shown that the activation energy can vary with conversion and the choice of model. For example, activation energy values in the range of 238–247 kJ/mol were reported using Friedman, FWO, and KAS isoconversional methods for pure HDPE with no catalyst [20]. Another study reported values of approximately 202 and 376 kJ mol^{-1} using KAS and Friedman methods, respectively [21]. We have investigated the catalytic pyrolysis of HDPE using HZSM-5 catalyst previously using three isoconversional methods (Friedman, FWO, and KAS) and two non-isoconversional methods

(Arrhenius and Coats–Redfern) [22]. We found that the incorporation of the HZSM-5 catalyst can significantly decrease the activation energy and the activation energy values vary with the HZSM-5/HDPE mass ratio.

In this study, we investigate activation energy of the catalytic thermal decomposition of HDPE over HZSM-5 catalyst using the STK and DAEM isoconversional methods. In addition, the advanced isoconversional method (AIC) developed by Vyazovkin et al. [23], is investigated which have been shown previously to provide a more accurate analysis of kinetic parameters for the degradation of various polymeric materials [24], [25]. Our results are then contrasted with our previous work. We show that the addition of HZSM-5 catalyst decreases the activation energy by 99 kJ mol^{-1} for a HZSM-5/HDPE ratio of 0.5 and using the STK and DAEM methods. Further increase in the HZSM-5/HDPE ratio to unity decreases the activation barrier to an average value of ~180 kJ mol^{-1}. The AIC method also predicts a significant decrease in the activation energy for the catalytic decomposition of HDPE by 79–105 kJ mol^{-1}. The activation energy values predicted here using the STK, DAEM, and AIC methods are in range with the previously published data using the Friedman, FWO, and KAS methods [22].

2. Materials and Methods

2.1. Experimental Procedure

A full description of the experimental procedure and materials characteristics can be found in our previous work [22]. Here, we summarize the TGA experimental setup. Samples of HDPE polymer were obtained from Ipoh SY Recycle Plastic, Malaysia. The HZSM-5 was purchased from Alfa Aesar in Ward Hill, Massachusetts, USA. Different catalyst to polymer weight ratios (0, 0.5, and 1.0) were used to prepare the test samples. The cracking studies were conducted under 50 ml/min N_2 as an inert gas with a Mettler Toledo TGA/SDTA851e analyzer (Polaris Parkway, Columbus, OH, United States). V7.01 STARe software was used to evaluate the results. In this experiment, heating rates of 5, 10, and 15 K/min were applied. For the pure HDPE with no catalyst, heating rates of 5, 10, and 40 K/min were used. To carry out the measurements, samples were heated from ambient temperature to 973 K using TGA equipment.

2.2. Kinetic Theory

The generalized kinetic rate equation (r) can be expressed in terms conversion (α) as:

$$r = \frac{d\alpha}{dt} = A \exp\left(-\frac{E}{RT}\right) f(\alpha) \tag{1}$$

where t is the time, A is the pre-exponential factor, E is the activation energy, R is the universal gas constant, and T is the temperature. The conversion is given by:

$$\alpha = \frac{w_o - w}{w_o - w_f} \tag{2}$$

where w_o, w_f, and w are the initial, final, and instantaneous weight of the sample, respectively. For non-isothermal methods, the heating rate (β) can be expressed as:

$$\beta = \frac{dT}{dt} \tag{3}$$

Combining Equation 3 and 1:

$$\frac{d\alpha}{dT} = \frac{A}{\beta} \exp\left(-\frac{E}{RT}\right) f(\alpha) \tag{4}$$

Integrating Equation 4 yields the following equation:

$$\int_0^{\alpha} \frac{d\alpha}{f(\alpha)} = g(\alpha) = \frac{A}{\beta}\int_0^{T} \exp\left(-\frac{E}{RT}\right) dT \qquad (5)$$

Substituting:

$$x = \frac{E}{RT} \qquad (6)$$

into Equation 5 yields:

$$g(\alpha) = \frac{AE}{\beta R}\int_x^{\infty} \frac{\exp(-x)}{x^2} dx = \frac{AE}{\beta R} p(x) \qquad (7)$$

where *p(x)* is the temperature integral expression and it does not have an analytical solution. However, it can be estimated using various kinetic models and approximations to evaluate the kinetic parameters (activation energy and pre-exponential factor). Three different model-free isoconversional methods have been employed here in this study (STK, DAEM, and AIC) as summarized in Table 1.

Table 1. A summary of the model-free isoconversional methods used in this study.

Method	Expression
STK [15]	$ln\left(\frac{\beta}{T^{1.92}}\right) = ln\left(\frac{AE}{Rg(x)}\right) - 1.000\frac{E}{RT}$
DAEM [16], [17]	$ln\left(\frac{\beta}{T^{2}}\right) = ln\left(\frac{AR}{E}\right) + 0.6075 - \frac{E}{RT}$
AIC [23]	$\phi(E_\alpha) = \sum_{i=1}^{n}\sum_{j\neq 1}^{n} \frac{I(E_\alpha, T_{\alpha,i})\beta_j}{I(E_\alpha, T_{\alpha,j})\beta_i} = 0$

3. Results and Discussion

3.1. TGA Analysis

Figure 1 provides insights into the HDPE decomposition process through TGA data analysis. The temperature-dependent mass loss (TGA curves) and its rate of change (DTG curves) are shown at different heating rates and catalyst-to-polymer ratios. An increase in the heating rate shifts both the mass loss and DTG curves rightward, indicating higher decomposition temperatures and a slower breakdown process. The addition of HZSM-5 catalyst significantly affects the reaction, causing both curves to shift leftward. This leftward shift signifies earlier and faster polymer degradation due to the catalytic cracking effect of HZSM-5. These catalytic cracking reactions occur within the nanoscale pores of HZSM-5, where the high surface area and unique pore structure promote the efficient breakdown of HDPE. Notably, even with the catalyst, the kinetic parameters derived from TGA capture a narrow temperature range around the peak decomposition rate, suggesting one major reaction region for catalytic cracking. The analysis also reveals that increasing the HZSM-5 to HDPE ratio generally reduces the peak temperature, while the influence of catalyst weight diminishes as the heating rate increases.

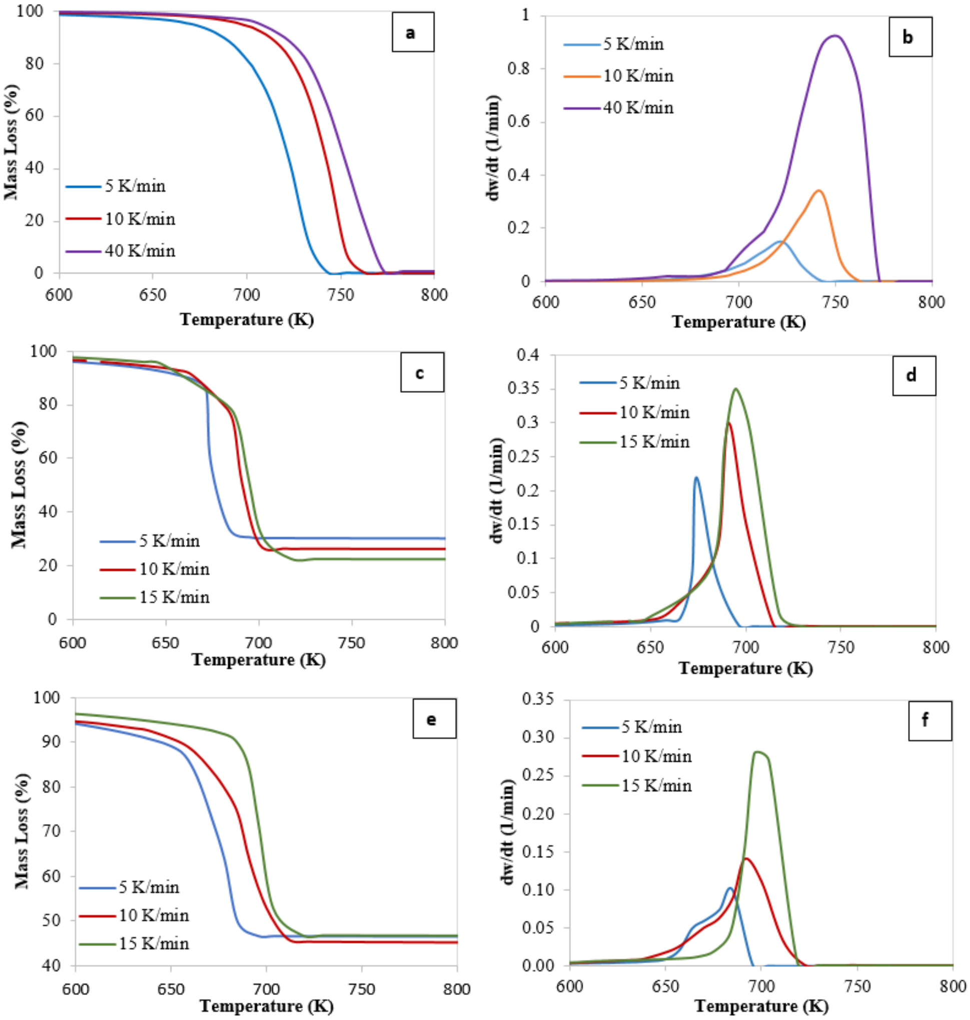

Figure 1. TGA and DTG curves of various HZSM-5 catalyst/HDPE polymer ratio using different heating rates. **(a)** TGA of HZSM-5/HDPE = 0, **(b)** DTG of HZSM-5/HDPE = 0, **(c)** TGA of HZSM-5/HDPE = 0.5, **(d)** DTG of HZSM-5/HDPE = 0.5, **(e)** TGA of HZSM-5/HDPE = 1, **(f)** DTG of HZSM-5/HDPE = 1. Adapted from Ref. [22].

Figure 2 shows the impact of increasing the HZSM-5 catalyst to HDPE ratio on the resulting mass loss and residue after catalytic cracking. The addition of HZSM-5 significantly reduces mass loss and increases the residual material. For example, at HZSM-5/HDPE ratios of 0.5 and 1, the mass loss is 74% and 54%, respectively, compared to nearly 99% for pure HDPE. This remaining mass is directly attributed to the presence of the HZSM-5 catalyst. Therefore, a higher catalyst loading leads to faster and more efficient HDPE decomposition. Importantly, the HZSM-5 catalyst can be recycled and regenerated for further use in catalytic cracking processes.

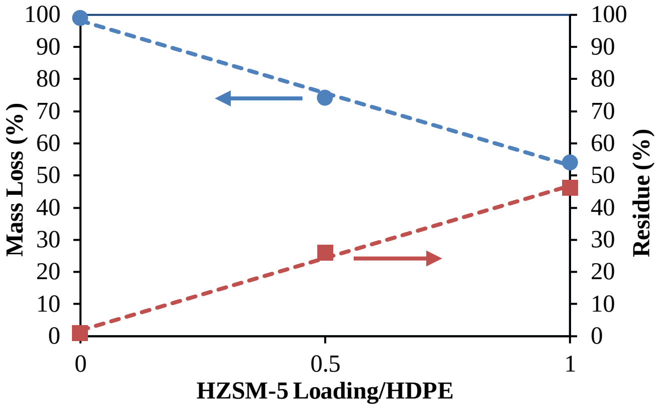

Figure 2. The influence of HZSM-5 catalyst to HDPE ratio with respect to mass loss and material residue. Adapted from Ref. [22].

3.2. Kinetic Analysis

In this section, we analyze the activation energy (E_a) at different conversions as a function of catalyst-to-polymer ratios, utilizing three model-free models (STK, DAEM, and AIC), as presented in Table 2. These results are subsequently compared with previously published data for HZSM-5/HDPE, employing other isoconversional models (Friedman, FWO, and KAS) [22]. A conversion (α) range of 0.5–0.8 was chosen, considering that lower conversions yield less accurate results, as previously demonstrated [26].

Table 2 provides the activation energy (E) and correlation coefficient (R^2) for the three models (STK, DAEM, and AIC) in the catalytic thermal decomposition of HDPE, using a catalyst-to-polymer ratio (HZSM-5/HDPE) of 0, 0.5, and 1. At a conversion of 0.5, using the STK model, activation energy values decrease with an increase in the HZSM-5/HDPE ratio, from 304 kJ mol^{-1} with no catalyst to 202 kJ mol^{-1} at HZSM-5/HDPE = 0.5, further dropping to 168 kJ mol^{-}1 at HZSM-5/HDPE = 1. This signifies that the addition of HZSM-5 catalyst rapidly reduces the activation barrier, enhancing the reaction rate. Increasing the conversion from 0.5 to 0.8 marginally affects the activation energy, except for HZSM-5/HDPE = 1, where the activation energy increases from 168 kJ mol^{-1} to 192 kJ mol^{-1}. On average, the STK model predicts a significant decrease in activation energy, from 295 kJ mol^{-1} in the absence of a catalyst to 196 kJ mol^{-1} at HZSM-5/HDPE = 0.5. A further increase in the HZSM-5/HDPE ratio to 1 slightly decreases the average activation energy by 15 kJ mol^{-1} to a value of 181 kJ mol^{-1} (Table 2). In all cases, the correlation coefficient (R^2) exceeds 0.9, affirming the reliability of this model in describing the kinetic parameters for the catalytic thermal decomposition of HDPE.

Table 2. Estimated activation energy values using the STK, DAEM, and AIC methods.

conversion	**HZSM-5/HDPE = 0**		**HZSM-5/HDPE = 0.5**		**HZSM-5/HDPE = 1.0**	
	E (kJ mol^{-1})	**R^2**	**E (kJ mol^{-1})**	**R^2**	**E (kJ mol^{-1})**	**R^2**
STK						
0.5	304	0.9963	202	0.9595	168	0.9997
0.6	302	0.9992	190	0.9666	182	0.9999
0.7	296	0.9999	197	0.9775	195	0.9970
0.8	289	0.9999	198	0.9724	192	0.9681
Average	**295**	**0.9984**	**196**	**0.9713**	**181**	**0.9735**
DAEM						
0.5	303	0.9963	202	0.9593	167	0.9997
0.6	301	0.9992	190	0.9665	182	0.9999
0.7	295	0.9999	196	0.9774	195	0.9970

0.8	289	0.9999	197	0.9800	191	0.9080
Average	**294**	**0.9984**	**195**	**0.9727**	**180**	**0.9614**
AIC						
0.5	296	–	199	–	173	–
0.6	299	–	214	–	187	–
0.7	300	–	219	–	197	–
0.8	277	–	223	–	195	–
Average	**293**	–	**214**	–	**188**	–

The DAEM model exhibits results closely resembling the STK model, as anticipated due to the similarity in the mathematical expressions of these two models (Table 1). The activation energy values obtained from the DAEM model are nearly identical to those derived from the STK model, as illustrated in Table 2. Figure 3 shows the activation energy values as a function of conversion for all three models examined in this study. Both the STK and DAEM models demonstrate identical trends in activation energy as a function of conversion for all HZSM-5/HDPE ratios investigated. In comparison, the average activation energy values obtained using the STK and DAEM models align closely with those derived previously using other models (Friedman, FWO, and KAS) [22]. For instance, at HZSM-5/HDPE = 0.5, these three models collectively predict an average activation energy range of 186–196 kJ mol^{-1}, consistent with the values obtained in this study (195–196 kJ mol^{-1}).

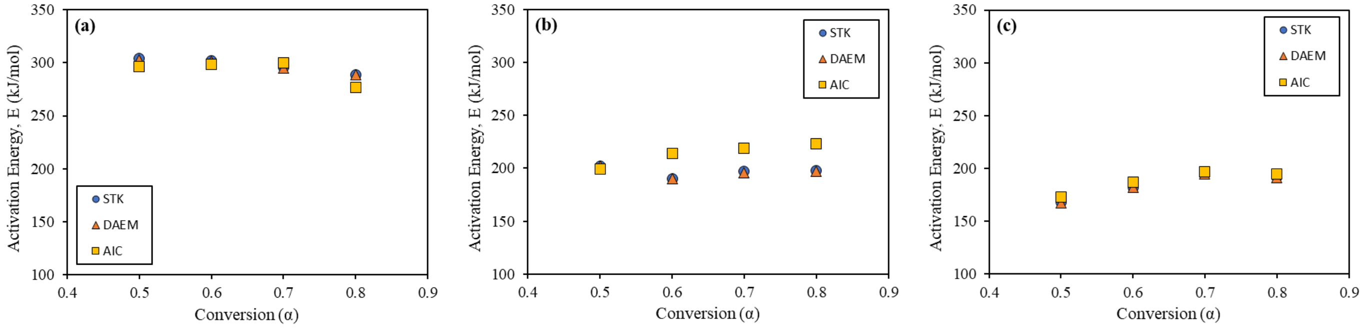

Figure 3. The activation energy (E) as a function of conversion (α) for (**a**) HZSM-5/HDPE = 0, (**b**) HZSM-5/HDPE = 0.5, and (**c**) HZSM-5/HDPE = 1.

The advanced isoconversional method (AIC), developed by Vyazovkin et al. [23], has demonstrated superior accuracy in analyzing kinetic parameters, as evidenced in previous studies involving low-density polyethylene (LDPE), polypropylene (PP), polylactic acid (PLA), and polyethylene terephthalate (PET) [24], [25]. It employs sophisticated numerical integration of the TGA data and accurately predicts the activation energy as a function conversion. For pure HDPE, the AIC method predicts an activation energy in the range of 296–300 kJ mol^{-1} at 0.5–0.7 conversion, before it drops to 277 kJ mol^{-1} at 0.8 conversion (Table 2). The average activation energy for this sample is 293 kJ mol^{-1}, consistent with the average values obtained by both STK and DAEM methods (294–295 kJ mol^{-1}). The addition of HZSM-5 decreased the average activation energy by 79 kJ mol^{-1}, yielding a value of 214 kJ mol^{-1} at HZSM-5/HDPE = 0.5, compared to an average value of 195–196 kJ mol^{-1} for the STK and DAEM methods. However, in contrast to the STK and DAEM models, the activation energy values predicted by the AIC method increase with increasing the conversion (Figure 3b) from 199 kJ mol^{-1} to 223 kJ mol^{-1} for 0.5–0.8 conversion. At the maximum catalyst-to-polymer ratio examined here (HZSM-5/HDPE = 1), the average activation energy predicted by the AIC method further decreases to 188 kJ mol^{-1}, and we notice a similar increasing trend for the activation energy as a function of conversion (Figure 3c).

In general, all three methods predict an average activation energy in the range of 293–295 kJ mol^{-1} for the pure catalyst, 195–214 kJ mol^{-1} for HZSM-5/HDPE = 0.5, and 180–188 kJ mol^{-1} for HZSM-5/HDPE = 1. Thus, increasing the catalyst-to-polymer ratio indeed decreases the activation energy for the thermal decomposition of HDPE. The relatively narrow range of these average activation energy values also suggests the applicability of all three models examined in this study.

4. Conclusions

This study investigates the catalytic pyrolysis of HDPE using HZSM-5 catalyst, emphasizing the impact of catalyst-to-polymer ratios. Employing three model-free methods—Sarink (STK), DAEM, and the advanced isoconversional method (AIC)—we conducted a detailed analysis for the activation energy as a function of conversion ranging from 0.5 to 0.8 for improved accuracy. Results obtained from STK and DAEM models consistently exhibited a substantial decrease in activation energy with increasing HZSM-5/HDPE ratio. The STK model indicated a remarkable reduction in the average activation energy from 295 kJ mol^{-1} (no catalyst) to 181 kJ mol^{-1} at HZSM-5/HDPE = 1. The DAEM model corroborated these findings, aligning with the mathematical similarities between the two models. These trends were further confirmed by comparison with previously published data utilizing other isoconversional models (Friedman, FWO, and KAS). The AIC method, known for its better accuracy in predicting kinetic parameters, predicts a substantial decrease in activation energy, ranging from 79 to 105 kJ mol^{-1}. The consistent range of average activation energy values across the three methods examined here suggest their applicability and reliability in describing the kinetic parameters of this catalytic process. This study provides valuable insights into the nanocatalytic pyrolysis of HDPE using HZSM-5, guiding the development of environmentally sustainable polymer disposal methods.

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