International Journal of Nanoelectronics and Materials
IJNeaM
ISSN 1985-5761 | E-ISSN 2232-1535

Reduction of iron oxide with polypropylene and polyurethane by thermogravimetric analysis for ironmaking processing

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Received 10 May 2026, Revised 25 May 2026, Accepted 5 June 2026

ABSTRACT

This study investigates the reduction kinetics and thermal degradation behavior of hematite (Fe_2O_3) mixed with recycled polypropylene (PP) and polyurethane (PU) using non-isothermal thermogravimetric analysis (TGA). Kinetic parameters were derived via the Coats-Redfern solid-state reaction model. Our findings demonstrate that PP and PU exhibit fundamentally different degradation mechanisms when utilized as reductants. Pure PP degrades in a sharp, single step around 480–490°C due to random chain scission, whereas PU decomposition is a complex multi-stage process initiating at lower temperatures (250–280°C) but ultimately leaving a resilient char residue. Kinetic evaluations of the polymer-iron oxide composites reveal that the PP composite undergoes rapid devolatilization with an activation energy of 192 kJ/mol, effectively promoting early indirect reduction through the rapid release of light hydrocarbons and hydrogen. Conversely, the PU composite exhibits a pronounced dual-stage kinetic profile: an initial low-energy devolatilization (134.3 kJ/mol) followed by a high-energy barrier (335.9 kJ/mol) representing carbothermic reduction between 850°C and 950°C. The delayed high-temperature reactivity and stable char formation characteristic of PU make it highly advantageous as a resilient internal reductant, capable of supporting rapid metallization. Ultimately, both polymer wastes offer distinct kinetic advantages, reinforcing their viability as strategic metallurgical resources for green ironmaking.

Keywords: *Reduction kinetics, Iron oxide, Polypropylene, Polyurethane, Thermogravimetry*

1. INTRODUCTION

The iron and steel industry is currently reducing the coal-based reduction processes by using alternative polymeric materials such as waste styrene-butadiene rubber (SBR), high-density polyethylene (HDPE), polypropylene (PP), polycarbonate (PC), polyethylene terephthalate (PET), polyethylene (PE), and acrylonitrile butadiene styrene (ABS) [1–4]. Polymers have emerged as promising substitutes due to their high calorific value with high hydrogen content, which facilitates faster reduction kinetics compared to conventional coal/coke. Polymer-based reductants exhibit significantly higher reduction rates and reactivity at lower temperatures than conventional coke or coal [5]. Instead of relying on the slower Boudouard reaction ($C + CO_2 \rightarrow 2CO$) for reducing gas generation, polymers decompose quickly via thermal cracking at temperatures of 350–500°C [6]. This process releases reactive volatile compounds, including hydrogen (H_2) and light hydrocarbons (C_nH_m), which initiates the reduction of hematite (Fe_2O_3) at temperatures lower than those required for coal gasification [7, 8]. The reduction of iron oxide in the iron oxide–polyethylene–graphite composite proceeds by polyethylene-derived reducing agents below 817°C when the composite is heated at a low heating rate, with only small temperature differences between the inside and the outside of the composite observed by Murakami and Kasai [9]. Dehydrated pisolitic ore, which exhibits a high specific surface area, effectively promotes reduction when polyethylene is employed as a reducing agent, resulting in reduced reduction temperatures [9]. In other research, HDPE containing minimal or no fixed carbon, methane created during the first heating phase was transformed into nascent carbon and hydrogen, with the process catalysed by freshly synthesized metallic iron [10]. When PP was applied as a carbon reductant at 900°C, the transformation of hematite into magnetite was observed with a few morphological changes. One can only notice where a more continuous structure of solids with smaller constituent grains is less visible. The iron phase is quite continuous and was expected to be wustite with the development of a porous structure [4]. Moreover, some researchers reported that partial replacement of coke using polymers could reduce the operation temperature by about 100 to 200°C by saving up to 60 GJ/t [5]. Thus, polymer wastes have a large potential as reductants for iron ores because their major elements are hydrogen and carbon. If these wastes could be effectively used in the ironmaking process, the total CO_2 emissions caused by fossil fuels would decrease because a significant amount of plastic waste is still simply incinerated without effective heat recovery.

The kinetics reduction using polymers with iron oxide was discovered that using biomass and polymer-derived carbon

successfully lowers the apparent activation energy (Ea) during the reaction between iron oxide [3, 11, 12]. This kinetic advantage derives from the initial action of hydrogen-rich gases, which penetrate the iron ore lattice more efficiently than carbon monoxide alone [7, 13]. Thermogravimetric Analysis (TGA) is one of the methods for determining the multi-step reduction sequence ($Fe_2O_3 \rightarrow e_3O_4 \rightarrow FeO \rightarrow Fe$) and clarifying these reaction processes. The isoconversional Friedman technique was employed to determine the activation energy as a function of fractional mass loss, revealing that the activation energy for PP is not constant but rises with conversion, ranging from 105 to 150 kJ/mol [14]. While the thermal degradation of PU was investigated using a one-step reaction, Shufen's approach simplified the activation energy to 155 kJ/mol [15]. Current kinetic modelling, including model-fitting techniques like Coats-Redfern, facilitates the identification of rate-controlling phases of polymer composites with iron oxide, which generally shift from chemical reaction control at the interface to diffusion-limited regimes as the activation energies increase [7, 16]. However, chemical kinetics are intrinsically linked to the physical evolution of the composite material. The escape of polymer volatiles during the heating cycle dramatically alters the pellets' internal structure.

From the above literature, it was expected that the reduction performance would increase when using polymers as carbon reductants. Thus, this study aims to provide a comprehensive investigation into the reduction behavior of iron oxide using polymers from PP and PU. By integrating TGA-based kinetic studies, this research seeks to contribute to the development of a circular economy by transforming non-recyclable plastic waste into a strategic carbon resource for the green ironmaking process.

2. MATERIALS AND METHODS

In this study, reagent-grade iron oxide (HmbG Chemicals) containing 96 wt.% hematite (Fe_2O_3) was utilized. In addition, the iron oxide included SiO_2 (0.40 wt.%), CaO (0.02 wt.%), and MnO (0.02 wt.%). Recycled polymers from PP and PU as carbon reductants were supplied by SLT Plastic Sdn. Bhd. Prior to thermogravimetric analysis, the recycled PP and PU materials were ground into powder form with particle sizes ranging from 45 to 63 µm.

The proximate analysis of the carbonaceous material was performed following ASTM D3172–3175 standards to measure moisture content, volatile matter, ash, and fixed carbon content. The ultimate or elemental analysis, determining carbon, hydrogen, nitrogen, and oxygen content, was carried out using a Perkin Elmer 2400 Series II elemental analyser. The analysis was presented in Table 1. The volatile content for both carbons from PP and PU in the ranges of 93.77 wt.% and 82.46 wt.%, respectively. The high carbon content from PP (84.06 wt.%) was expected to increase the performance reduction compared to PU (63.89 wt.%).

2.1. Thermogravimetric Analysis

A Perkin Elmer Co., Waltham, Massachusetts, USA, 8000 series thermogravimetric was used to pyrolyze PP, PU, PP mixed iron oxide, and PU mixed iron oxide at heating rates of 10°C/min under non-isothermal conditions. The mixed ratio for iron oxide to polymers was set at 1:1. The mixed sample was labelled as PP + Iron Oxide and PU + Iron Oxide. About 10 mg of polymers and mixed powder samples were utilized in all TGA studies in an inert environment of pure N_2 (99.999%) gas flowing at 20 ml/min.

2.2. Reduction in Kinetics and Activation Energy Estimation

The Coats and Redfern (CR) method was selected to calculate the reduction kinetics for non-isothermal conditions. The fraction converted (α) defines the kinetics (Equation (1)). This will represent how extensive the degradation reaction has occurred at a given temperature and will be directly obtained from the raw weight data.

$$\alpha = \frac{W_0 - W_t}{W_0 - W_f} \tag{1}$$

In this formula, W_0 stands for the initial weight of PP, W_t represents the weight of PP when prepared at a certain temperature (T), and W_f denotes the final residual (unreacted) weight of PP after the degradation step is completed (the data shows that it stabilizes around 45% residual weight due to unreacted iron oxide, based on the graph data).

During the thermal breakdown of polyolefins in PP/PU, the predominant mechanism for the degradation will be random chain scission.) For example, in modelling kinetics, it is reasonable to assume that $n = 1$ (which is defined as a ratio of 1) as a reference point to represent the behavior of this process. When using the Coats-Redfern Equation, assuming $n = 1$ creates the following linear relationship (Equation (2)):

$$\ln\left(-\frac{\ln(1-\alpha)}{T^2}\right) = \ln\left(\frac{AR}{\beta E_a}\left(1 - \frac{2RT}{E_a}\right)\right) - \frac{E_a}{RT} \tag{2}$$

Table 1. Proximate and ultimate analysis of carbon reductants from PP and PU

Ultimate (wt.%)			Proximate (wt.%)		
Composition	**PP**	**PU**	**Composition**	**PP**	**PU**
Carbon	84.06	63.89	Moisture	0.16	1.86
Hydrogen	14.69	4.39	Ash	4.45	5.41
Nitrogen	0.40	6.67	Volatile matter	93.77	82.46
Sulphur	0.21	-	Fixed carbon	1.62	9.85
Oxygen	0.28	15.79			

$2RT/E_a$ is significantly smaller than 1 for high-temperature solid-state reactions; thus, the equation simplifies to a standard linear form $y = mx + c$) as in Equation (3):

$$\ln\left(-\frac{\ln(1-\alpha)}{T^2}\right) \approx \ln\left(\frac{AR}{\beta E_a}\right) - \frac{E_a}{RT} \quad (3)$$

where: T is the absolute temperature (in Kelvin). R is the universal gas constant (8.314 J/(mol·K)). β is the heating rate (assumed here to be a standard 10°C/min or 0.167 K/s based on typical runs).

3. RESULTS AND DISCUSSION

3.1. Thermal Degradation Analysis of Carbon Reductants

The provided thermogravimetric (TG) and derivative thermogravimetric (DTG) thermograms illustrate the contrasting thermal degradation behaviours of PP and PU in Figure 1. The TG curve for PP demonstrates excellent initial thermal stability, showing the degradation occurring at a temperature of approximately 400°C. From the figure, PP showed the single-step weight loss, evidenced by an exceptionally sharp DTG peak between 480°C and 490°C. The thermal behavior aligns with the random chain scission of PP's saturated hydrocarbon backbone, which contains the high-energy carbon bonds that require significant thermal input to break before rapidly propagating into volatile monomeric and oligomeric units. Consequently, PP leaves a negligible residual weight of nearly 0% to 2% at 600°C [17].

In contrast, the thermal degradation of PU is a complex process consisting of multiple steps and occurring at much lower temperatures than those at which polyolefin thermal degradation occurs. PU thermal degradation begins to take place at approximately 250–280°C, as shown by the beginning of weight loss at these temperatures (Figure 1). The polyurethane DTG profile shows a single, sharp peak in the decomposition rate at approximately 320–340°C, which is attributed to the initial breakdown of the relatively thermally unstable urethane linkages present in the PU hard segments. The secondary degradation phase of PU continues to occur up to about 450°C; the secondary degradation phase is due to the thermal breakdown of the polyether or polyester soft segments into smaller volatile fragments [18, 19]. PU degradation produces a significant amount of solid residue (approximately 10–12% residue) on degradation, compared to the low amount of solid residue produced on degradation of hydrocarbons (e.g., polyolefins). Most of the solid residues produced from polyurethane degradation are caused by the cross-linking and carbonization of the isocyanate-derived segments into a thermally stable char. Moreover, comparative analysis highlights the structural differences between a simple polyolefin and a segmented condensation polymer. While PP exhibits higher initial thermal resistance before volatilizing entirely without charring, the urethane bond in PU acts as a weak link that initiates decomposition much earlier. However, the intrinsic char-forming capability of PU, which is absent in standard PP, suggests differing combustion profiles and serves as a fundamental baseline for designing flame-retardant materials [15].

For reduction reactions, volatile components and solid residues in the raw materials (PP and PU) are the main factors affecting the yield of liquid oil during pyrolysis (Table 1). A higher proportion of volatile components is conducive to the formation of liquid oil, while a higher proportion of solid residues reduces the yield and leads to increased gas generation and heat production [8]. Analysis shows that the content of volatile components in all plastics is significantly higher than the relatively low content of solid residues, thus suitable for iron reduction processing. Catalysts play a crucial role, such as iron oxides, hydroxides, fluid catalytic cracking (FCC), selective zeolites, and synthetic zeolites, improving the quality of pyrolysis products, reducing reaction temperature, and shortening reaction time [15, 20]. Catalysts can effectively increase the proportion of decomposition reactions, increase the gas yield, and reduce the liquid oil yield [7]. The long-chain carbon compounds physically combine with the catalyst or further decompose into short-chain carbon compounds, where the catalytic activity of the catalyst is influenced by parameters such as pore size, pore volume, and acidity.

3.2. Thermal Degradation Analysis from the Reduction of PP/PU with Iron Oxides

Figure 2 presents TG/DTG profiles that indicate that, in general, the PP + Iron oxide composites and PU + Iron oxide composites show fundamentally different thermal deterioration characteristics. Therefore, these differences are significant for how each of the two types of composites may be used to reduce iron oxide during iron production. Both types of composites begin to lose mass around the 280°C to 300°C range; however, for the PP + Iron Oxide, mass loss occurs dramatically at these temperatures and reaches completion in the primary mass loss temperature range of 450°C. This fact is also indicated by the singular peak of the DTG curve at this temperature. The thermogravimetric analysis indicates that PP and PU have

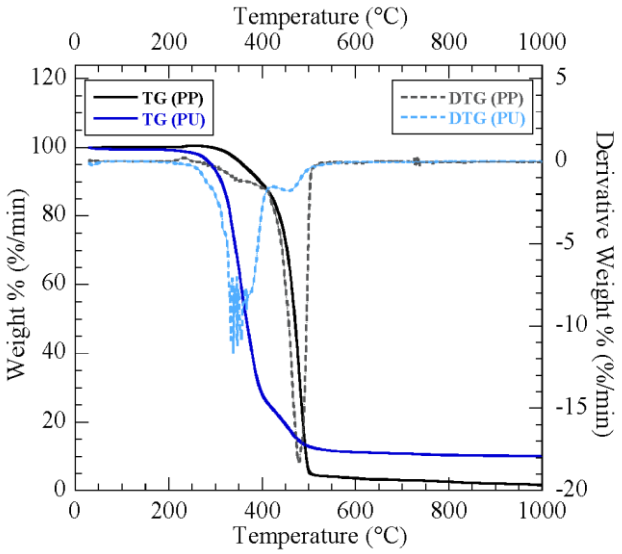

Figure 1. The TG/DTG analysis of PP and PU

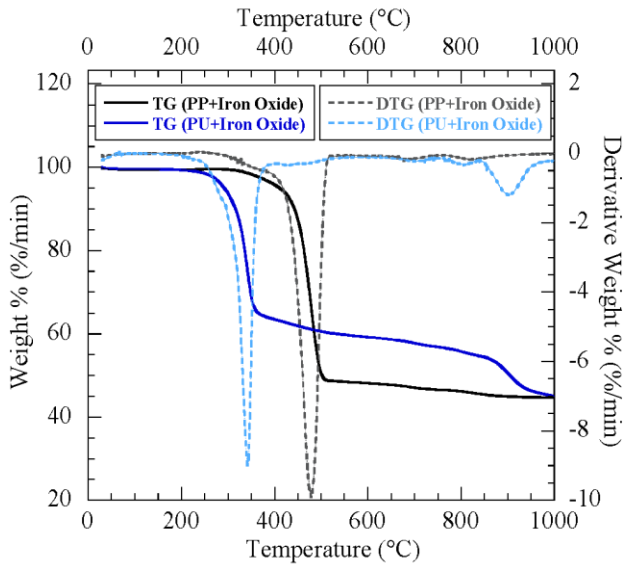

Figure 2. TG/DTG analysis of PP + Iron Oxide and PU + Iron Oxide

quite different pyrolytic characteristics when they are heated with iron oxide, which shows that they are possible replacements for waste plastics as an equivalent carbon source in iron and steelmaking [4, 21].

From Figure 2, the PU + Iron oxide system experiences a volume of mass loss over a much greater temperature range between 592°C to 595°C, and that concludes at approximately 700–900°C; and this volume of mass loss occurs over multiple stages, indicating the PU has a much more complex/multi-segmented backbone than that of PP due to the PU molecule being broken down by multiple mechanisms (e.g. - urethane molecular dissociation, soft-segment decomposition and the oxidation of residual char). The thermal behavior of the two types of composites will ultimately affect how effectively each composite performs as a reductant.

The continuous mass loss observed during the thermogravimetric analysis of the polymer-iron oxide composites reflects two concurrent mechanisms: the thermal devolatilization of the polymer reductants and the progressive removal of oxygen from the iron ore lattice [7], [9]. As the polymers decompose, they release highly reactive volatiles, including hydrogen and carbon monoxide, which initiate the sequential indirect reduction of the ore ($Fe_2O_3 \rightarrow Fe_3O_4 \rightarrow FeO \rightarrow Fe$ [7, 13]. In the PP composite, this mass reduction is sharp and reaches completion near 450°C due to rapid, single-step chain scission, which provides an early, concentrated burst of reducing gases [17]. Conversely, the PU composite exhibits a prolonged, multi-stage mass loss extending between 700°C and 900°C [18, 19]. This broader mass loss profile is characterized by the initial breakdown of urethane linkages, followed by a highly endothermic carbothermic reduction phase where PU-derived stable char continuously strips oxygen from the iron oxide lattice at elevated temperatures [3]. Ultimately, the mass loss stabilizes, leaving approximately 45% residual weight, which represents the remaining reduced iron phases and any unreacted iron oxide after the transient gases and stripped oxygen have completely exited the system.

Additionally, it is important to note that the dynamic conditions of the thermogravimetric analyser significantly influence the observed high-temperature reduction behaviours. Because the analysis employs a continuous nitrogen flow (20 ml/min), the volatile hydrocarbons and hydrogen generated by the complete degradation of PP at 400–500°C are rapidly swept away. Lacking the necessary residence time to remain in contact with the solid iron oxide, these transient gases cannot participate in further high-temperature indirect reduction between 600°C and 900°C, causing the PP composite's mass loss to plateau early. In contrast, PU forms a resilient carbonized char during its primary degradation phase [15]. This solid char remains trapped within the composite matrix, enabling it to actively drive the endothermic carbothermic reduction of the iron oxide at elevated temperatures (700–950°C), as accurately reflected by the prolonged mass loss curve in Figure 2.

3.3. Kinetics and Activation Energy

This study utilizes the Coats-Redfern (CR) model-fitting technique compared to the model-free isoconversional approaches, such as the Friedman method, to demonstrate how activation energy fluctuates with fractional mass loss [14]. The fundamental difference lies in the kinetic assumption: isoconversional methods calculate E_a without assuming a specific reaction model, capturing shifting mechanisms over time, whereas the CR method calculates a single apparent Ea based on a predefined reaction model. For this study, the first-order reaction model ($n = 1$) was intentionally selected because the predominant thermal breakdown mechanism for polyolefins and the initial degradation of polyurethane occurs via random chain scission, a process that fundamentally obeys first-order kinetics [17]. Deriving a single, representative activation energy for distinct thermal stages, specifically devolatilization versus high temperature carbothermic reduction, provides a more practical macroscopic baseline for comparing these polymers as bulk metallurgical reductants. The high validity of this assumed kinetic model is mathematically confirmed by the excellent linear correlation coefficients ($R^2 > 0.98$) obtained across all calculated stages in Figures 3 and 4, ensuring the reliability of the derived kinetic parameters.

The thermogravimetric analysis for reduction kinetics (TG and DTG) for both carbon reductants (PP and PU) was further analysed by the CR method. The solid-state reaction models were fitted to Equation (2), and linear fits were performed on the plots in Figure 3. From the results, the R^2 value close to 1.0 indicates a perfect linear correlation for PP (R^2 = 0.9996), while PU had R^2 = 0.9974 for an early-stage breakdown of the urethane linkages, which adheres well to the first-order chain scission model, and secondary degradation had R^2 = 0.9973. The activation energy E_a for the PP was 192 kJ/mol. It should be noted that the calculated activation energy for PP devolatilization using the CR method (192–197 kJ/mol) is higher than the 105–

150 kJ/mol range previously reported using the isoconversional Friedman method [14]. The Friedman method calculates a fluctuating activation energy that often captures the lower energy thresholds of early-stage degradation, such as the breaking of weak structural links. In contrast, the CR method assumes a specific reaction model to yield a single, overarching apparent activation energy for the primary rate-controlling step. For PP, this dominant step is the random homolytic scission of the main carbon-carbon (C–C) aliphatic backbone. The theoretical bond dissociation energy required for the cleavage of these robust aliphatic chains typically ranges from 170 to 220 kJ/mol [17]. Therefore, the CR-derived values of ~195 kJ/mol are thermodynamically reflected in the bulk macroscopic energy barrier required to fully devolatilize the polymer into reactive reducing gases [18].

It is noteworthy that the calculated activation energy for the PP + Iron oxide composite (197 kJ/mol) is nearly identical to that of pure PP (192 kJ/mol). This negligible difference indicates that the solid iron oxide particles do not chemically alter or catalyse the primary degradation pathway of the polymer. Because PP is a non-polar, saturated polyolefin, its thermal degradation relies entirely on the homolytic random chain scission of strong carbon-carbon bonds, a process dictated by intrinsic thermal energy rather than interfacial reactions with the ore. During this phase, the iron oxide acts primarily as an inert physical component; its role in the reduction sequence only begins after the PP has fully devolatilized and released reactive hydrogen and light hydrocarbons into the surrounding matrix.

Compared to PP, the difference between the two calculated activation energies (117 kJ/mol and 436 kJ/mol) elucidates PU's behavior as a reductant (Figure 3). The low activation energy indicates that early devolatilization is readily initiated, leading to rapid release of gaseous reductants at lower temperatures. The significant energy barrier for E_a (436 kJ/mol) is characteristic of carbothermic reduction. The removal of oxygen from an iron oxide lattice using solid carbon is a highly endothermic process that requires substantial thermal energy [9]. These results showed that PU is able to react at high temperatures of 850°C; thus, the reaction transformed directly into metallic iron from wustite [4].

The calculated R^2 values are significantly high (ranging from 0.9873 (PP+ Iron Oxide) to 0.9962 (PU + Iron oxide), and the Coats-Redfern equation is successfully acceptable (Figure 4). The high linearity proves that the initial assumption of first-order kinetics was correct. Figure 4 also represents the calculated energy and kinetics value of reduction from PP + Iron oxide, which aligns perfectly with the expected thermodynamic barrier for homolytic carbon-carbon bond cleavage in polyolefins. The E_a was 197 kJ/mol, showing the rapid devolatilization at 400°C. PP, as a carbon reductant, performed almost instantaneously with a kinetic force of complete devolatilization with light hydrocarbons, hydrogen, and carbon monoxide, promoting indirect reduction compared to PU + Iron oxide.

The contrast between these two calculated activation energies (134.3 kJ/mol vs. 335.9 kJ/mol) effectively quantifies the dual-action utility of PU as a carbon reductant when reacted with iron oxide (Figure 4). The very low temperature barrier for Ea indicates that early devolatilization is readily achieved, while higher energy is required for the carbothermic reduction, which starts at 850-950°C. The delay from the reactivity at high temperature caused by the Boudouard reactions, and represents PU, was highly advantageous to be resilient internal reductants in carbon-composite agglomerates in direct reduction ironmaking [3]. PU char was able to provide a highly concentrated reducing atmosphere at high temperatures, which is able to support rapid iron metallization compared to PP.

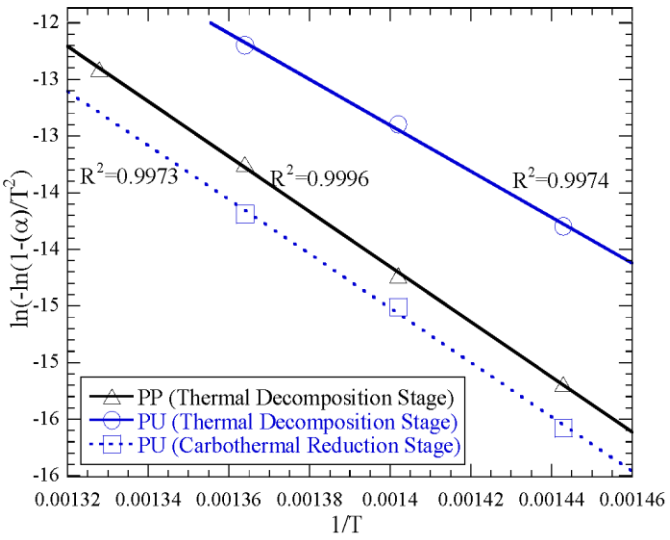

Figure 3. $\ln(-\ln(1-\alpha)/T^2$ versus $1/T$ plot with correlation coefficient R^2 using the CR method for PP and PU.

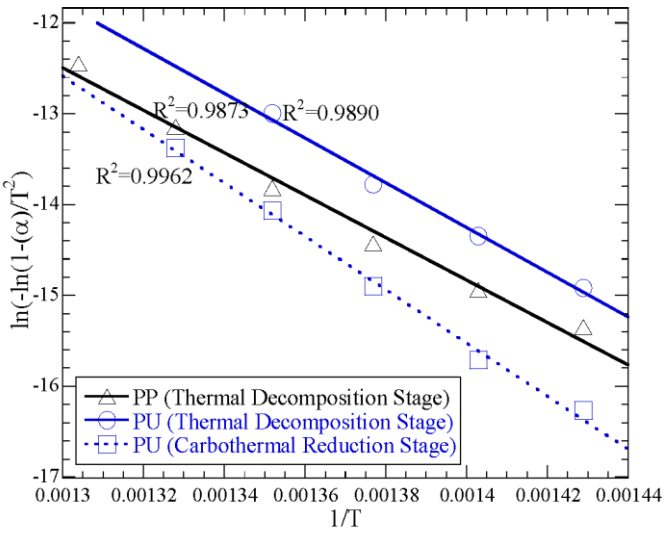

Figure 4. $\ln(-\ln(1-\alpha)/T^2$ versus $1/T$ plot with correlation coefficient R^2 using the CR method for PP + Iron oxide and PU + Iron Oxide.

From the above investigations, the current thermogravimetric and kinetic analyses establish a vital fundamental baseline for the reduction behavior of PP and PU composites, bridging the gap to practical industrial application that requires scaling up the experimental parameters. Future work should focus on evaluating the reduction performance of these polymer-iron oxide composites in pilot-scale reactors or under simulated industrial conditions, such as those found in blast furnaces or electric arc furnace (EAF) steelmaking [5]. Testing larger composite samples under significantly higher heating rates will be critical to accurately evaluating macroscopic mass and heat transfer limitations, as well as the realistic reaction efficiency of transient volatile reducing gases within a bulk system [13].

4. CONCLUSIONS

Based on the thermogravimetric and kinetic evaluations of polymer-iron oxide reduction, the following key insights were established:

(1) Polypropylene (PP) decomposes via a rapid, single-step process, exhibiting an activation energy of 192 kJ/mol. Polyurethane (PU), however, undergoes multi-stage degradation, which generates a stable carbonized char.
(2) PP + Iron oxide demonstrates highly efficient, single-stage devolatilization with an activation energy of 197 kJ/mol due to the rapid release of hydrogen-rich volatiles near 400 °C, while PU + Iron oxide demonstrates a highly functional dual-action mechanism. It releases initial reducing gases at a relatively low energy threshold (134.3 kJ/mol), while the subsequent carbothermic reduction phase demands a much higher activation energy (335.9 kJ/mol) at elevated temperatures of 850–950 °C, resulting in stable char that sustains a concentrated reducing atmosphere that drives rapid iron metallization at peak processing temperatures far better than PP.
(3) Both PP and PU have high carbon and hydrogen contents, necessary to replace conventional fossil fuels in ironmaking and lower the global carbon emissions.

ACKNOWLEDGMENTS

We extend our thanks to the Research Management and Innovation Center (RMIC), as well as the Faculty of Mechanical Engineering Technology and the Faculty of Chemical Engineering Technology at Universiti Malaysia Perlis (UniMAP), for the facilities they have made available to us.

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