

Thermal and kinetic analysis-guided selection of initial pyrolysis temperature for carbon nanotube synthesis from post-consumer recycled polypropylene

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ABSTRACT

The rapid escalation of post-consumer recycled polypropylene (PCR-PP) waste has raised environmental concerns and continues to exhaust municipal landfill capacity. Converting this plastic waste into high-value functional carbon materials is therefore necessary. This study investigates the thermal degradation behaviour of FeCp₂-catalysed PCR-PP using TGA and analyses its kinetics using the Coats-Redfern method. The thermal and kinetic data were used to identify the decomposition window and guide the selection of the initial pyrolysis temperature. CNTs were synthesised using a single-zone horizontal tube furnace with a sequential two-step heating protocol, comprising initial pyrolysis at the selected temperature followed by CNT growth at 900 °C. The products were characterised using SEM, XRD and Raman spectroscopy. The thermal decomposition of FeCp₂-catalysed PCR-PP was best described by an apparent first-order kinetic model with an activation energy (E_a) of 160.17 kJ/mol. Initial pyrolysis at a representative temperature of 400 °C generated carbon precursors to yield bundles of aligned, graphitic CNTs. The integrated Raman I_D/I_G ratio of 0.69 indicated relative structural ordering. The gross solid-product yield was 1.84 wt%, showing the need for further optimisation of carbon deposition and product recovery. Practically, the identified decomposition window and apparent activation energy provide baseline thermal and kinetic data for optimising initial heating conditions and hydrocarbon precursor generation in the single-zone PCR-PP to CNT process. This study integrates thermal analysis and kinetic evaluation into a workflow for CNT synthesis from PCR-PP.

Keywords: Post-consumer recycled polypropylene (PCR-PP); Carbon nanotubes; Sequential two-step heating protocol; Single-zone furnace, Thermogravimetric analysis, Coats-Redfern kinetics

1. INTRODUCTION

Approximately 318 million tonnes of plastic resin are produced annually due to the heavy utilisation of this material in packaging, which causes the generation of plastic waste globally [1]. Plastic waste is categorised into seven main groups based on the International Resin Identification Code (RIC) system. These include Code 1: Polyethylene Terephthalate (PET/PETE), Code 2: High-Density Polyethylene (HDPE), Code 3: Polyvinyl Chloride (PVC), Code 4: Low-Density Polyethylene (LDPE), Code 5: Polypropylene (PP), Code 6: Polystyrene/Styrofoam (PS) and Code 7: Others [2]. Among these, Code 5 (PP) is commonly used in manufacturing microwave-safe hot food takeaway containers, yoghurt cups, bottle caps, prescription medicine bottles and diaper liners.

The primary challenge with PP is its low recyclability. Although it is frequently sorted for reuse in automotive parts or outdoor equipment, sorting post-consumer food-grade PP is highly complex and economically demanding.

Consequently, this waste often ends up compressed in landfills. Furthermore, due to its highly stable structure and hydrophobic carbon-carbon backbone, natural degradation is virtually impossible. It takes decades to centuries under environmental conditions, for PP to decompose, slowly breaking down into microplastics [3]. These microplastics tend to absorb toxic chemical pollutants, such as pesticides and heavy metals, from surrounding water and soil. They are also easily ingested by marine life, birds and soil organisms. This ingestion not only blocks the digestive tracts of these animals but also, once absorbed, severely contaminates the food chain leading to humans [4]. Given these hazardous environment impacts, plastic waste valorisation has become imperative. PP is identified as a highly viable feedstock for conversion into value-added products, provided that its utilisation is strategically and systematically planned. Carbon nanotubes (CNTs) consist of hexagonal C-C structural layers rolled into hollow tubes. CNTs can be categorised as single-walled CNTs, double-walled CNTs, tripled-walled CNTs and multi-walled CNTs [5]. Generally, CNTs possess outstanding mechanical

(Young's modulus up to 1.8 TPa) [6], electrical (10^7 S/m) [7], and thermal (13,000 W/m.K) [8] properties. Due to these unique characteristics, they are applied in a wide range of applications such as composites [9], energy storage [10], sensors [11], and catalyst supports [12]. Since CNTs are conventionally synthesised from hydrocarbon-based precursors, extensive research has been conducted on alternative carbon sources to avoid relying solely on typical chemical sources such as methane [13], CO₂ [14] and liquid hydrocarbons or oxygenated hydrocarbons such as alcohols [15] and acetone [16]. Waste plastics are known to have a high carbon content, thereby offering the potential to significantly reduce feedstock costs. This approach represents a dual-benefit solution to both waste management and CNT synthesis.

Among these waste plastics, post-consumer polypropylene (PCR-PP) is rich in carbon and hydrogen. During thermal degradation, it yields volatile hydrocarbons, including methane and light C₂-C₅ hydrocarbons, making it a highly suitable precursor for CNT synthesis [17], [18]. Recent studies have demonstrated the feasibility of converting PP-derived feedstocks into CNTs using pyrolysis-CVD-based approaches [19],[20]. Single-stage and one-pot plastic-to-CNT conversions have also been reported. Mishra *et al.* [21] converted waste PP into MWCNTs using a single-stage CVD system at predetermined reaction temperatures of 600, 700, and 800 °C, with the preferred condition identified by comparing the resulting carbon products. Gong *et al.* [22] employed one-pot carbonisation of PP using combined activated carbon and Ni₂O₃ catalysts and investigated the effect of carbonisation temperature on CNT yield. Although these studies demonstrated simplified reactor configurations, their operating temperatures were evaluated mainly through experimental screening of predetermined conditions. Precursor-specific TGA/DTG and kinetic information were not used to define a distinct lower-temperature initial pyrolysis step before CNT growth.

In conventional two-zone pyrolysis-CVD systems, the overall process consists of two main stages. The first stage is used for the thermal decomposition of plastic and the generation of volatile carbon species, whereas the second stage is used for CNT growth through catalytic deposition of carbon. These stages are generally conducted in independently controlled heating zones. Zhu *et al.* [23] combined TG-derived pyrolysis characteristics and kinetic analysis with CNT production from real-world waste plastics. However, their experiments employed two separately controlled furnaces, with plastic pyrolysis conducted at 500 °C and the catalytic zone maintained at 800 °C. Their thermal and kinetic analyses were used mainly to compare the degradation behaviour and CNT-production potential of different waste-plastic feedstocks rather than to define a sequential initial pyrolysis set point within a single-zone furnace. Essawy and Fathy [24] selected a pyrolysis temperature of 500 °C based on TGA, but their study involved scrap rubber and did not include kinetic modelling.

In contrast, the present study employs a single-zone furnace operated using sequential heating steps. Therefore, the selected initial temperature represents the first thermal set point within the same furnace rather than the operating temperature of a physically separate pyrolysis zone. This initial step is intended to initiate the thermal decomposition of PCR-PP and the generation of volatile carbon species before the higher-temperature CNT-growth step.

Understanding the thermal behaviour of carbon feedstock is important. Thermogravimetric analysis (TGA) and derivative thermogravimetry (DTG) are commonly used to identify the onset temperature (T_{onset}), maximum degradation rate temperature (T_{max}), and region decomposition. Beyond that, evaluating TGA data kinetically is highly useful to determine the apparent activation energy (E_a) and estimate the degradation mechanism. The Coats-Redfern (CR) method is commonly used in solid-state kinetic studies and is suitable for evaluating polymer degradation behaviour [25]. Kinetic parameters can be obtained from the slope and intercept of the linear plot [26]. The method also allows several solid-state kinetic models and reaction orders to be compared to identify the most suitable apparent degradation model [27]. However, studies on thermal degradation and kinetic modelling are usually reported separately from subsequent CNT synthesis studies [28], [29].

Taken together, previous studies have addressed either single-stage or one-pot plastic-to-CNT conversion using experimentally screened reaction temperatures or TGA and kinetic characterisation coupled with two-zone CNT-production systems. However, limited attention has been given to translating the TGA/DTG degradation window of FeCp₂-catalysed PCR-PP into a representative initial pyrolysis set point for a sequential two-step heating protocol in a single-zone furnace, while using CR analysis to quantify and interpret the catalyst-induced changes in degradation behaviour. Therefore, the research gap addressed here is not the general combination of TGA, kinetic analysis, and CNT synthesis, but their precursor-specific application to define and interpret the initial heating step of a single-zone synthesis protocol.

Accordingly, this study investigates the thermal degradation behaviour of PCR-PP and FeCp₂-catalysed PCR-PP, determines their apparent kinetic parameters using CR analysis, and identifies a representative TGA-informed initial pyrolysis temperature. The initial temperature was selected principally from the TGA/DTG parameters, including T_{onset} , T_{max} and the principal decomposition region, whereas the CR analysis was used to quantify the apparent activation energy, identify the most representative apparent degradation model, and interpret the influence of FeCp₂ on PCR-PP degradation. Based on the selected temperature, CNT synthesis was performed in a single-zone furnace operated under a sequential two-step heating protocol. The synthesised carbon products were characterised morphologically and structurally using SEM, XRD, and Raman spectroscopy. This study therefore demonstrates the feasibility of a precursor-specific,

thermally informed workflow for CNT synthesis from PCR-PP.

2. METHODOLOGY

2.1. Materials

Post-consumer polypropylene (PCR-PP), sourced from discarded food containers, was used as the carbon precursor. Ferrocene (FeCp₂, 98% purity), used as the metal catalyst precursor, was purchased from Sigma-Aldrich. High-purity argon (Ar) gas (Linde Malaysia Sdn. Bhd., 99.99% purity) was employed as both carrier gas and purging gas to maintain an inert atmosphere during synthesis.

2.2. Preparation of Materials

The PCR-PP was thoroughly washed with distilled water and subsequently fan-dried at room temperature until completely dry. It was then cut into small pieces of approximately 2 × 2 mm.

2.3. Thermal Degradation Analysis (TGA)

Thermogravimetric analysis (TGA) was conducted utilising a Pyris 1 thermogravimetric analyser (Perkin Elmer, USA). This analysis was performed to characterise the thermal degradation window, thermal stability, and decomposition behaviour of both the pure PCR-PP and the PP-FeCp₂ mixture at a 10:1 weight ratio. The samples were heated from 25 °C to 900 °C under continuous nitrogen (N₂) flow of 30 mL/min at a constant heating rate of 10 °C/min [30], [31]. The resulting temperature profiles were used to identify the temperature window for the initial pyrolysis in the subsequent CNT synthesis.

2.4. Kinetic Analysis using the Coats-Redfern method

The experimental TGA data were extracted and analysed kinetically using the linearised integrated Coats-Redfern equation [32]:

$$\ln \left[\frac{g(\alpha)}{T^2} \right] = \ln \left[\frac{AR}{\beta E_a} \right] - \frac{E_a}{R} \left(\frac{1}{T} \right) \quad (1)$$

where:

$g(x)$ = Integrated form of the kinetic model
 T = absolute temperature (K)
 E_a = activation energy (J/mol)
 A = pre-exponential factors (s⁻¹ or min⁻¹)
 R = universal gas constant (8.314 J/mol.K)
 β = heating rate (K/min)

α is the conversion degree (fractional mass loss) at any given temperature T during the thermal degradation process and calculated using the following formula:

$$\alpha = \frac{m_0 - m_t}{m_0 - m_\infty} \quad (2)$$

Where:

m_0 = initial mass of the sample at the onset temperature of the degradation step (g)
 m_t = mass of sample at a specific temperature T (or time t) during degradation (g)
 m_∞ = final mass of the sample at the offset (completion) temperature of the degradation step (g)

The activation energy (E_a) was extracted from the slope of the linear regression plot of:

$$y = \ln \left[\frac{g(\alpha)}{T^2} \right] \text{ versus } x = \frac{1}{T} \text{ and } E_a = -\text{slope} \times R$$

Data fitting was restricted strictly to the stable conversion range of $\alpha = 0.2 - 0.8$ to eliminate physical fluctuations commonly associated with reaction initiation ($\alpha < 0.2$) and final residue sintering/coking ($\alpha > 0.8$) [13].

2.5. Screening of Solid-Sate Kinetic Models

To determine the apparent thermal degradation mechanism, five different solid-state kinetic models $g(\alpha)$ representing various physical mechanisms were screened, as listed in Table 1. The best-fitting model was chosen based on the highest adjusted R^2 (closest to 1).

Table 1. Kinetic models and their corresponding $g(\alpha)$ functions used in Coats-Redfern method [33].

Model	$g(\alpha)$
First-order	$-\ln(1 - \alpha)$
Second-order	$\frac{1}{1 - \alpha} - 1$
Contracting volume	$1 - (1 - \alpha)^{\frac{1}{3}}$
Three-dimensional diffusion	$1 - \frac{2}{3}\alpha - (1 - \alpha)^{\frac{2}{3}}$
Nucleation and growth (Avrami-Erofeev, $n = 2$)	$[-\ln(1 - \alpha)]^{\frac{1}{2}}$

2.6. CNT Synthesis and Solid-Product Yield

CNTs were synthesised in a single-zone horizontal tube furnace using a sequential two-step heating protocol under floating catalytic chemical vapour deposition (FC-CVD) conditions. First, 10.0 g of PCR-PP and 1.0 g of FeCp₂ were weighed and placed in separate ceramic boats, while a third ceramic boat was used to collect the carbon product. The ceramic boats were positioned inside the ceramic tube, and the flange bolts were tightened. Ar was introduced at 30 mL/min through the horizontal ceramic tube (ID = 3.8 cm; heated length = 30.3 cm) to purge the air. The process consisted of two sequential temperature-controlled steps in the same furnace, both using a heating rate of 10 °C/min:

Step 1 (initial pyrolysis): The PCR-PP was pyrolysed to the selected temperature identified in Section 2.3 for 10 minutes. During this stage, the Ar flow was stopped, and the gas outlet remained open. This holding period was used to

allow the initial melting and release of volatile hydrocarbons from PCR-PP while reducing their immediate removal by the flowing Ar gas. The open outlet allowed the evolved gases to leave the ceramic tube and avoided pressure build-up.

Step 2 (FC-CVD): Following the 10-minute initial pyrolysis period, the Ar flow was resumed at 30 mL/min before the furnace temperature was increased to 900 °C [34] for a 90-minute soaking period.

After the reaction, the furnace was cooled to room temperature while maintaining the Ar flow to prevent the hot CNTs from oxidising. Once cooled, the as-synthesised CNTs were then collected from the third ceramic boat for further characterisation.

Since the collected product was weighed without purification, it is referred to as the gross solid-product yield and calculated using Eq (3) [35]:

$$Y_{solid\ product}(\%) = \left(\frac{m_{collected\ product}}{m_{initial\ PCR-PP}} \right) \times 100 \quad (3)$$

Where:

$m_{collected\ product}$ = Mass of solid product collected (g)

$m_{initial\ PCR-PP}$ = Initial mass of PCR-PP (g)

2.7. Characterisation Techniques

2.7.1. Analysis of Morphology of CNTs

The morphology of the CNT samples was examined using scanning electron microscopy (SEM) and field-emission scanning electron microscopy (FESEM). Initial morphological observation was performed using SEM (JSM-646LA, JEOL, Japan) at an accelerating voltage of 10 kV, with magnifications ranging from 100× to 20,000×. High-magnification observation was further conducted using FESEM (Nova NanoSEM 450, USA) at 15 kV to examine the tubular structure of the CNTs. The outer diameters of the CNTs were measured from high-magnification FESEM images using ImageJ software.

2.7.2. Analysis of the Structural Properties of CNTs

For further verification of CNTs' presence, X-ray diffraction (XRD) and Raman spectroscopy were performed. For XRD analysis, a D2 PHASER (Bruker, Germany) was used to analyse the crystalline structure of the materials. The copper (Cu) anode produced angle (2θ) was set from 20° to 90°. The voltage was set at 30 kV with a time step of 0.1 s. The average crystallite size (D_c) was determined using the Scherrer equation [36]:

$$D_c = \frac{K\lambda}{\beta \cos\theta} \quad (4)$$

Where:

K = dimensionless shape factor (taken as 0.94)

λ = X-ray wavelength (1.5406 Å)

β = full width at half maximum (FWHM) of the diffraction peak in radians

θ = Bragg angle (half of the 2θ peak position)

Raman spectroscopy was performed using a Renishaw inVia Raman microscope with a 20 mW HeNe laser at 633 nm. The operating specifications included 1800 grooves/g/mm (1 cm⁻¹), 10% laser power, and a 50 × N-plan objective lens with a 1 μm laser spot size. SynchroScan encompassed a spectral range of 100 cm⁻¹ to 3200 cm⁻¹. The defects and structural graphitisation quality of the as-synthesised CNTs were calculated from the ratio of integrated area of the D-band to the G-band [37]:

$$\frac{I_D}{I_G} = \frac{A_D}{A_G} \quad (5)$$

Where:

$\frac{I_D}{I_G}$ = Defect to graphitic intensity ratio

A_D = Integrated area under the curve for D-band peak.

A_G = Integrated area under the curve for G-band peak.

Spectral processing and data analysis were conducted using Origin software. The diameter of the single-walled CNTs (d_t , nm) was estimated from the radial breathing mode (RBM) frequency (ω_{RBM} , in cm⁻¹) using the empirical relation [38]:

$$d_t = \frac{248}{\omega_{RBM}} \quad (6)$$

3. RESULTS AND DISCUSSION

3.1. Thermal and Volatilisation Behaviour (TGA/DTG Analysis)

Figure 1 (a) shows TGA curves of PCR-PP, FeCp₂-catalysed PCR-PP and pure FeCp₂ during heating up to 900 °C under nitrogen flow. Both PCR-PP and pure FeCp₂ show a single-step thermal degradation, whereas the FeCp₂-catalysed PCR-PP displays an additional low-temperature mass loss step before the main polymer degradation.

Figure 1 (b) shows the corresponding DTG curves. The catalysed sample shows a minor downward DTG peak at 119.19 °C, attributed to FeCp₂ volatilisation, followed by the main polymer degradation peak at 431.24 °C. In comparison, the main DTG peak of neat PCR-PP occurs at 446.46 °C. The thermal parameters extracted from the TGA and DTG curves are summarised in Table 2.

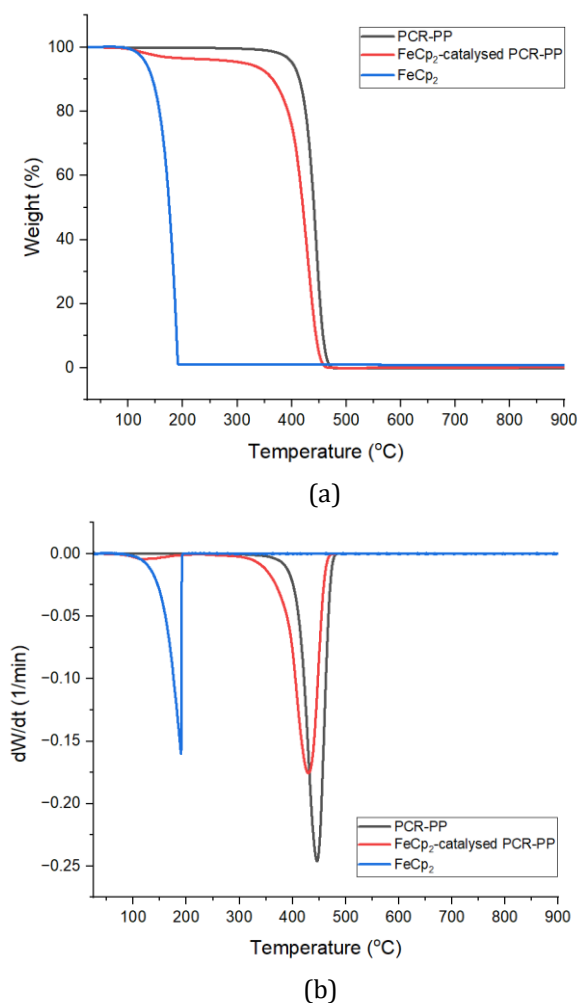


Figure 1. (a) TGA and (b) DTG curves of PCR, FeCp₂-catalysed PCR-PP and FeCp₂

Table 2. Thermal parameters extracted from TGA and DTG curves.

Sample	T _{onset} (°C)	T _{offset} (°C)	ΔT (°C)	T _{max} (°C)	Residual Weight (%)
FeCp ₂	143.60	198.67	55.07	190.03	0.76
PCR-PP	411.47	466.31	54.84	446.46	0.00
FeCp ₂ -catalysed PCR-PP	373.23	459.33	86.10	119.19 (weak) ^a 431.24 ^b	0.29

Note: ^aWeak DTG peak for FeCp₂ sublimation, and ^bmain DTG peak for polymer degradation

3.4. Thermal Behaviour of the FeCp₂-catalysed PCR-PP

The TGA curve of FeCp₂-catalysed PCR-PP in Figure 1(a) shows a reduced onset degradation temperature (373.23 °C) compared to the neat PCR-PP (411.47 °C), indicating that FeCp₂ promotes earlier thermal degradation. The catalysed sample also shows a broader decomposition range (ΔT=86.10°C), suggesting a more gradual mass loss. Its main decomposition occurs between 373.23 °C and

3.2. Thermal Degradation Behaviour of PCR-PP

PCR-PP exhibits one major degradation step between 411.47 °C to 466.31 °C in Figure 1(a), corresponding to the breakdown of the polymer backbone. This result is comparable with those reported by Esmizadeh *et al.* [39] for commercial PP homopolymer powder. This is characteristic of thermal cracking of polypropylene, resulting in complete volatilisation of organic fragments and no detectable residue at 900 °C. The degradation is consistent with a chain scission process, where the polymer backbone progressively breaks into smaller volatile hydrocarbon species. These species are released as gases during heating, resulting in complete mass loss.

3.3. Thermal Degradation Behaviour of FeCp₂

The TGA curve of pure FeCp₂ shows an initial mass loss beginning at 143.6 °C in Figure 1(a), while the corresponding DTG peak occurs at 190.03 °C in Figure 1(b). This mass loss is associated with volatilisation and thermal decomposition at higher temperatures. This is consistent with other studies [40], [41]. The decomposition of FeCp₂ leads to the formation of iron-containing species which remain as residue after thermal treatment. The residual mass of 0.76% indicates the presence of thermally stable iron-based species at high temperature.

431.24 °C, representing the active volatilisation region for hydrocarbon generation.

The DTG curve in Figure 1(b) shows that the main polymer degradation peak shifts from 446.46 °C for neat PCR-PP to 431.24 °C for the FeCp₂-catalysed sample. This reduction of 15.22 °C indicates that the maximum degradation rate occurs at a lower temperature and is consistent with the lower apparent activation energy discussed in Section 3.5. The residual mass of 0.29% suggests the presence of

thermally stable iron-containing species at high temperature. Similar behaviour has been reported for PP pyrolysis over low-cost mineral catalyst such as natural zeolite and bentonite [42].

3.5. Kinetic Modeling and Solid-State Mechanism Screening

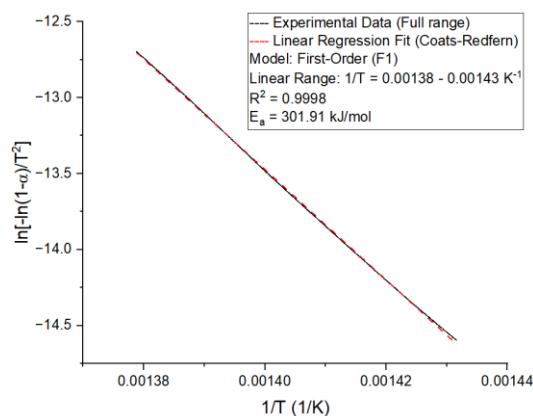
The TGA data were analysed to identify the best-fitting kinetic model and screen the apparent solid-state degradation mechanisms for PCR-PP and FeCp₂-catalysed PCR-PP. The intercept and slope for each model were used to calculate the activation energy (E_a). Tables 3.2 and 3.3 summarise the calculated kinetic parameters for PCR-PP and FeCp₂-catalysed PCR-PP obtained over the conversion range $\alpha = 0.2 - 0.8$.

As shown in Figure 2, the best-fitting model for both PCR-PP and FeCp₂-catalysed PCR-PP was the apparent first-order kinetic solid-state model, with adjusted R^2 values of 0.99975 and 0.99887, respectively. The addition of FeCp₂ did not change the solid-state degradation mechanism. However, it significantly reduces the activation energy from 301.91 kJ/mol to 160.17 kJ/mol, corresponding to a decrease of 46.95%. This trend is consistent with previous studies [43], [44], although the magnitude of the reduction varies with catalyst type, acidity, pore structure, catalyst loading, and whether the system involves pure PP or co-pyrolysis.

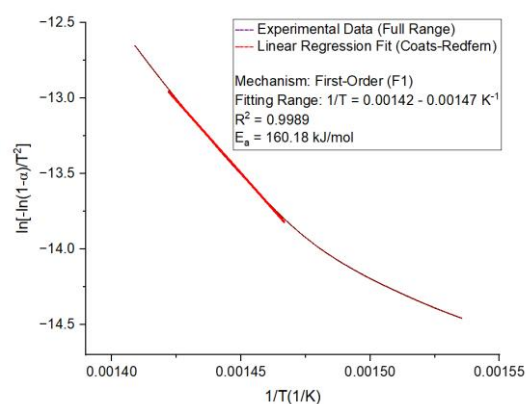
The lower E_a indicates that FeCp₂ facilitates thermal degradation by reducing the apparent energy barrier for polymer chain scission [45]. This result is consistent with the lower T_{onset} and broader decomposition range of FeCp₂-catalysed PCR-PP discussed in Section 3.1. Despite the reduction in activation energy, the degradation behaviour of both samples still follows first-order kinetics, in which the degradation rate depends on the amount of remaining PCR-PP.

For both samples, the first-order model is the most appropriate, as PCR-PP undergoes complete melting prior to degradation, allowing relatively uniform interaction within the reaction medium. Under these conditions, the degradation rate is mainly governed by the concentration of the remaining polymer, rather than changes in surface area or diffusion effects. However, comparison with other studies does not show a universal first-order mechanism. The stronger consensus is that PP usually degrades in one main step, while the fitted kinetic mechanism depends on temperature window, model selection, and the presence of catalysts, fillers, or other polymers [46], [47].

Other models, including the second-order, contracting-volume, diffusion-controlled, and Avrami-Erofeev models, produced slightly lower adjusted R^2 values. This further supports that the degradation process is not controlled by surface contraction, diffusion limitation, or nucleation-growth mechanisms.



(a)



(b)

Figure 2. Coats-Redfern plots for (a) PCR-PP and (b) FeCp₂-catalysed PCR-PP.

Table 3. Kinetic parameters of PCR-PP within $\alpha = 0.2 - 0.8$.

Mechanism Model	Intercept	Slope	Adj. R ²	E _a (kJ/mol)
First-Order	37.38	-36323.15	0.99975	301.91
Avrami-Erofeev (n=2)	11.12	-17449.83	0.99973	145.09
3D Diffusion (Jander)	76.36	-66164.68	0.99969	550.12
Contracting Volume	30.61	-32370.60	0.99967	269.14
Second-Order	57.42	-50319.90	0.99173	418.38

Table 4. Kinetic parameters of FeCp₂-catalysed PCR-PP within $\alpha = 0.2 - 0.8$.

Mechanism Model	Intercept	Slope	Adj. R ²	E _a (kJ/mol)
First-Order	14.44	-19264.74	0.99887	160.17
Contracting Volume	8.82	-16220.02	0.99840	134.85
3D Diffusion (Jander)	32.59	-33737.48	0.99839	280.49

Mechanism Model	Intercept	Slope	Adj. R ²	E _a (kJ/mol)
Avrami-Erofeev (n=2)	-0.92	-8524.18	0.99617	70.870
Second-Order	24.96	-26256.33	0.98915	218.29

3.6. Pyrolysis Temperature Selection

To establish an integrated workflow for CNT synthesis, the first-stage operating temperature was selected from the thermal parameters of the catalysed polymer. Figure 1 shows an onset degradation temperature of 373.23 °C, while Figure 1(b) shows a main degradation peak at 431.24 °C. This range represents the active decomposition region used to select the initial condition for volatile hydrocarbon generation. CR analysis gave an apparent activation energy of 160.17 kJ/mol and supported the interpretation of the catalysed degradation behaviour. Based on the TGA/DTG results, 400 °C was selected as a representative initial pyrolysis temperature. This temperature is lower than those commonly employed in conventional PP pyrolysis studies, which typically exceed 500 °C [48]. The selected temperature therefore falls within the active decomposition region while reducing unnecessary thermal severity.

3.7. Solid-Product Yield

The complete sequential synthesis process produced 0.1844 g of collected solid product from 10.0 g of initial PCR-PP, corresponding to a gross solid-product yield of 1.84 wt%. This shows that only a small fraction of the initial PCR-PP mass was recovered as a solid product. The low product recovery may be related to the rapid release of volatile compounds during the 10-min no-flow pyrolysis at 400 °C. Some hydrocarbon vapours may have escaped through the open outlet before decomposing to form solid carbon, thereby reducing the amount of solid product recovered. Oil and wax-like deposits were also observed in the cooler section of the ceramic tube near the outlet. This suggests that some of the vapours condensed instead of contributing to carbon deposition. The collected solid product was characterised morphologically and structurally without further purification.

3.8. Morphological and Structural Validation of Synthesised CNTs

The low-magnification (100×) SEM image in Figure 3(a) shows dense, brush-like CNT bundles with a multi-directional growth pattern. Some bundles stand vertically, while others are tilted or lie flat. This morphology is typical of FC-CVD, where CNT growth is not limited to a horizontal metal substrate surface [49]. Increasing the magnification to 500×, as shown in Figure 3(b), reveals closely packed tubes, with some areas of the bundle surfaces covered with impurity aggregates. The 80,000× image in Figure 3(c) shows individual, distinct and high-contrast tubular

structures. The outer walls of the nanotubes in this selected area are generally clean and smooth. Statistical outer diameter distribution analysis of these high-resolution images indicates a tube diameter range from 34.2 nm to 109.9 nm, with the dominant population peak centred around 72.1 ±15.4 nm, as shown in Figure 3(d). This outer diameter distribution supports the predominant formation of multi-walled carbon nanotubes (MWCNTs).

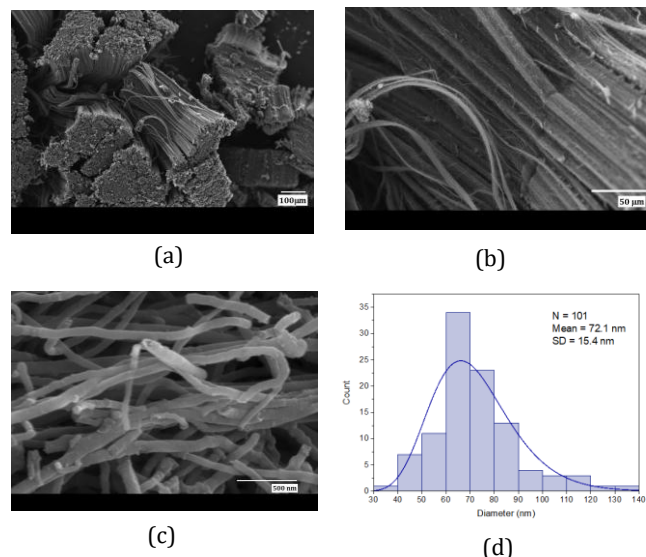


Figure 3. SEM analysis of the as-synthesised CNTs: (a) 100×, (b) 500×, (c) 80,000× magnification, with (d) outer diameter distribution.

The XRD pattern in Figure 4 was analysed to evaluate the crystalline structure of the as-synthesised carbon product. The sharpest peak centred at $2\theta = 25.15^\circ$ is assigned to the (002) plane of graphitic multilayer carbon and supports the formation of MWCNTs [50]. Along with that, the in-plane atom arrangement within the hexagonal graphene sheets also confirms the structural integrity of the honeycomb carbon network along the tube walls. This is represented by the minor diffraction feature around $2\theta = 41.99^\circ$, assigned to the (100)/(101) planes.

The (002) diffraction peak exhibited an FWHM of 0.017 rad, indicating crystalline ordering within the carbon structure [51]. The calculated crystallite size of 8.26 nm further confirms the presence of nanoscale graphitic domains associated with CNT formation. In addition to the graphitic carbon phase, secondary phases corresponding to magnetite (Fe_3O_4) and iron carbide (Fe_3C) were also detected. The Fe_3C phase is likely formed through interactions between carbon species and residual iron catalyst particles during the FC-CVD process. On the other hand, the Fe_3O_4 phase may have been generated through the oxidation of Fe species by oxygen-containing compounds present in or released from PCR-PP during heating. These compounds may originate from additives, such as antioxidants and processing stabilisers, or from degradation products, including organic acids and aldehydes such as 2,2-dimethylpropanoic acid, heptanal and dodecanal [52].

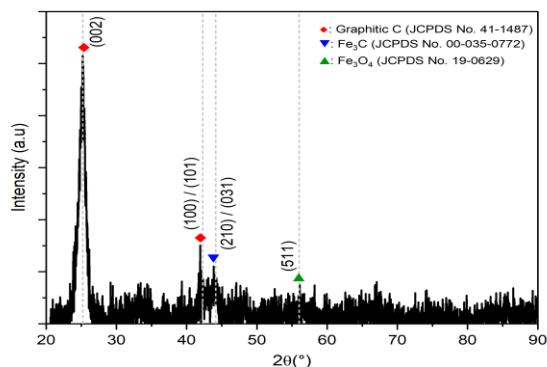


Figure 4. XRD pattern of the synthesised CNTs

While XRD identified the crystalline phases, Raman spectroscopy was used to further evaluate the structural quality and carbon bonding of the CNTs as presented in Figure 35.

The peak at G-band (1575.21 cm^{-1}) is characteristic of sp^2 -hybridised graphitic carbon lattice and is consistent with the XRD (002) peak [53]. The presence of disordered carbonaceous species or structural defects, specifically in graphene-like sp^2 carbon domains, is determined from the D-band (1323.78 cm^{-1}) [54]. This confirms the presence of carbonaceous impurities, aligning with the morphological observations of amorphous aggregates in Figure 3.3 (b). The calculated I_D/I_G ratio from the integrated peak area ratio of the D band to the G band is 0.69, which indicates a moderate level of structural defects or non-crystalline carbon within the MWCNT structure.

The existence of a low-frequency peak at 215.63 cm^{-1} , and 277.71 cm^{-1} , which is less than 500 cm^{-1} which assigned to the Radial Breathing Mode (RBM), reveals the co-existence of single-walled carbon nanotubes (SWCNTs) with an estimated tube diameter of 0.89 nm to 1.15 nm, alongside the larger MWCNTs. Interestingly, the 2D band (2649.22 cm^{-1}) possesses the highest intensity, indicating a strong resonance response from the highly crystalline, conductive SWCNT structure within the nanocarbon mixture [55]. However, further TEM analysis is required to confirm this assignment.

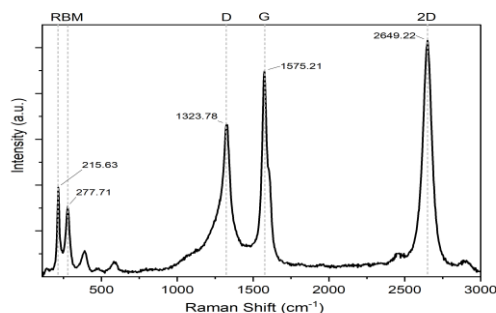


Figure 5. Raman spectra of the synthesised CNTs

3.9. CNT Formation Mechanism

The CNTs in this study were synthesised using a sequential two-step heating process in a single-zone furnace. The process involved initial thermal decomposition of FeCp_2 -catalysed PCR-PP followed by catalytic chemical vapour deposition (FC-CVD). The TGA identified the active decomposition region used to select the representative operating condition for the first pyrolysis process.

During the initial pyrolysis, FeCp_2 -catalysed PCR-PP undergoes thermal cracking to generate volatile carbonaceous species, which act as carbon precursors for CNT growth. The Coats–Redfern kinetic analysis described this degradation using an apparent first-order model, in which the reaction rate of FeCp_2 -catalysed PCR-PP depends on the amount of PCR-PP remaining.

During the subsequent FC-CVD step, the generated carbon species are exposed to active Fe sites derived from FeCp_2 , leading to nucleation and growth of CNTs. The formation of tubular graphitic structures was confirmed by SEM analysis, while Raman spectroscopy and XRD further verified the presence of graphitic carbon and improved structural ordering.

Overall, the results indicate that TGA-guided initial pyrolysis at $400\text{ }^\circ\text{C}$ generated carbon precursors that subsequently formed a CNT-containing carbon product in the single-zone sequential-heating process. The low gross solid-product yield indicates that further optimisation is required to improve carbon deposition and product recovery.

4. CONCLUSION

Post-consumer recycled polypropylene (PCR-PP) waste is abundantly generated and discarded, highlighting a strong need for its valorisation into high-value functional carbon nanomaterials. In this study, the thermal degradation behaviours of PCR-PP and FeCp_2 -catalysed PCR-PP were investigated using TGA. Coats–Redfern kinetic screening confirmed that the thermal degradation of both systems followed an apparent first-order solid-state kinetic model. The addition of the catalyst precursor lowered the activation energy (E_a) by nearly half, from 301.91 to 160.17 kJ/mol, corresponding to a reduction of 46.95%.

Based on the active decomposition window identified from the TGA profiles ($373.23\text{--}431.24\text{ }^\circ\text{C}$), a representative temperature of $400\text{ }^\circ\text{C}$ was selected for the initial pyrolysis step. The generated gaseous carbon precursors were then exposed to active Fe sites derived from the thermal decomposition of FeCp_2 during FC-CVD growth at $900\text{ }^\circ\text{C}$. This integrated process produced highly bundled, graphitic CNTs, as validated by morphological and structural characterisation using SEM, XRD and Raman spectroscopy. The gross solid-product yield was 1.84 wt%. Overall, this work demonstrates the potential of a thermally and kinetically guided workflow to support the development of

nanocarbon synthesis from PCR-PP using a single-zone sequential-heating CVD process. However, this study was limited to one initial pyrolysis condition and the characterisation of an unpurified product. Future work should evaluate different PCR-PP loadings and initial pyrolysis times, together with product purification, to improve and quantify CNT recovery.

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