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Synthesis of Carbon Nanomaterials from Polyethylene Terephthalate (PET) Waste Using Chemical Vapor Deposition

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Abstract: Upcycling is an effective approach to reduce plastic waste, polyethylene terephthalate (PET) and promote sustainability. Plastic bottles usually were made from PET polymer and a raw material to produce carbon nanomaterials (CNMs). CNMs are synthesized using chemical vapor deposition (CVD) process and purified to eliminate catalysts and unwanted compounds. Various catalysts were used to investigate the economic and effective in producing the CNMs. Metal catalysts such as ferrocene, cobalt and iron are the important elements in the CVD process as they provide surfaces for carbon to attach. CNMs morphology and graphitic structure were observed from Raman analysis and TEM analysis. The application of upcycling offers the advantage of utilizing low-cost raw materials to produce higher-value products, providing additional benefits.

Keywords: Carbon nanomaterials; Chemical vapor deposition; PET waste

Introduction

Plastics play an important role as one of the largest plastic manufacturers in Malaysia and exported over 30 billion Malaysian Ringgit (MYR) value of resin to the industries (Chen et al., 2021). Number of plastic productions keeps rising makes the world flood with plastics waste and it is overwhelmed to deal when plastic can be consistent in the environment more than 450 years (Sharifian & Asasian-Kolur, 2022; Kibria et al., 2023; Shaaban et al., 2024). More than 85% of trash collected were sent to the landfill as it is the easiest way and cost efficient in solid waste management (Chen et al., 2021). Upcycling method can be an alternative approach to reuse the material in such way to create of a higher quality or value from its original (Zaharuddin et al., 2025). Upcycling promotes sustainability in a way of circular economy to discard the plastic waste where it can be used as raw material to create a valuable product (Yuan et al., 2021). A plastic namely polyethylene terephthalate (PET) is thermoplastic polymer that consists of terephthalate acid monomers derived from petroleum (De Cort Suzanne et al., 2017) and ethylene chain (Glaser et al., 200 C.E.). The characteristics of PET such as durable, light, shatterproof and strong inert materials make it ideal for food packaging applications, especially for drinking bottles (Coniglio et al., 2020).

Moreover, PET bottles can recycle almost entire of the materials into many forms as PET is a thermoplastic polymer that can be processed at high temperature (Nisticò, 2020). PET from plastic bottles can be the feedstock to synthesize carbon nanomaterials (CNMs) via the thermal degradation, chemical vapor deposition (CVD) process (Sharifian & Asasian-Kolur, 2022). Catalyst activation is required to provide an active site and crucial for quality of carbon nanotubes (CNTs) growth (Sharifian & Asasian-Kolur, 2022b) . The growth and yield of CNTs are influenced from the selection of catalyst and the temperature (Magrez et al., 2010). The effectiveness of different catalysts to produce high yield

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of CNTs that are cost effective were investigated in this study. The composition of catalyst and process parameters on CNTs are investigated to identify a cost-effective and sustainable synthesis route. Thus, the CNTs can be applied in various applications including coating surfactant (Paul et al., 2010) and additives (Harun et al., 2018; Harun et al., 2024).

Method

Materials

A catalyst provides an active site for carbon to attach and bind on the catalyst surfaces. Several catalysts were applied to produce the CNTs. Ferrocene, Ferrocene/Aluminum oxide, Cobalt/Aluminum oxide, Iron and Iron/aluminum oxide to identify the suitable and cost-effective for producing the high yield of CNT. The ratio between metal and support is 1: 2 for 5.0 of catalyst. The source of PET comes from waste plastic bottles such as mineral bottles that were collected and crushes using crushing machines into fine pieces of PET flakes around 10 - 15 mm. After crushing process, 5.0 g of PET flakes and 2.0 g of catalyst powder were inserted into the vessel of Chemical Vapor Deposition (CVD) machine for 2 hours of the process at 800 °C and gas nitrogen, N_2 as the inert gas. The CNTs produced resulting from the process undergoes purification process, reflux to separate the catalyst from CNTs. CNTs is cleaned with deionized water by centrifugation process with 10 000 rpm for 15-20 minutes. The sediment of CNTs obtained at the bottom of the centrifuge tube while the supernatant was discarded and replaced with deionized water. This step was repeated several times until the supernatant pH is 7. The defects and diameter distribution of carbon was investigated by analyzing Raman band, D band, G band and 2D band (Li et al., 2023). The intensity ration between peaks intensity of D-band and intensity of G band ($\frac{I_D}{I_G}$) used as an indicator the quality of CNTs where low ratio of ($\frac{I_D}{I_G}$) gives the higher quality of CNTs. Thermogravimetric analysis (TGA) provides the basis for monitoring the thermal degradation of CNTs. TGA is used for analyzing the weight fluctuation of mass when the heat is introduced (Martincic et al., 2024). CNMs have hydrophobic properties and usually appear in bulk solid. The morphology of nano materials can be observed by transmission Electron Microscopy (TEM) analysis, and it gives the structure and purity information.

Results and Discussion

Yield of CNTs during CVD process

The yield of synthesis of CNTs produced after one cycle of CVD process can be observed in Table 1 below. The weight of CNTs using cobalt catalyst shows the highest amount which is 2.3732 g followed by CNTs using iron catalyst, 2.2365 g and the lowest yield of CNTs using the ferrocene catalyst, 0.0419 g. Cobalt catalyst shows a better growth rate surpass the other metal catalysts due to the catalytic activity for hydrocarbon diffusion on the active site (Suriani et al., 2013). One cycle of CVD process used 2.0 g of each catalyst including the catalyst with support which is the available active site from the catalyst that only contain metal is high compared to the catalyst with support. Comparing the available active sites from the catalyst between iron catalyst and iron/alumina oxide, iron provides more active sites than iron/alumina oxide. However, yield of CNTs from iron/alumina oxide catalyst, 2.2225 g near to yield of CNTs from cobalt catalyst, 2.2365 g. This can be due to the dispersion of iron being finely across the porous surface of Al_2O_3 support enhancing the catalytic activity (Shukrullah et al., 2019; Hattori et al., 2021). The support from iron/alumina oxide enhances stability and prevents the iron from sintering resulting in maintaining iron/alumina oxide catalyst during high temperature CVD process (Magrez et al., 2010).

Table 1. Yield of CNMs using different catalysts

Catalyst	Yield of CNMs, g
Ferrocene, $Fe(C_5H_5)_2$	0.0419
Ferrocene/alumina oxide, $Fe(C_5H_5)_2/Al_2O_3$	1.0185
Cobalt, Co	2.3732
Cobalt/alumina oxide, Co/Al_2O_3	1.9511
Iron, Fe	2.2365
Iron/alumina oxide, Fe/Al_2O_3	2.2225

Raman Spectroscopy result

The grown CNTs on the catalyst were observed peaks at ~1300-1500 cm-1 for disordered bands (D peaks), ~1500-700 cm-1 for graphitic bands (G peaks) and ~2600-2900 cm-1 for second order bands (2D) peaks.

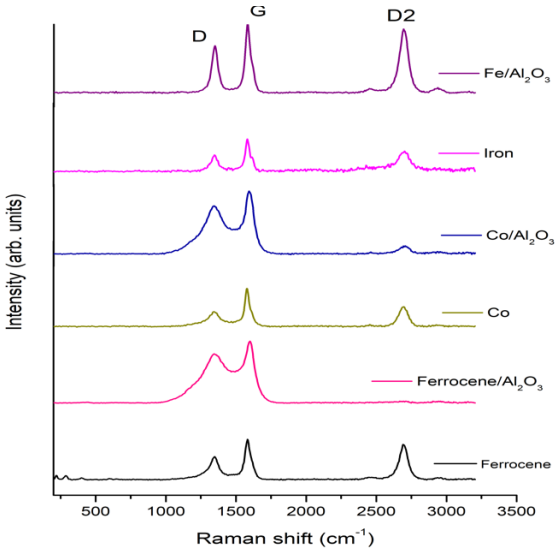

Figure 1. Raman spectra for analyzing the quality of CNMs using different catalysts

The spectra displayed in Figure 1 contain a mixture of graphitic and disordered carbon and the catalyst with presence of alumina oxide, Al_2O_3 support did not significantly affect the defect density, as indicated by the $\frac{I_D}{I_G}$ ratio. Overall, the value the $\frac{I_D}{I_G}$ ratio displays the same value with different ± 0.01. Ferrocene/alumina oxide and cobalt/alumina oxide are the lowest $\frac{I_D}{I_G}$ ratio, 0.84, which indicates similarity quality of CNTs. Ferrocene/alumina oxide and cobalt/ alumina oxide catalyst illustrate a broad peak of D and G band peak showing that CNTs have low crystallinity and graphitic structure (Baitinger et al., 2011). On the other hand, ferrocene and iron/alumina oxide catalysts show a clear 2D peak compared to the other catalysts. However, peaks from ferrocene catalyst are not intense as iron/alumina oxide catalyst and the height is similar with iron and cobalt catalyst. Iron/alumina oxide catalyst displays the most intense and sharp peak explaining the crystallinity of the CNTs are excellent and have a strong graphitic structure. A secondary defect band, D_2 indicates the disorder within sp^2 carbon layer (Baitinger et al., 2011). The 2D peaks presence at ferrocene, cobalt, iron and iron/alumina oxide graph suggests the structure of graphene appears but iron/alumina oxide have the highest peak while the 2D peak on ferrocene/alumina oxide and cobalt/alumina oxide catalyst did not appear. This suggests less graphitic character or poor layering of CNTs produce (Dong et al., 2013; Syduzzaman et al., 2025). Table 2 shows the scattering of CNTs peaks and $\frac{I_D}{I_G}$ ratio from different catalysts.

Table 2. Shows the scattering of CNMs peaks from different catalysts

Catalysts	D-peaks (cm^{-1})	2D-peaks (cm^{-1})	G-peaks (cm^{-1})	$\frac{I_D}{I_G}$ ratio
Ferrocene, $Fe(C_5H_5)_2$	1349.00	2691.55	1582.60	0.85
Ferrocene/alumina oxide, $Fe(C_5H_5)_2/Al_2O_3$	1346.40	-	1600.30	0.84
Cobalt, Co	1343.80	2695.77	1577.53	0.85
Cobalt/ alumina oxide, / Al_2O_3	1341.19	2705.97	1592.72	0.84
Iron, Fe	1346.40	2705.97	1592.72	0.85
Iron/alumina oxide, Fe/Al_2O_3	1349.00	2693.75	1582.60	0.85

Thermogravimetric Analysis

The selected synthesized CNTs from ferrocene, cobalt and iron catalysts that have been purified were sent to of purified CNTs from ferrocene, cobalt and iron catalysts.

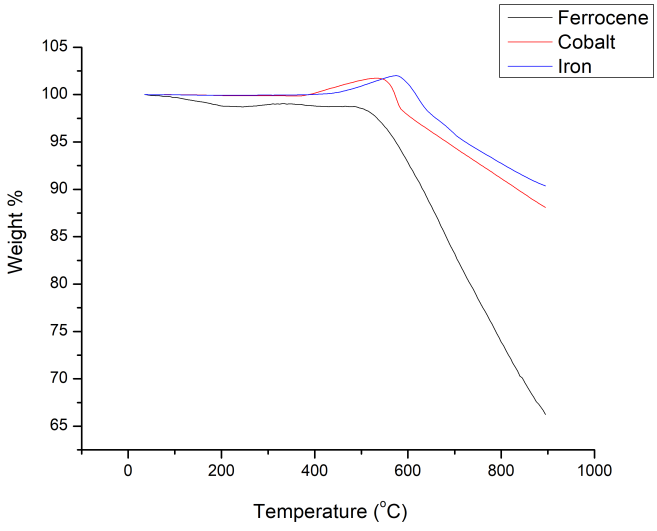

Figure 2. TGA curves of purified CNMs from selected catalysts

The thermal decomposition in Figure 2 starts from 0 °C until 900 °C and the condition is under nitrogen, N_2 atmosphere. CNTs from ferrocene catalysts show a weight loss at low temperatures approximately until 200°C indicates the volatilization of moisture or organic compound that is still left in the CNTs after purification (Betar et al., 2021).

The weight of CNTs is constant until 400°C and gradually decreases at higher temperatures around 600°C. Greater weight loss suggests the presence of more amorphous carbon or volatile components in the CNTs (Martincic et al., 2024). On the other hand, CNTs from cobalt and iron catalysts show a different behavior than ferrocene (Kathyayini et al., 2004) where the weight (%) loss of CNTs is minimal suggesting the CNTs derived from these catalysts have better thermal stability, more graphitized CNTs or low content of volatile compound (Leino et al., 2013). However, the weight of the CNTs slightly increases at higher temperature, 400 °C which can be due to oxidation of iron occurred (Ayillath Kutteri et al., 2018).

Transmission Electron Microscopy (TEM) Analysis

TEM analysis was conducted to survey the morphology of synthesized carbon nanotubes (CNTs) by CVD process and comparing with the carbon nanotubes pristine as the control sample. Calcination is a thermal treatment at high temperature but below its melting point to remove the impurities resulting a pure CNTs (Pillai et al., 2008). Figure 7 (a) shows the TEM image of pristine carbon nanotubes (CNTs) exhibit a smooth, entangled tubular structure with relatively uniform

diameter, indicating well-formed multi-walled CNTs (MWCNTs) (Zaine et al., 2014). Figure 7(b) displays irregular morphology, and dense structure indicates incomplete graphitization and the presence of impurities (Hansson et al., 2020). This indicates the presence of residue catalysts during the synthesis process (Harun et al., 2018). After calcination, Figure 7(c) represents clearer tubular structures with fewer amorphous carbon and impurities. This shows thermal treatment helps in improving the crystalline of carbon (Cheng et al., 2023). The differences in morphology between uncalcined CNTs and calcined CNTs showing the purification of CNTs was effective (Hansson et al., 2020).

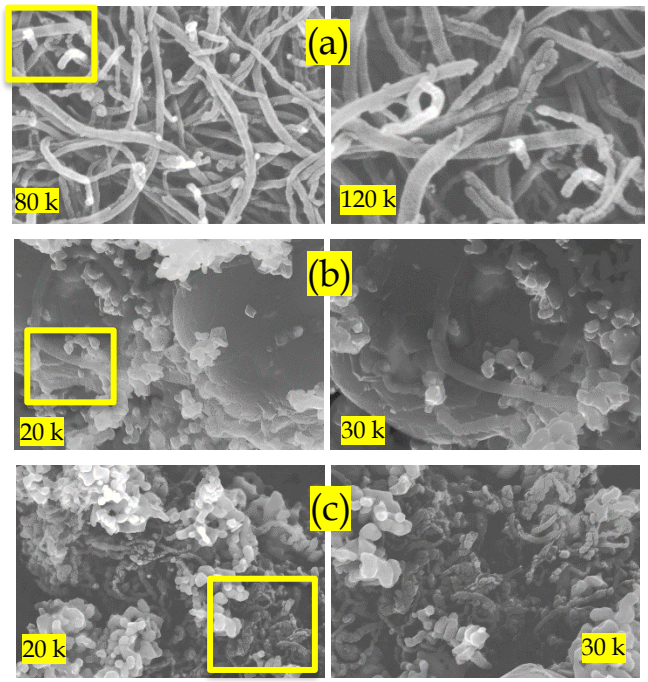

Figure 7. SEM images showing the morphology of (a) pristine CNTs, (b) uncalcined CNTs, and (c) calcined CNTs at different magnifications.

Conclusion

Synthesis of CNTs was successful using thermal techniques which CVD process. Iron/alumina oxide catalyst shows the best quality of CNTs compared to other catalysts which can be seen from Raman spectroscopy and the yield in one cycle. The $\frac{I_D}{I_G}$ ratio of iron/alumina oxide is 0.85, similar to CNTs produced from ferrocene, cobalt and iron catalyst. However, the CNTs from iron/alumina oxide present intense and high peak of the D-band G-band and 2D band while the CNTs from ferrocene/alumina oxide and cobalt/alumina oxide catalyst peaks is broad indicating less in crystallinity and graphitic structure. The high and intense D_2 band suggests the presence of graphene or multi-wall carbon nanomaterials (MWNTs). The TGA curve displays CNTs from cobalt and iron catalysts are more thermal stability compared to CNTs from ferrocene catalysts. The slight increase in percentage weight of CNTs due to the oxidation reaction of iron catalyst. This result shows that structural of CNTs from cobalt and iron catalysts are well-ordered. TEM analysis proves the significant of CNTs purification enhancing their morphology and tubular structure in coating formulation. It also highlights calcined CNTs produce a better morphology structure and eliminates the impurities. Investigating the outcome of using different catalysts and varying the doses of gamma irradiation contributes to the optimization in synthesis functionalized CNTs, enhancing its potential for various applications.

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Author Contributions

Conceptualization, M. H. H, F. F. H., M. H and M. S. A; methodology, K. A. K.; software, N. O and K. A. K.; validation, M. H. H, M. H, R. T., M. F. A. R. and M. S. A; formal analysis, K. A. K, I. M. Z. and S. N. E. W. M. A.; investigation, K. A. K, I. M. Z. and S. N. E. W. M. A.; resources, M. H. H.; data curation, K. A. K, I. M. Z., K. N. K. U. and S. N. E. W. M. A.; writing—original draft preparation N. A. M. E., K. A. K. and M. H. H.; writing—review and editing, M.H. and M. S. A.; visualization, M. H. H. and M. S. A; supervision, M. H. H and M. S. A.; project administration, M. S. A. and M.H. H.; funding acquisition, M.H.H. All authors have read and agreed to the published version of the manuscript.

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Conflicts of Interest

The authors declare no conflict of interest.

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