



*Review Article*

## Hydrocarbon Sources for the Carbon Nanotubes Production by Chemical Vapour Deposition: A Review

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### ABSTRACT

The synthesis of carbon nanotubes (CNTs) using a chemical vapour deposition (CVD) method requires the use of hydrocarbon as the carbon precursor. Among the commonly used hydrocarbons are methane and acetylene, which are both light gas-phase substances. Besides that, other carbon-rich sources, such as carbon monoxide and coal, have also been reportedly used. Nowadays, researches have also been conducted into utilising heavier hydrocarbons and petrochemical products for the production of CNTs, such as kerosene and diesel oil. Therefore, this article reviews the different kind of hydrocarbon sources for CNTs production using a CVD method. The method is used for it allows the decomposition of the carbon-rich source with the aid of a catalyst at a temperature in the range 600-1200°C. This synthesis technique gives an advantage as a high yield and high-quality CNTs can be produced at a relatively low cost process. As compared to other processes for CNTs production such as arc discharge and laser ablation, they may produce high quality CNTs but has a disadvantage for use as large scale synthesis routes.

*Keywords:* Hydrocarbon source, carbon nanotube, catalyst, chemical vapour deposition

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### INTRODUCTION

Carbon nanotubes (CNTs) have received much attention since their discovery by Iijima in 1991 (Iijima, 1991). CNTs display unique mechanical, electrical, optical and chemical characteristics (Eatemadi et al., 2014; Kotsilkova et al., 2015). These particles

are used for clean energy applications, such as a solar cell (Wang et al., 2015), hydrogen storage (Su et al., 2016) and supercapacitor (Lva et al., 2012). CNTs are also applied in micro and nano electromechanical system (Fedorovskaya et al., 2016) and high-performance low-detecting limit sensor (Cheraghi & Taher, 2016). These nanoparticles can be categorised into single-walled CNTs (SWCNTs), with a typical diameter in the range 0.6-3 nm, and multi-walled CNTs (MWCNTs) with a diameter in the range of 10-50 nm. SWCNTs have a rolled of graphene sheet to form a cylinder, whereas MWCNTs consist of multiple cylindrical rolled graphene sheets (Camilli et al., 2011) (Figure 1). CNTs are generally synthesised through arc discharge, laser ablation, and chemical vapour deposition (CVD). Both arc discharge and laser ablation are practical for synthesising SWCNTs (Ka et al., 2012; Maria & Mieno, 2015). The technique involves the use of graphite (Liew et al., 2014), and recently, coal has also been used as a source material that was vapourised and later deposited as CNTs (Moothi et al., 2012). High quality CNTs are produced but the high energy consumption in generating the discharge for the former and laser processing for the later contributed to high costs of equipment and energy. On the other hand, CVD is considered to be the most convenient method to grow all varieties of CNTs. Moreover, CVD is the best choice for large-scale production of CNTs at a relatively low cost under mild growth conditions (Szabó et al., 2010). The utilisation of hydrocarbon sources for the production of high-value chemicals and materials such as CNTs has an obvious impact on sustainable development. As a result, several research groups have explored the use of hydrocarbons as a carbon source for CNT synthesis.

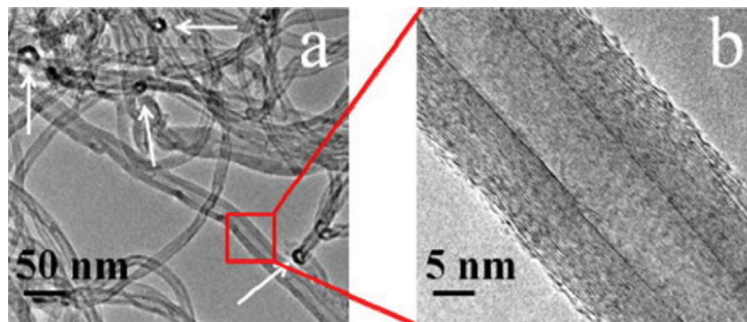


Figure 1. TEM images showing the multi-wall nature of CNTs (Camilli et al., 2011)

### SYNTHESIS OF CARBON NANOTUBES BY CHEMICAL VAPOUR DEPOSITION METHOD

CVD is the most commonly used technique to grow all varieties of CNTs. First, a catalyst and a carbon precursor easily react in CVD because this technique only requires an oven designed with a tubular reactor such as a quartz tube and a few gas flow controllers (Nerushev et al., 2001; Hashempour et al., 2014). Second, numerous parameters can be investigated, not only during catalyst treatment but also during CNTs growth, which influences the quality, purity and yield of the CNTs (Cantoro et al., 2006; Inami et al., 2007; Bansal et al., 2010).

Temperature is the vital parameter for CNTs synthesis by CVD. Hydrocarbons are decomposing into reactive radical objects in the temperature range of 600–1200°C during CVD. These reactive species diffuse to a catalyst surface where they remain bonded, forming CNTs. The commonly used energy source is resistive heating (Lee et al., 2001; Gohier et al., 2008; Chiangga et al., 2009).

Two processing system patterns, namely horizontal and vertical, are generally used to synthesise CNTs using CVD. A typical horizontal system has been demonstrated because of its versatility. Floating and fixed-bed catalyst is used in the horizontal system (Figure 2). Floating catalysts are mainly applied when a mixture of reactants and catalyst are in the gas phase in the reactor at an elevated temperature during CVD. The catalyst in the gas phase is transformed by the redox gases and/or the elevated temperature, forming solid-phase nanoparticles where the CVD reaction occurs. This method overcomes the obstacle that prevents the coalescence of nanoparticles and reactants. The solid catalyst nanoparticles can have adequate residence time for CNTs to grow when they hold on to the reactor surfaces. In other words, any unreacted gas precursor and solid catalyst nanoparticles, which cannot react and hold on the reactor surfaces at adequately elevated temperatures, are swept away from the reactor with the carrier gases and unused reactants. Therefore, this phenomenon reduces the process efficiency (Zhou et al., 2003; Chena et al., 2006; Maghrebi et al., 2009).

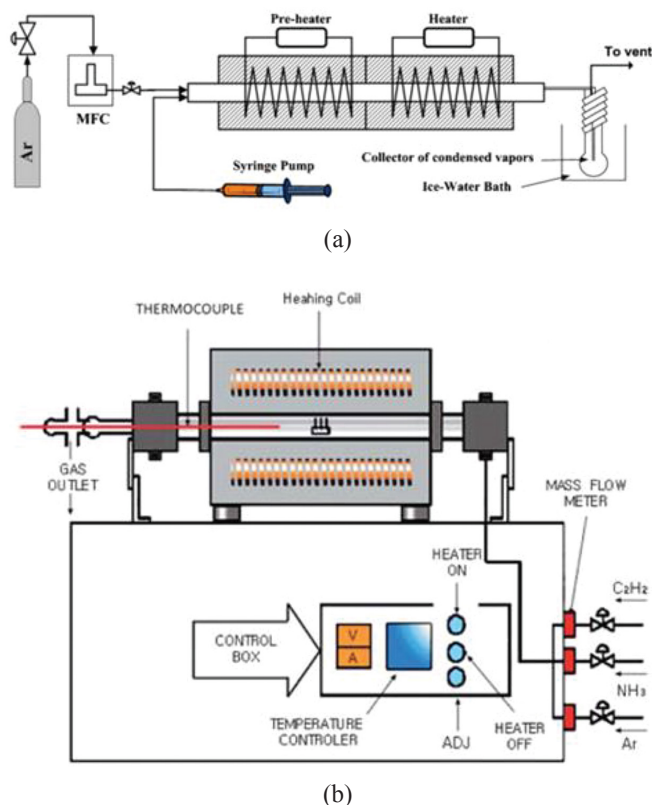


Figure 2. Horizontal CVD: (a) Floating technique; (b) Fixed bed technique (Maghrebi et al., 2009; Nessim, 2010).

By contrast, the catalyst in the fixed bed process is in the solid phase. The catalyst is set in boats and placed inside the reactor. Then, the gas-phase reactant and a carrier are introduced at an elevated temperature where they react during the CVD. The efficiency and productivity of CNTs growth in this system are limited by the heterogeneous contact between the gaseous state and solid reactant as well as the rise in the gradient temperature of the reactor (Koziol et al., 2011). Increasing nanotubes on the catalyst surface leads to a reduction in the diffusion of carbon precursor to the catalyst nanoparticles, thereby reducing the effectiveness of the catalysts (Nessim, 2010; Izadi et al., 2011).

## CATALYST USED IN THE CHEMICAL VAPOUR DEPOSITION METHOD

Catalysts play an essential role in the synthesis of CNTs during CVD. The quality and yield of CNTs are improved by enhancing the suitable characteristics of the desired catalyst. Transition metals, such as iron (Fe) (Emmeneggera et al., 2003), cobalt (Co) (Chen et al., 2006), nickel (Ni) (Jehng et al., 2007) and their alloys (Flahaut et al., 2005; Niu & Fang, 2006; Policicchio et al., 2007; Lobiak et al., 2015), are the most effective catalysts for CNT synthesis. These catalysts can grow CNTs in three steps according to the vapour–liquid–solid mechanism. First, a gas precursor produces carbon, which absorbs and dissociates on the surface of the catalyst particles to form elementary carbon atoms. Second, the carbon atoms dissolve in the mass of nanoparticles to form liquid metastable carbide and diffuse within the particles. Finally, solid carbons precipitate at the backside of the nanoparticles to form CNTs.

Two reverse processes cause CNTs growth. The first process is the well-known “root growth model,” which occurs when the catalyst is strongly bound to the substrate, resulting in a closed carbon network. This characteristic then gives rise to the metal in the form of a seamless graphite cylinder. A second process, which is also known as the “tip growth model,” occurs because of the weak contact between the catalyst and the substrate, and CNTs precipitation pushes the metal particles up from the substance. Therefore, diffusion occurs under the metal particle, and the CNTs continues to elongate (Figure 3) (Kukovitsky et al., 2000; Zhao et al., 2006; Kumar, 2011; Bathgate et al., 2012; Purohit et al., 2014). Solid organometallobenes, such as nickelocene, cobaltocene and ferrocene (Malek Abbaslou et al., 2010; Zhang et al., 2011; Zhu et al., 2014), are extensively used as catalysts because they deliver solid metal nanoparticles, which affect the decomposition of the hydrocarbons. Experimentally, catalyst particle size affects the diameter of the CNTs. Figure 3(c) summarises the effects of the diameter of the catalyst on the structure of the utilised nanocarbon. Catalyst nanoparticles with 1 nm diameter mainly produce SWCNTs, whereas MWCNTs are produced from catalyst nanoparticles with diameters in the range of 10–50 nm. The number of MWCNT layers increases with catalyst diameter. Additionally, another nanocarbon structure, i.e. the nano-onion, is produced when the diameter of the catalyst nanoparticles exceeds 50 nm (Gore & Sane, 2011).

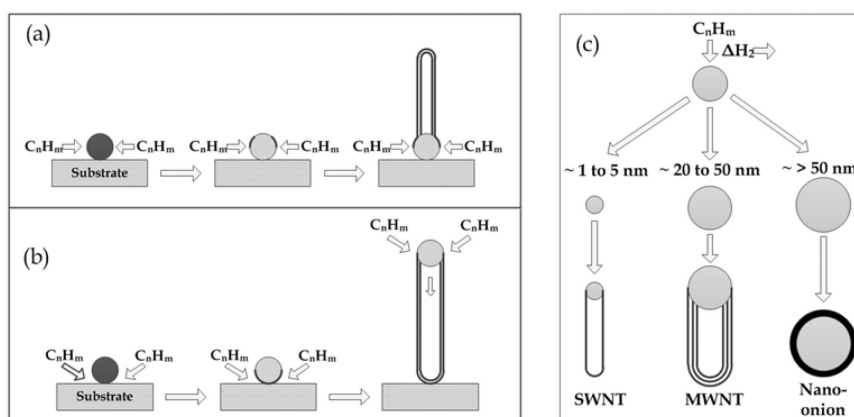


Figure 3. Mechanisms for carbon nanotube growth; (a) Base growth; (b) Tip growth; and (c) Structural dependence on catalyst particle size (Gore & Sane, 2011).

Hydrocarbon decomposition on the catalyst surface, that liberates and prevents aerial decomposition, is the key to obtaining pure CNTs. Furthermore, alloys have been proven to gain higher catalytic activity compared with pure metals (Lobiak et al., 2015). Iron (Fe), cobalt (Co), and nickel (Ni) nanoparticles are effective catalysts for CNTs synthesis. Other metals, such as copper (Cu), gold (Au), palladium (Pd), and platinum (Pt), have also been discovered to be catalysts for CNT growth from a variety of hydrocarbons (Lee et al., 2000; Seung-Yup et al., 2005; Takenaka et al., 2009; Yang et al., 2012; Asokan et al., 2015).

## HYDROCARBON DECOMPOSITION AND THERMODYNAMICS

Thermodynamics are useful in elucidating the reaction during CVD. The reaction needed to create solid CNTs is thermodynamically useful under the selected temperature and pressure conditions. Gibbs free energy ( $\Delta G$ ) is a key to extract this information.  $\Delta G$  depends on the reactivity of the hydrocarbon during pyrolysis. Methane decomposition is thermodynamically preferable above 600 °C because it is the most stable hydrocarbon molecule. By contrast,  $\Delta G$  for ethylene, acetylene, or benzene is already negative at 200°C (Figure 4). CNTs are frequently synthesised by CVD using methane as a precursor at a temperature above 850°C because of the slow reaction rate. Meanwhile, CVD synthesis of CNTs using acetylene is often performed at 500°C - 750°C, and 650°C - 850°C for ethylene (See & Harris, 2007; MacKenzie et al., 2010; Tessonnier & Su, 2011).

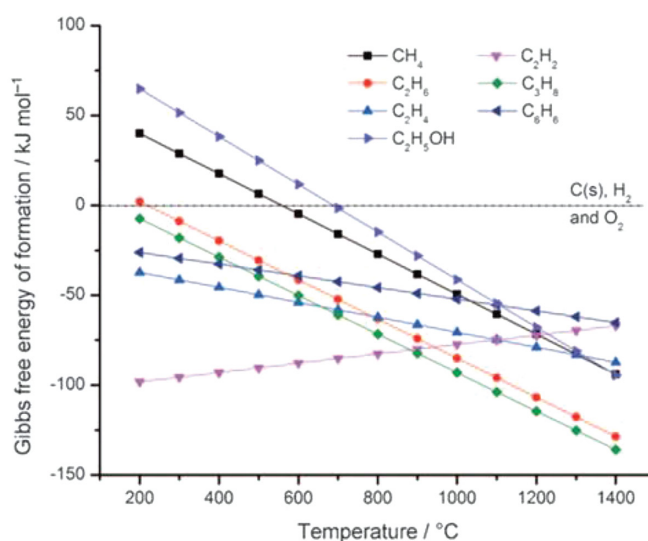


Figure 4. Thermodynamic data calculated using ChemKin database. Gibbs free energies of formation of various carbon precursors. The energies are normalised to the number of carbon atoms in the precursor and correspond to its pyrolysis (Tessonier & Su, 2011).

## HYDROCARBON PRECURSORS FOR SYNTHESIS CARBON NANOTUBES

The most common hydrocarbon sources used in CNTs production by CVD are derived from either petrochemical products (Matar & Hatch, 2001), such as methane, ethane, acetylene, and cyclohexane, or petroleum products (Jones, 2006) such as natural gas, kerosene and diesel oil (Table 1).

Table 1  
Hydrocarbon sources for the production of CNTs by CVD method

| Petrochemical Products |                                   |                  |                  |                                 |                 |                         |
|------------------------|-----------------------------------|------------------|------------------|---------------------------------|-----------------|-------------------------|
| Hydrocarbon Source     | Catalyst                          | CVD type         | Temperature (°C) | Carrier gas                     | CNT type        | Ref.                    |
| Methane                | Co-Mo-MgO                         | CVD              | 1500-1750        | N <sub>2</sub> , H <sub>2</sub> | SWCNT           | Izadi et al. (2011)     |
| Methane                | Co-Mo/MgO                         | CCVD             | 800              | N <sub>2</sub>                  | MWCNT           | Hata et al. (2004)      |
| Ethane                 | Fe/Al <sub>2</sub> O <sub>3</sub> | CVD              | 750              | H <sub>2</sub>                  | MWCNT           | Maghsoodi et al. (2010) |
| Acetylene              | Fe/ SiO <sub>2</sub>              | CVD              | 690              | N <sub>2</sub> , H <sub>2</sub> | MWCNT           | Allaadini et al. (2015) |
| Acetylene              | Co-Mo/MgO                         | CVD              | 800              | H <sub>2</sub>                  | SWCNT,<br>MWCNT | Zhan et al. (2011)      |
| Hexane                 | Ferrocene                         | Two stage<br>CVD | 140, 900         | N <sub>2</sub> , H <sub>2</sub> | MWCNT           | Tran et al. (2007)      |
| Cyclohexane            | Ferrocene                         | ACVD             | 800              | N <sub>2</sub> , H <sub>2</sub> | MWCNT           | Kishore et al. (2012)   |

Table 1 (continue)

| Petrochemical Products  |                      |                   |                  |                    |              |                          |
|-------------------------|----------------------|-------------------|------------------|--------------------|--------------|--------------------------|
| Hydrocarbon Source      | Catalyst             | CVD type          | Temperature (°C) | Carrier gas        | CNT type     | Ref.                     |
| Benzene                 | Ferrocene            | Spray Pyrolysis   | 900              | Ar                 | MWCNT        | Escobar (2007)           |
| Toluene                 | Ferrocene            | CVD               | 850              | Ar                 | MWCNT        | Altalhi et al. (2010)    |
| Xylene                  | Ferrocene            | Floating catalyst | 750              | Ar, H <sub>2</sub> | MWCNT        | Kunadian et al. (2009)   |
| Petroleum Products      |                      |                   |                  |                    |              |                          |
| Hydrocarbon Source      | Catalyst             | CVD type          | Temperature (°C) | Carrier gas        | CNT type     | Ref.                     |
| Natural Gas             | Fe-Mo/MgO            | CCVD              | 950- 1000        | Ar, H <sub>2</sub> | CNT          | Tapaszto et al. (2005)   |
| Propane                 | Fe/ SiO <sub>2</sub> | CCVD              | 600- 1100        | Ar, H <sub>2</sub> | SWCNT, MWCNT | Bachmatiuk et al. (2008) |
| Liquefied petroleum gas | Ferrocene            | Two stage CVD     | 800              | Ar, H <sub>2</sub> | MWCNT        | Huang et al. (2008)      |
| Kerosene                | Fe, Ni               | Two stage CVD     | 200, 1000        | N <sub>2</sub>     | CNT          | Altalhi et al. (2010)    |
| Diesel oil              | -                    | Modified CVD      | 950              | -                  | SWCNT        | Kunadian et al. (2009)   |
| Coal gas                | Ferrocene            | Two stage CVD     | 200, 950         | -                  | SWCNT        | Qiu et al. (2004)        |
| Heavy oil residue       | Fe, Co, Ni, Au, Pt   | CVD               | 900              | Ar, H <sub>2</sub> | SWCNT        | Li et al. (2012)         |
| Asphalt                 | Ferrocene            | CVD               | 1000             | Ar, H <sub>2</sub> | CNT          | Liu et al. (2007)        |

The carbon precursor plays an important role in the growth, characteristics and properties of CNTs because of their binding energy, type and the role of reactive groups, as well as thermodynamic properties. CNTs growth efficiency using gaseous carbon precursors depends strongly on the concentration and reactivity of gas-phase intermediates produced simultaneously with reactive radical species as a result of hydrocarbon pyrolysis. Therefore, the most capable intermediates with ability of physisorption or chemisorption on the catalyst surface to initiate CNTs growth must be produced in the gas phase (Mizuno et al., 2005; Prasek et al., 2011). A comparison of CNTs characteristics showed a relationship between the chemical structures of hydrocarbons and CNTs formation ( Lee et al., 2003; Li et al., 2004). Hernadi et al. (2000) affirmed that unsaturated hydrocarbons have much higher yield and deposition rate than saturated gases. Additionally, saturated hydrocarbon gases produce highly graphitised filaments with fewer walls compared with unsaturated gases. Consequently, saturated hydrocarbons are favoured for SWCNTs growth, whereas unsaturated hydrocarbons are favoured for MWCNTs (Hernadi et al., 2000). Nevertheless, SWCNTs have been obtained from a highly diluted unsaturated hydrocarbon (Q. Li et al., 2004; Yun et al., 2006; Zheng et al., 2006; Escobar et al., 2007). The growth of clean SWCNTs was observed at relatively low temperatures using

alcohols with various catalysts. Thus, alcohols are much better carbon sources for SWCNTs than hydrocarbons. This phenomenon is probably due to the ability of OH radicals to etch away amorphous carbon deposits (Li et al., 2007; Zhu et al., 2012; Ren et al., 2014).

Most studies have shown that low-temperature CVD at approximately 600°C - 800°C produces MWCNTs, whereas high reaction temperatures of 800°C - 1200°C favour SWCNTs. The results indicate that SWCNTs have the higher energy of formation, caused by their small diameter, and high curvature, which tolerate the high strain energy. Therefore, SWCNTs grow from only selected carbon source, such as carbon monoxide, and methane, which has an equitable stability at high temperature. By contrast, common effective precursors for MWCNTs such as acetylene, benzene, and xylene are unstable at high temperatures, resulting in a large deposit of amorphous carbon (Niu & Fang, 2006; Loebick et al., 2009; Bahgat et al., 2011; Gallego et al., 2011; Monthieux, 2011; Atchudan & Pandurangan, 2012; Abdullahi et al., 2014).

Hata et al. (2004) synthesised highly efficient and pure SWCNTs by water-assisted ethylene on the substrate using CVD. The controllable rate of steam into the CVD reactor functioned as a mild oxidizer, leading to the selective removal of amorphous carbon without adversely affecting the growth of CNTs (Hata et al., 2004). Controlling the relative rate of ethylene and steam was essential to minimize the poison of the catalyst. These studies ascertain that carbon precursors play an important part in CNTs growth. Accurate selection of carbon precursor and water vapour rate can optimise the growth and lifetime of CNTs, thereby increasing the quality and yield of these nanoparticles (Zhong et al., 2009; Saengmee-anupharb et al., 2011).

Hydrocarbons, such as methane (Maghsoodi et al., 2010; Allaedini et al., 2015), ethylene (Tran et al., 2007; Zhan et al., 2011), acetylene (Hayashi et al., 2011; Kishore & Pandurangan, 2012), benzene (Narkiewicz et al., 2010; Liu et al., 2011), cyclohexane (Chena et al., 2006; Bachmatiuk et al., 2008), toluene (Altalhi et al., 2010; Gspann et al., 2014) and xylene (Kunadian et al., 2009; Yah et al., 2011) are among the most commonly used CNTs precursors. MWCNTs are typically grown from the decomposition of benzene at 1100°C and acetylene at 700°C, with Fe nanoparticles as catalyst. MWCNTs have also been grown from various other hydrocarbon precursors such as cyclohexane and fullerene (Maruyama et al., 2003). MWCNTs have also been synthesized from supercritical toluene at 600°C and ferrocene as growth catalysts. Toluene served not only as the carbon source for the nanotube formation but also as the solvent (Lee et al., 2004).

## HIGH MOLECULAR WEIGHT CARBON PRECURSOR SOURCES

In addition to the well-defined petrochemical products described in section 5, CNTs have also been successfully and systematically synthesised from local fuels, such as kerosene (Pradhan & Sharon, 2002), liquefied petroleum gas (LPG) (Huang et al., 2008), natural gas (Tapasztó et al., 2005), coal gas (Qiu et al., 2004) and asphalt (Liu et al., 2007).

Coiled and open-ended nanotubes are from commercial kerosene using Ni and Fe catalysts via CVD. These metals assist the growth of straight and coiled nanotubes at 1000°C. Increasing the decomposition temperature to 1100°C leads to the formation of carbon nanobeads, which are a spherical, solid and hollow carbon nanomaterial of size 600-900 nm (Pradhan & Sharon, 2002). These nanobeads are rarely stated in literature (Sharon et al., 1998).



SWCNTs were effectively synthesised from coal gas over ferrocene catalyst in a two-stage catalytic CVD technique. The temperature of the first chamber was maintained at 200°C, whereas that in the second chamber was increased to 850–950°C (Qiu et al., 2004). Another study showed that iron carbide–oxide-filled CNTs were successfully synthesised from coal gas by CVD using ferrocene as a catalyst in a two-stage furnace. The effective reaction temperature was 180°C and 950°C for the first and second furnaces, respectively. Transmission electron micrographs show that the CNTs were filled with iron carbide–oxide (Figure 5) (Qiu et al., 2006).

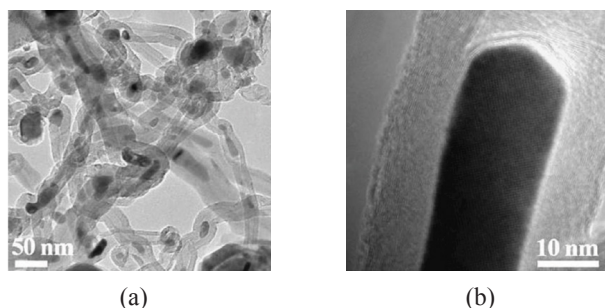


Figure 5. TEM images of iron carbide–oxide filled CNTs obtained via CVD of coal-gas: (a) low-resolution TEM image of partly filled CNTs; (b) high-resolution TEM image of partially filled CNT (Qiu et al., 2006).

Several reports have successfully used LPG as a carbon source for CNT synthesis (Huang et al., 2008; Qian et al., 2002; Zhang et al., 2007; Zhang et al., 2008). Qian et al. (2002) reported the direct synthesis of agglomerated MWCNTs from the pyrolysis of LPG containing sulphur with Fe/Mo/Al<sub>2</sub>O<sub>3</sub> catalyst. The synthesised MWCNTs were similar to that attained from propylene, which is a pure carbon source (Figure 6) (Qian et al., 2002). LPG has been employed as a carbon source to produce CNTs arrays on ceramic spherical surfaces in the floating catalyst process in a two-stage furnace. Improved alignments of CNTs have been obtained, with purity as high as 97.5%. Controlling the growth temperature resulted in CNTs with aligned form and a diameter of approximately 13 nm. Thus, large-scale and low-cost production of CNTs arrays are feasible using synthesised industrial fuel as a carbon source and a ceramic substrate (Zhang et al., 2007).

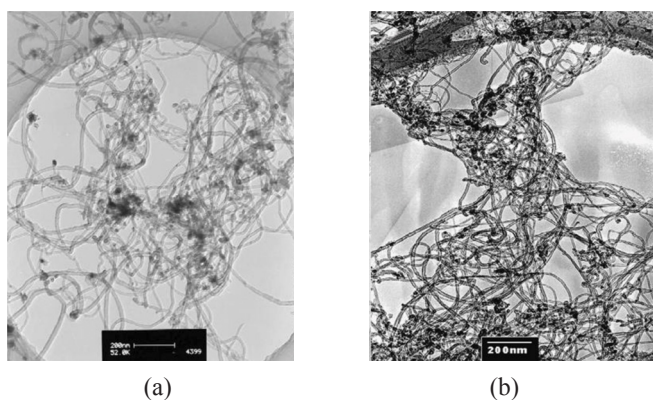


Figure 6. The morphology of as-grown CNTs from: (a) propylene; (b) LPG decomposition (Qian et al., 2002).

CNTs were also reportedly synthesised from heavy petroleum fractions, such as light diesel oil (LDO) and furnace oil (FO) by modifying the CVD method characterised by transmission electron microscopy (TEM) and scanning electron microscopy (SEM). A local reactor was designed to synthesise and collect soot. Collected soot was purified by a Soxhlet extraction apparatus. The purified CNTs were then oxidised with diluted nitric acid. The utilized CNTs were dispersed in different solvents. The dispersed samples were analysed at various temperatures. The results demonstrate that the soot was highly dispersed in distilled water and acetone compared with that in ethanol and methanol. SWCNTs had diameters of approximately 70 nm from FO and 90 nm from LDO (Singh et al., 2011).

In another work, it was reported the synthesis of SWCNTs were synthesised by CVD utilising heavy oil residues as a carbon source. Two categories of metals, namely, magnetic transition metals such as Fe, Co, and Ni, and nonmagnetic metals, namely, Au and Pt, were used as catalysts in the growth of SWCNTs.

The structure and morphology of the synthesised SWCNTs were characterised by Raman spectroscopy, atomic force microscopy, SEM, and TEM. The results demonstrated that high-quality SWCNTs can be synthesised by a CVD method with inexpensive heavy oil residue as the starting material. The diameter distribution of as-grown SWCNTs strongly depends on the type of catalysts. SWCNTs grown from transition metals (e.g., Fe, Co, and Ni) have smaller diameters compared with those synthesised from nonmagnetic catalysts (i.e., Au and Pt). This outcome establishes the feasibility of controlling SWCNT diameters by selecting the catalysts. Additionally, the reaction temperature is the key factor affecting the formation of SWCNTs from oil residues (Li et al., 2012).

The production of CNTs from waste oil has been investigated when Suriani et al. (2015) synthesised for the first time quasi-aligned CNTs from the decomposition of waste engine oil as a carbon source mixed with ferrocene as a catalyst in a two-stage furnace. The synthesis was performed at 500°C and 570°C for precursor and CNTs synthesis, respectively, with a successful ferrocene catalyst ratio of 17.99wt%. Field emission SEM images of quasi-aligned CNTs produced regulated diameters of 18.0 nm to 29.8 nm with lengths from 11.8 µm to 17.1 µm. TEM, SEM, and micro-Raman analysis exposed a dense mixture of quasi-aligned SWCNTs and MWCNTs (Suriani et al., 2015).

## CONCLUSION

The synthesis of various CNTs either single-walled or multi-walled is influenced by the type of hydrocarbon source used in CVD method. The process parameters involved, which are the type of catalyst, carrier gas, reaction temperature and reaction time, also contributed to the different structure of CNTs. However, the type of hydrocarbon precursor is conclusively playing an important role in CNTs growth. Therefore, this review paper looked into the effect of utilising different types of hydrocarbon source derived from petroleum and petroleum products in affecting the formation of carbon structures. Through CVD method, a controlled synthesis ambience for the carbon vaporization and nucleation lead to the formation of CNTs with desired quality and improved production yield. The method also favours solubility of hydrocarbon source and provides the conditions and controlled synthesis, which allows for the substrate-catalyst interaction.

There is a clear parallel between petroleum and petrochemical products such as hydrocarbons sources, suggesting that the decomposition of hydrocarbon source at elevated temperature and diffuses through catalytic particle is a key factor in the CNTs growth.

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