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Synthesis of MWCNTs by Chemical Vapor Deposition from Kerosene

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Abstract

A carbon nanotubes CNTs with less than 7 sheets of Graphene (few walled according to classification of carbon tubes with Nano scales) were prepared by chemical vapor deposition from kerosene as sources of carbon. The prepared carbon nano materials were SP2 hybridization consisting of tubes with nano meter for radius and micro meters in lengths and homogeneous in distributions. The identifications were represented by Raman spectroscopy, FT-IR, and XPS for detect nature of function groups while crystalline of tubular structures was done by X-ray diffraction XRD while stability of structure was achieved with thermogravimetric analysis TGA. The outer morphology and detection outer and inner diameters were taken by electron microscopy with scanning techniques (SEM). The average values were 13 nm and 6 nm for outer and inner diameters respectively and that refers to 4-7 graphene sheets and lengths were more than 1 μm.

Keywords

Tube Furnace,
FWCNTs,
H2O2,
Kerosene, Graphene.

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1. Introduction

carbon nanotubes CNT have been a variety of techniques and modified techniques for producing linear, branching, helixes with single, double, or few and multi graphene sheets [1]. May be the variety of methods were able to produce many types of CNTs and all the techniques characterized by specific properties, but that did not prevent to share a set of essential characteristics which is a source of carbon, a source of heat, and presence of precipitation surface [2]. The specificity for every technique such chemical vapor deposition CVD, Arc discharge AD, and laser ablations LA [3] could be achieve the selectivity and sensitivity for types or nature of tubes and quantities. The common classification of carbon nanotubes includes single SWCNT, double DWCNT, few FWCNT, and multi walled carbon nanotubes MWCNT [3].

The most important characteristic of FWCNTs is combines the average physical and chemical properties of SWCNTs and MWCNTs thus it could replace instead SWCNTs [4]. Many materials were used as a carbon source may be most prominent and cheap oil derivatives were liquid petroleum gas [5] and kerosene [6]. Many literatures focus on the machine that could be produced nanomaterials during decomposition of full such diesel engine. Suzuki and Mori [7] reported that the exhaust gas from a diesel engine forming CNTs with soot from gas oil with three conditions high ethanol fraction in fuel, high sulfur loading, and presence of catalyst sources in the fuel. Another work for Suzuki and Mori [8] were used biodiesel and fatty-acid methyl ester as a source of carbon and energy which blended with ethanol and promoter by added molybdenum/sulfur in mixed fuel to synthesize CNTs during combustion in an engine.

The experiments were used 1-Decanol and n- Dodecane as main fuel were carried out and Ferrocene as a catalyst for growth tubular structure .The results showed that fatty-acid methyl ester and 1-decanol had higher ability to produce CNTs when forming larger CO amount compare with n-Dodecane based fuel even at a low ethanol fraction in fuel. Ntaote and Ezekiel [9] mentions that Diesel, kerosene and candle wax can be used for the production of carbon nanomaterial without a catalyst. The studies confirm the spherical nanomaterials of soot particles forming amorphous and spherically shaped carbon Nano spheres with double walled carbon nanotubes. Wrinkled graphene, and few layer graphene-like Nano carbon was synthesized by Ramya et al. [10] from a facile thermal decomposition and chemical method of kerosene. Carbon nanoparticles (CNPs) were synthesized by Hossain and Islam [11] from kerosene with incomplete combustion.

The SEMs analysis for product shown non-uniform with 20-100 nm in particles size. The crystallography analysis by X-ray differentiation XRDs confirmed large amounts of amorphous carbon material and hexagonal graphite lattice for product. Oukrit et al. reported [12] that uniform morphology and substantial yield of carbon nanotubes can produce by certain amount of ferrocene in a quartz tube reactor through co-pyrolysis of mixture of kerosene and ferrocene. The characterization process Based on TGA and SEM, confirmed that synthesized CNTs with a weight ratio of Ferrocene /Kerosene mixed and preloaded Ferrocene of 1:1 showed better thermal stability and more uniform morphology with an average diameter of less than 100 nm. In our previous works we used kerosene for synthesizing Carbon Black Nanoparticles CBN by flame method [6] and as co-catalyst with sulfur to synthesized MWCNTs from coal by flame deposition method. [13-15]

In this work kerosene use as a source of carbon to synthesis FWCNTs by chemical vapor deposition CVD method with two steps, convert kerosene from liquid to vapor phase before second step which forming fragments of carbon free radicals which responsible to produce FWCNTs.

2. Experimental

2. 1 Materials

Kerosene as sources of carbon was supplied from Al-Dora Refinery-Iraq, which used without any

purification, while Hydrogen peroxide 30% and Methanol with purities 99.85% were purchase from Barcelona-Spain and Hyman, England respectively. The N2 gas cylinder was supplied from Arabian gases company /UAE with purity more than 78%.

2.2 Method of synthesis CNTs

The tube furnace made in chine Lab Tube Furnace with range of heating reach to 1200 deg with heating control the temperature acquires within ± 2°C. Figure 1 shows the unite of boiling for kerosene and tube furnace with N2 gas which behaved as carer gas for kerosene vapor (80 cm3/min). the precipitation was done at 600 C and precipitation time was 15 min. After complete the precipitation switch off the heater of vertical unite and cool down to room temperature. The product was purified as we reported [16]. Briefly 100 mg of synthesized CNTs was dispersed with 50 ml of 30% H_2O_2 at 10 °C and stirring for 1 h, final part of synthesized was reach the product to room temperature before make purification. The process of purification required to shaken the product as it is by separating funnel for 15 minutes three times before separate the precipitation from the solution than 3 hours of drying at 90 C°.



Figure 1: The skim of reactor synthesized FWCNTs

3. Characterization

Raman spectroscopy analysis (infinity Sentara 1 Broker) was done by laser light at 530 nm, and intensity 2m W for 5 lops per 2s from 100-2000 cm^{-1} with resolution equal to 3-5 cm. The Fourier transfer infrared (Shimadzu IRAffinity-1 FTIR) spectra were collected with a resolution of 4 cm^{-1} for scanning from (400 to 4000) cm^{-1} and X-ray diffraction (Riga Rota flex RU-200B) was used to determine the crystalline of synthesized CNTs. X-ray photoelectron Spectroscopy (XPS, PHI5700/6600) with monochromatic radiation from Al Ka aluminum anode and the radiation energy of 1486.6 eV. Scanning electron microscopy SEM (Quanta 450 FEI) was used to shows the morphology of synthesized CNTs with 30 kV and 10 μA for operating voltage and current, respectively. Thermal analysis was conducted using a combined Differential Scanning Calorimetry and Thermal Gravimetric Analysis system (DSC/TGA; Netzsch, STA 449C Jupiter) equipped with a high temperature furnace and a Type-S sample carrier.

Raman spectra were presented in Figure 2a, which characterized by two bands D and G band. The G band at 1581 cm^{-1} is assigned to an E_2g symmetric vibrational mode of graphite layers while the D band at 1347 cm^{-1} is originated from the level of disordered carbons. On the left side of figure3a it can be seen two peaks at 292 and 594 cm^{-1} which can be related to radial birthing mode RBM. The RBMs [17] frequency (ω_r) can depend initial to predict the nanotube diameter d_t by

the relationship [ω_r =224(cm^{-1})/d_t] when diameters between 0.8 and 1.3 nm can give identical and more acceptable value. Raman spectrometers when shows single-grating for cutoff frequencies between 100 and 120 cm^{-1}, absolutely causing limits the range [18] of tube diameters to 2 - 2.5 nm thus mostly refers to FWCNTs due to RBMs cannot show with MWCNTs. Figure 2b, include FT-IR, shows the spectra of the synthesized CNTs with four peaks, the first at approximately 3500 cm^{-1}, characteristic of O–H stretch. A broad peak at 1610 $cm^{-1}$and 1420 cm^{-1} which can be assigned to C=O stretching C–O, respectively [19]corresponding to C,O stretching vibration of the carboxylic acid. The fourth peak at 1110 cm^{-1} refers to the C–O stretch vibration [20]. The XRD analysis in figure 2c, identify peaks that correspond to the graphitic lattice. (002), (100), (004) and (110) at 2θ equals to 26.08°, 43.27°, 52.81° and 77.62° XRD reflections from pristine CNTs respectfully [21] while 200 and 220 at 44.20° and 64.28° can be related to MgO [22] . Figure 2d represent XPS analysis was used to further characterize the nature of C=C graphitic of tubular structure. The XPS can improve the existent of C=C which refer to nanotubes or graphene sheets. The photoemission C1s peaks were studied between 282-292 eV a dominant peak at 284.5 eV regarding the sp2 hybridized C=C bonds in extensive conjugated systems [23-24] which mostly refer to graphene sheet in carbon nanotubes.

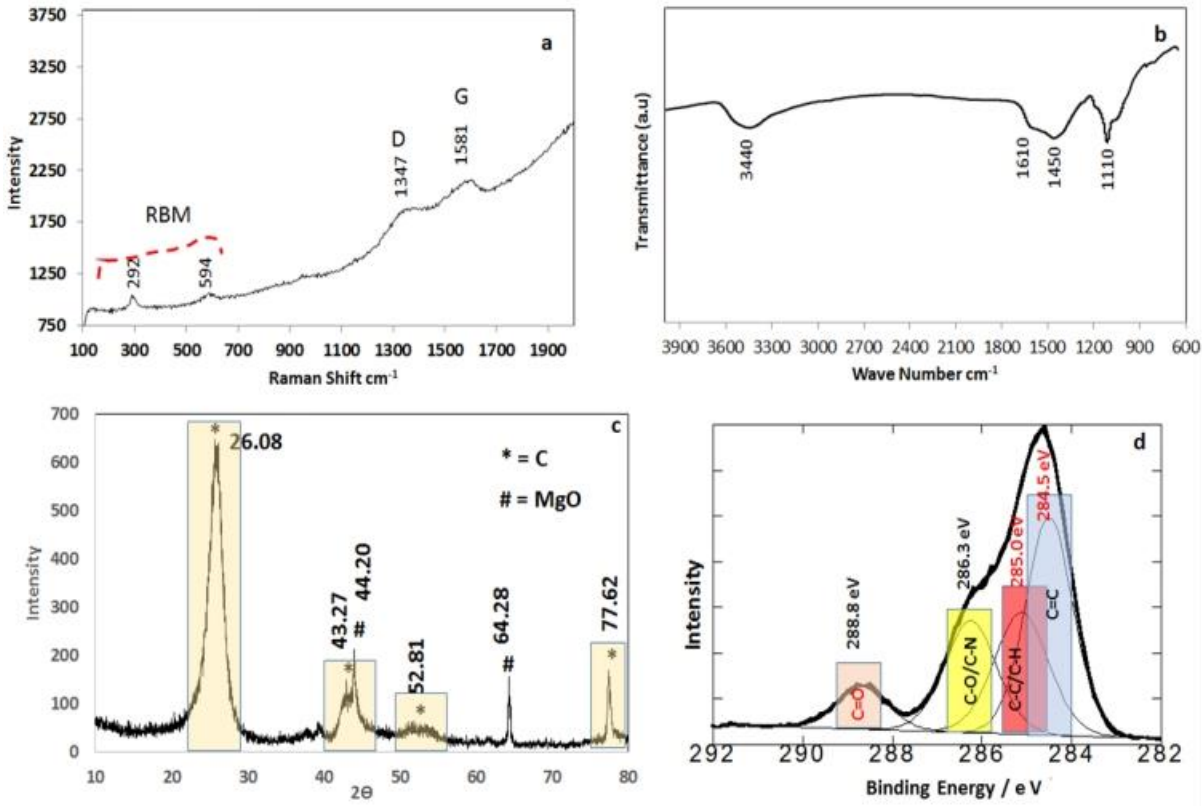


Figure 2: Identification of synthesized CNTs (a) Raman spectroscopy, (b) FT-IR, (c) XRD and (d) XPS analysis of the CNTs produced by Flam/CVD at 280°C from Kerosene.

The peak at 285.0 eV, refers to characteristic of sp3 hybridized C-C bonds and that present at defective locations [25] and asymmetry for tubular structure which related to C-H and C-C [26]. The photoemission peak at 286.3 eV is related to C atoms bonded to O atoms which refers to (C-O) and that mostly bonded with C-N. The second photoemission peak at 288.8 eV is identification for carbonyl groups C=O [27].

The SEM image in figure 3a for synthesized CNTs reveals the homogeneity of the nanotube which appears with high dense bundle tubular structure.

Figure 2b shows the visible and bright bundle with mostly more than micrometers in length. The two images were not observed graphitic species or amorphous carbon indicating the good purity CNTs. The diameters and lengths of the bundles CNTs as shown were determined to be 35–60 nm and about more than 1 µm, respectively, which mostly refer to few walled and multi walled carbon nanotubes. The value of tubular thickness refers to 4-7 sheets of graphene, thus the vast majority of the nanotubes in the synthesized sample are few walled carbon nanotubes FWCNTs [28].

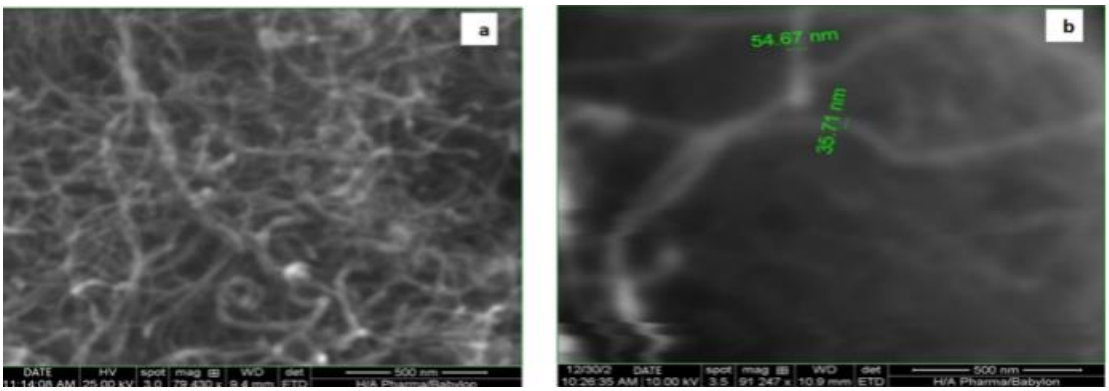


Figure 3: The images for synthesized FWCNTs with (a) and (b) SEM, for synthesized FWCNTs by CVD from Kerosene.

The TGA thermograms and their derivatives thermograms analysis (DTGA) of the synthesized CNTs were reported in figure 4. The thermograms show four parts which shows losing the weight from the sample, the first from 70- 210°C witnessed change in mass 2.03% from the samples may related to evaporation of water and alcohol that may adsorbed during purification [29]. The second reduction in weigh was decomposition of the unconverted or amorphous carbon from 210-370°C [30]. The third step showed reduced 67.92 % from the sample at the range 380-680 °C and that due to CNTs which mostly refer to higher ratios of FWCNTs with less ratios of MWCNTs [31]. The third part shows associated with an endothermic release in the DSC signal, typical of a decomposition/ evaporation at 601.97°C. The fourth part showed losing 13.36% until 910°C and that refer to remains value of the residue sample, mostly related to MgCO3 and MgO [32].

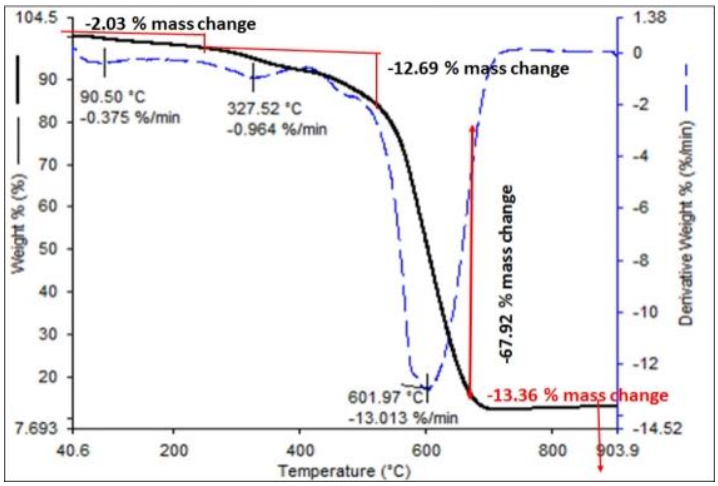


Figure 4: The TGA and DTGA curves of FWCNTs grown with kerosene by Flame/CVDs method at 600°C.

4. Mechanism of growth

Most of growth mechanism focus on the role of catalyst to forming tubular filaments made from carbon [33]. In our work the FWCNTs where growth in the support only without a catalyst, and the process includes two steps: the first convert kerosene in the liquid phase to carbon free radicals in vapor phase FRVP then the second steps which is condensation/precipitation for vapor to solid CNTs.

The cylindrical network after crystallizes can happen in two cases: existence catalyst or without catalyst, as we mention there is only support surface thus the first probability was neglected. The second case refers to our work was compatible with literatures [34-35] and that depend on reaching for crystallizes in the form of a cylindrical network is energetically stable due to no dangling bonds were formed. When the mixture of hydrocarbon after decomposes inter the hot tube, carbon diffuses vertically towards the upper part of tubes, with the time the concentration of diffuses carbon increased. Forming circular center for growing carbon free radical to longer and longer tubular structure [34] on the spot absolutely favorable thermodynamically and kinetically. Krupke et *al.* mentions, the most probability technique is growing-then-place [35] and that required forming nanotubes and then transferring or precipitation to the substrate, which mostly accrued in Arc discharge and laser ablation. Gilbert D. N. [34] enhance this probability when refers to supports this technique [34] due to no limitations or restrictions on the temperature used for forming CNTs just the value of energy to forming FRs and ability to control functionalities nature of CNTs and purify.

The two assumptions for Krupke et *al.* and Gilbert D. N. [34-35] together with our experiment can explain in figure 6, for forming tubular structure of CNTs. The factor that enhances this mechanism, a black precipitate was observed falling from inside the vertical tube of CVDs unite and that explain the purities of synthesized FWCNTs. Perhaps that indicates either weakens interaction between CNTs and support or, forming the tubular structures precedes reaching to support which was clear when easy removed the precipitate from CVDs unite by alcohol.

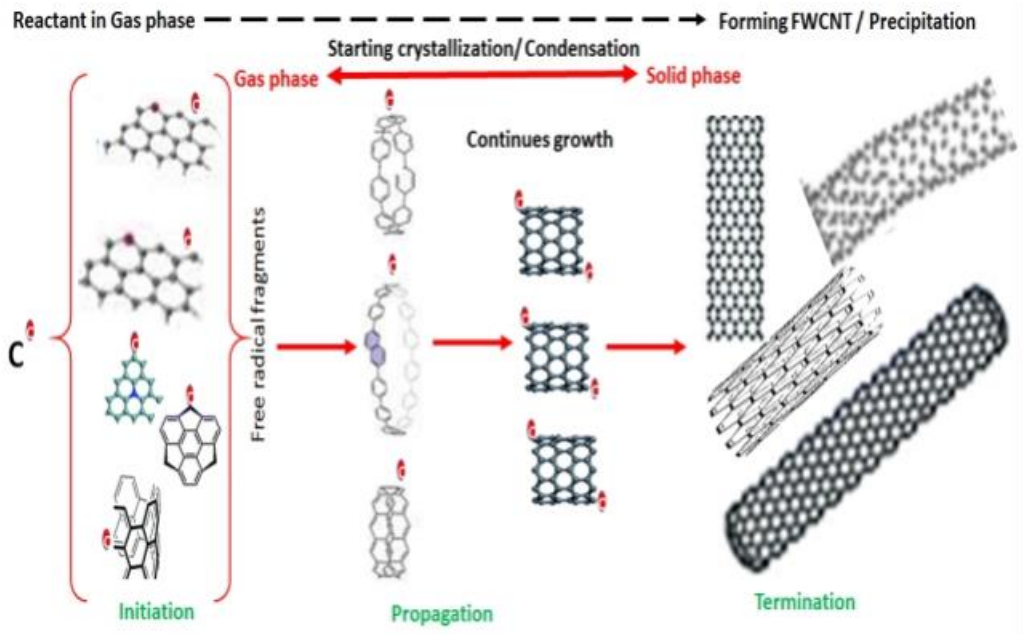


Figure 6: Growth mechanism of FWCNTs on the ceramic substrates heated at 600◦C.

5. Conclusion

This project offers a promising source of carbon, cheap and economical as compared with many sources that used for this purpose towards huge applications in the field of nanotechnology. The controlled precipitation and growth behavior of CNTs by changing the physical phase of carbon sources can achieve the best results. The CVDs method can reduce the complexity and cost of production in addition to remove many critical parameters that could reduce the quality and quantities of CNTs. The SEM images show without doubt, kerosene after flame unite not to form CNTs, tubular structure was formed after entering the vapor to CVDs unit which identified by Raman spectroscopy, FTIR, XRD and XPS.

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