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Vertical Growth Multi- Walled Carbon Nanotubes by the Catalytic Chemical Flame Deposition Method from Coal/Kerosene

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Vertical Growth Multi- Walled Carbon Nanotubes by the Catalytic Chemical Flame Deposition Method from Coal/Kerosene

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Abstract:

Multi- walled carbon nanotubes MWCNT were synthesized with vertical aligned V-MWCNT from mixture coal/kerosene/sulfur by the catalytic chemical flame deposition method CCFD with using homemade reactor. The condition of precipitation was accrued at 413 K in limited atmospheric oxygen with using sulfur as catalyst to enhance precipitation. The process of synthesis showed mixture of Coal/Kerosene did not produce MWCNTs without using sulfur. The product were characterized by X-ray diffraction analysis XRD, Raman spectroscopy, and scanning electron microscopy SEM. The analysis showed that multi-walled carbon nanotubes were growth vertically with length reach to more than 1.75 μm and diameter 40-53 nm.

Keywords: V-MWCNT, homemade reactor, coal, kerosene, sulfur

1-Introduction

Since characterized nanomaterials NMs which [1] showed specific properties compare with materials in classic dimensions, huge attentions focused on the strategy of synthesis NMs towards variety in applications. Carbon one of atoms forming nanomaterials CNM with specific in physiochemical properties such carbon dote, black carbon fullerenes and carbon nanotubes [2]. Carbon nanotubes CNTs specific types of CNMs knows as graphene or graphite allotropes on tubular structure forming graphite nanotubes GNTs and graphene nanotubes gNTs. The classification can be related to number of sheets that forming carbon planar. The GNTs refer to multi-walled carbon nanotubes MWCNT, while single SWCNTs, double DWCNTs and few FWCNTs walled carbon nanotubes represent the gNTs [3]. The common technique for synthesis CNTs include Arc-discharge, laser ablation, and chemical vapor deposition CVD [3]. Commonly these methods are expensive, and complicated due to difficulties of requirements to create conditions for preparation and purification[4]. In flame method FMs, the hydrocarbon precursor is pyrolysis or dissociated with or without suitable metal catalysts which easy for used and expensive [5-8]. The important



requirement for FMs involves combustion of hydrocarbons in the gas or liquid phase [6-7] with using specific chamber for reaction to precipitate CNTs. The solid materials such coal as sources of carbon to synthesized carbon nanotubes in FMs were rarely reported [5-7]. In this work vertical aligned of multi-walled carbon nanotubes V-MWCNTs was synthesized from coal with extent of kerosene/sulfur by chemical flame deposition method CFDM which done by homemade reactor. The synthesized product were characterized by X-ray diffraction XRD, Raman spectroscopy, and scanning electron microscopy SEM.

2-Experimental

2-1 Materials

The coal which used as source of carbon was purchase from local market also kerosene and sulfur were obtained from the local market. Hydrogen peroxide H_2O_2 was purchase from Barcelona, Spain with 60% percent weight.

2-2 Chamber of synthesis V-MWCNT by CFDMs

The reactor homed for applying CFDMs to synthesized V- MWCNT was homemade which explained by the skim diagram in figure 1. The reactor consist of cylindrical chamber with 50 cm in length and 30 cm in radius include three heating coil U tubes Heater, 1.5 kW. The upper side of chamber include thermal cable contact the system to control temperature with accuracy $\pm 1\text{ }^{\circ}\text{C}$.

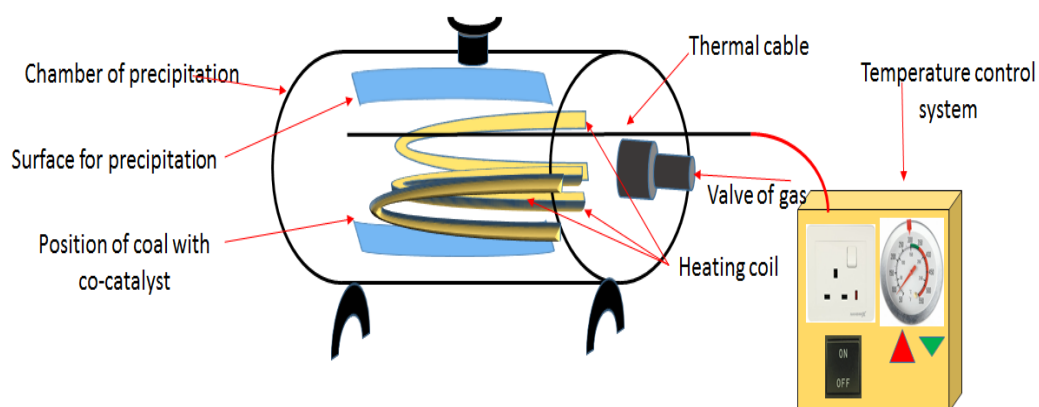


Figure 1: Skim diagram for reactor of CFDM

The process of synthesized CNTs was include mixture coal/kerosene/sulfur with ratio (4 / 1.3 / 1) respectively which put on the lower part of chamber. For the growth of V-MWCNTs, inside a CFDs chamber CO gas supply for 20 min before precipitation than kept temperature at $413\text{ }^{\circ}\text{C}$ for 15 minute. After precipitation the apparatus was switched off before cool the system to starting the purification and characterization.

2-3 Purification procedure of MWNTs:

The synthesized CNTs were purified by hydrogen peroxide as we reported in our previous work [9]. Briefly synthesized CNTs was dispersion with 25 mL of hydrogen peroxide at 20 °C with stirring for three an hour than allowed to reach room temperature with constant stirring. After that shake the product by separating funnel for 15 minutes, then separate the precipitation than thermal treatment at 100 C° for 3 hours.

3-CHARACTERIZATION

A (Riga Rotaflex) (RU-200B) X-ray diffractometer was used to analysis the crystallography of MWCNTs with 0.15405 nm radiation from Cu K α between 10° -90° with 5°/min in a scan rate and 0.02° for resolution. Raman spectroscopy was done by Sentara infinity 1 Broker, intensity 2m W for 5 lops per 2s at 530 nm using light leaser and resolution 3-5 cm. Scanning electron microscopy SEM measurements were carried out by a JEOL JSM-6700F.

Figure 2 and 3 refer to XRD patterns for synthesized CNTs before and after purification respectively. The two figures show two peaks at $\approx 24^\circ$ and 43° which refer to 002 and 100 planar of tubular graphite structure [10] respectively. The peak at 36° can be related to the remaining of Fe which used as support surface of precipitation [11]. The peaks which refer to Fe was reduce after purification with hydrogen peroxide when succeed to remove most of unconverted carbon [11].

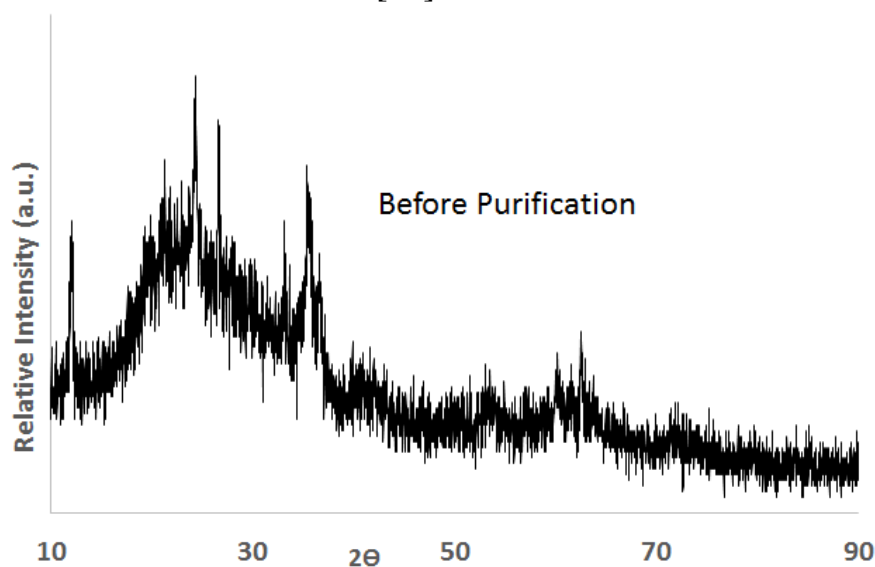


Figure 2: XRD patterns for as prepared CNTs

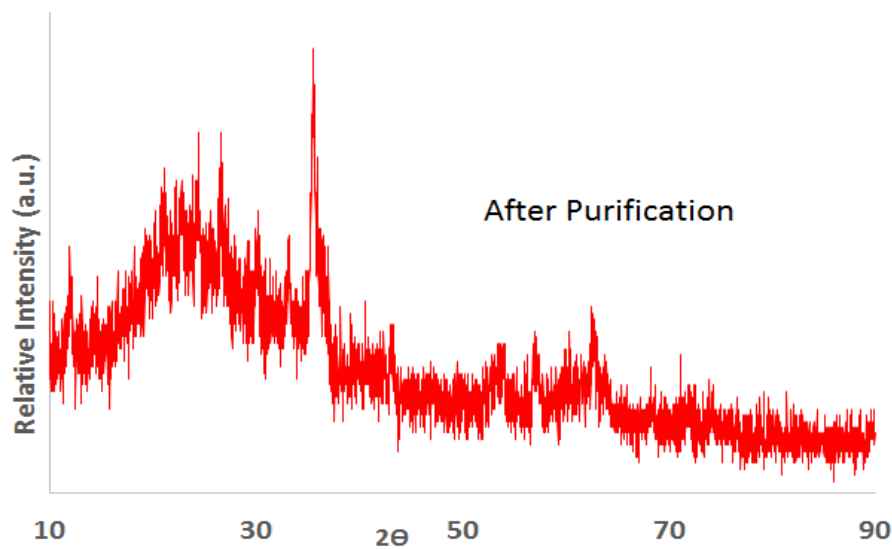


Figure 3: XRD patterns for synthesized CNTs after purification

Figure 4, shows Raman spectrum of a purified CNTs by hydrogen peroxide which showed two peaks at 1357 cm^{-1} and 1597 cm^{-1} due to disorder (D-band) and graphite (G-band) bands, respectively [12]. The ratio of I_G/I_D was 1.75 which refer to high degree of graphite order will less distortion for the multi-walls carbon nanotubes [13].

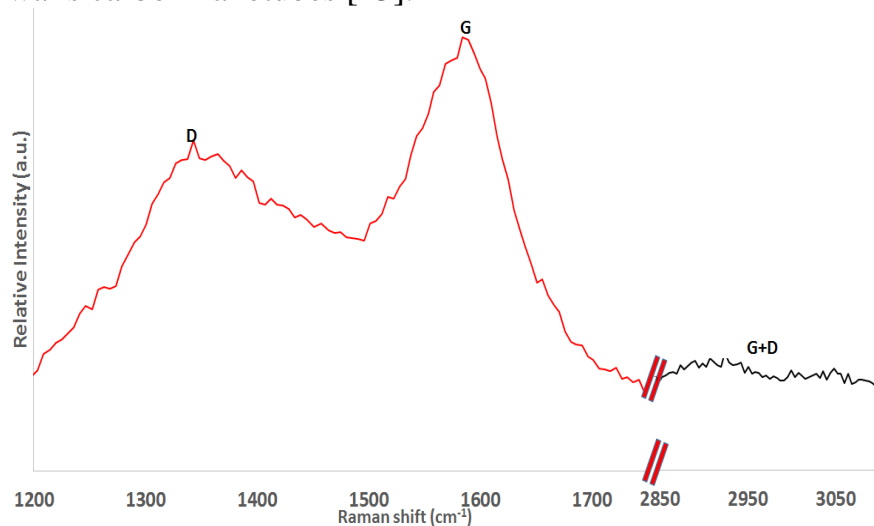


Figure 4: Raman spectroscopy for synthesized CNTs after purification
SEM image of carbon filament were plotted in Figure 5 which shows diameter 40- 53 nm and length reach to more than $1.75\mu\text{m}$ which refer to multi-walled carbon nanotubes MWCNTs. The image shows forest of MWCNTs growth over support of iron in vertical plane.

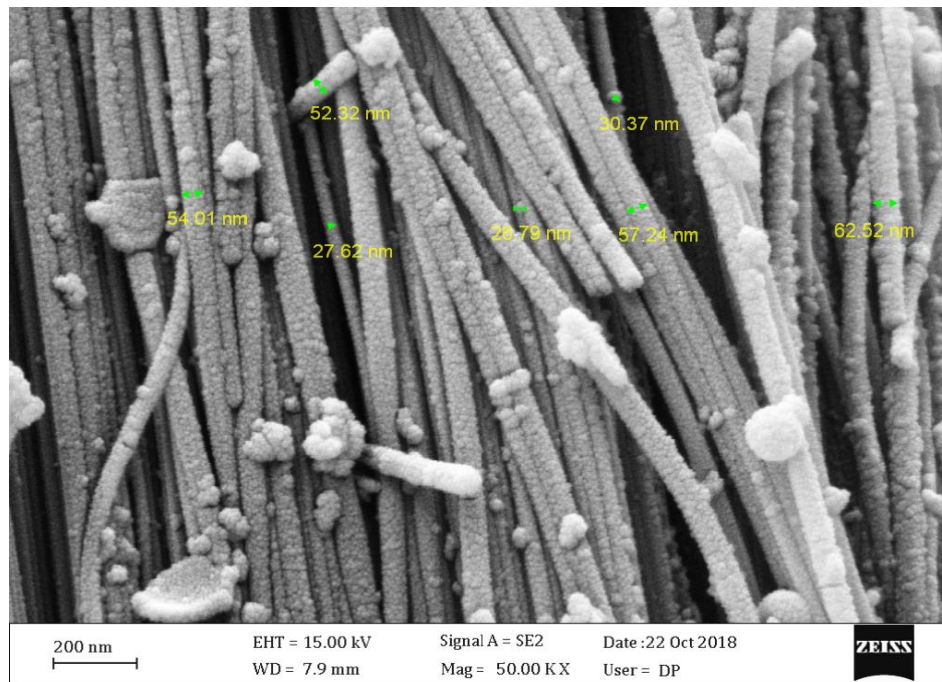


Figure 5: SEM images for synthesized MWCNTs after purification

Figure 6 represent simple explanation for the suppose mechanism. The mechanism of growth CNTs influence with two parameters: temperature of coal ignition and condensation temperature. The first parameter needed for accrue in limited oxygen atmosphere in exist of kerosene and sulfur to forming CO and fragments carbon free radical [6]. The second parameter represent by the ideal condition for precipitation for tubular structure and prevent or at least reduce forming non-tubular structures.

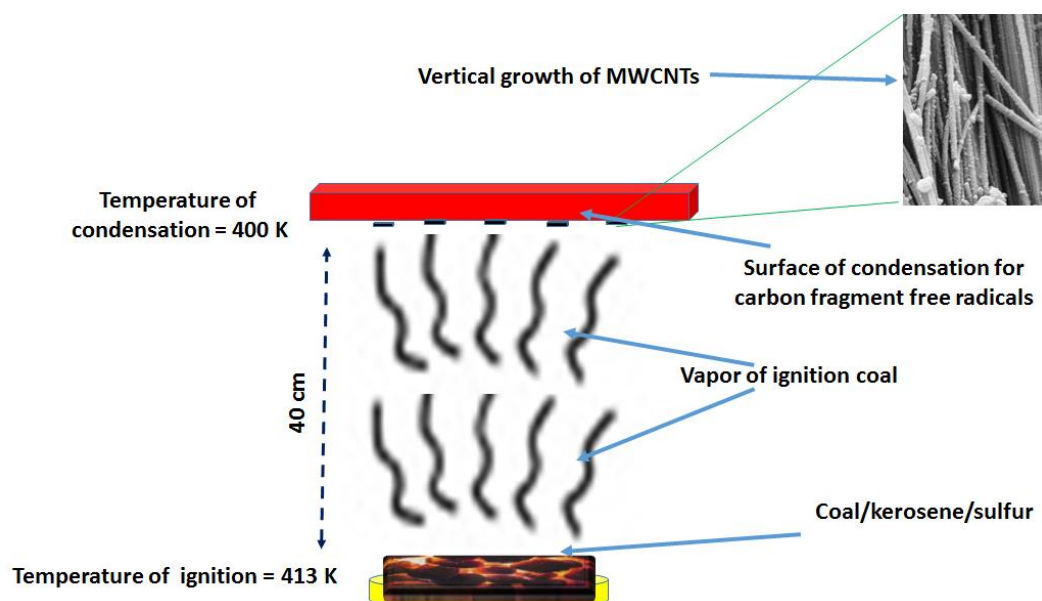


Figure 6: image for ignition of (coal/kerosene/sulfur) mixture and precipitation V-MWCNTs

The conditions which decided the physical form for product such MWCNT and SWCNT or amorphous carbon can be related to nature of support and catalyst particle size with precipitation rate [14]. When the carbon free radicals diffusion in equilibrium with precipitation rate, more regular precipitation for sp^2 hybridization on the support which forming layers. While at low temperature slow diffusion rate as compare with the precipitation rate that enhance sp^2 hybridization on the support to forming tubular structure which mostly MWCNTs due to large area for precipitation [15]. The effect of CO was very important in deposition of CNTs when react with O_2 to reduce it in chamber than enhance forming free radical without inhibition. The direction of diffusion free radical towards upper side of chamber where condensation accrued make the growth vertical for tubes. Generally flame in this conditions may provide an ideal environment for growth of vertical MWCNTs on a large scale.

Conclusion:

For the first time MWCNTs were synthesized by CFDMs from coal as source of carbon in low temperature. SEM images show that the forest of multi-walled carbon nanotubes were synthesized by the CFDM which growth vertically. Two parameter which responsible for the orientations of free radicals towards growth temperature of decomposition for coal and temperature with direction of condensation for free radical. The mixture of sulfur and kerosene were succeed to reduce the temperature of coal ignition which accelerate for higher diffusion rate for tubular precipitation of growth V-MWCNTs by flam method.

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