

# Synthesis of few-wall carbon nanotubes using methanol/ propanol mixture by chemical vapour deposition

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## Abstract

Carbon nanotubes with few walls are highly appreciated for specific physiochemical properties and technological with huge applications. In this paper, we present an alternative process that can lead to lowering the manufacturing in time and cost. The process of synthesized were done by chemical vapor deposition with homemade portable tube furnace reactor. Methanol/ Propanol mixture is utilized as a source of carbon on silica surface caver with carbon black (CB) as support for growth CNTs at two temperature 450 and 750°C. The characterization of FWNTs was performed by X-Ray diffraction, Raman spectroscopy, transmission electron microscopy TEM, Brunauer, Emmett and Teller BET and Thermalanalysis TGA. The main interest of this study are to give the basis of an efficient simple synthesis process to produce CNTs of few walls for removing pollutant by adsorption. The method was succeeded to synthesize FWNT450 which more surface area and more active site than FWNT750 in adsorption properties.

## Introduction

The process of synthesized and purified nanomaterials represent a large challenge due to the cost which is still higher than the micro materials. Large efforts to manufacture nanomaterials for their enormous applications such nano-adsorbents which used to remove pollutants from environmental with high activity and sensitivity [1]. Carbon nanomaterials CNMs one of the new nanomaterials entered the science with all forms such fullerene and tubular structures CNTs [2]. The extraordinary physiochemical properties and less toxicity for carbon nanotubes then fullerene encourage laboratories in all world to study and try to take advantage of these structures [3]. Generally, carbon nanotubes include two form: mono layers tubes or single wall carbon nanotubes SWNTs and multi-layer tubes MWNTs [4]. Recently, few-walled carbon nanotubes FWNTs new class refer to double or triple sheet until seven graphene sheets [5]. They can be considered as an intermediate structure and properties with cost between SWNTs and MWNTs. Arc-discharge, laser ablation, and chemical vapor deposition CVD represent the primary techniques for synthesis CNTs [5]. Chemical vapor deposition is simple and law cost with less complicated as compare with the ether techniques [5]. Many hydrocarbon materials are use as sources of carbon to synthesized CNTs such Methane acetylene, benzene and ethanol [6-9]. Methanol mostly used with the mixture to synthesized CNTs Due to produce high ratios of unconverted carbon, but enhancement effect of the OH radical which evolves from dissociation encourage to use it [4,10]. Vander *et al.* [11] suggested that catalytic behavior can be explained by electron donation to the support when creating d-vacancies, which acts as an active site to exit the hydrocarbon dissociation. Reilly and Whitten [12] completed this view when assuming that the donor or accept electrons enhance the formation and condensation of free radicals. During the pyrolysis of hydrocarbon, the bonds will be broken between the carbon and other atoms to produce different fragments of free radicals allowing for the rapid rearrangement of carbon bonds. The yield of CNTs mostly reduced under the presence of oxygen atoms, on the precursor

molecule, with high-temperature oxidation is possible [13,14]. Mitri *et al.* [15] and Rajard *et al.* [16] studied the effects of temperature on the synthesis of carbon nanotubes. Both two groups confirmed to important facts which should be chosen the optimum temperature for growth specific crystalline, when estimates by finding the ratios  $I_D/I_G$  [15,16]. Use of natural resources and development of reactors are taken as advantageous for the research. Many literatures were reported preparing CNTs from natural sources of modifying CVD method. Nady [17] use rice straw as waste materials and source of carbon with different ratios of ferrocene /Ni. Kure *et al.* [18] reported modified commercial microwave ovens with quartz tubes to synthesized CNTs from Polyethylene. Gonzalez *et al.* [19] doping carbon nanotubes with Nitrogen from Pyridine/ ferrocene as sources of carbon and catalyst respectively, by modified chemical vapor deposition method using a novel two steps thermal technique. Many literatures such mentions previously, include modified strategy for preparation of complicating causing to increase the cost of preparing in addition to pre-treatment for sources of carbon before use. In this work, we use simple homemade portable reactor to synthesized FWNTs from methanol/ propanol as sources of carbon which is cheap and available in the chemical and physical labs. The change in temperature with an effect of free radical was applied to synthesize two kinds FWNTs with various on active site and surface area, which act perfect nano adsorbent materials. The Congo Red as an adsorbent substance was chosen to study the activities of adsorption for both synthesized FWNTs.

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## Experiment

### Materials

The material used in this experiment was methanol (99.93%) from Alfa Assar and propanol (>99%) from Sigma-Aldridge which used without further purification. Black carbon was purchase from Sigma-Aldrich with average pore diameter  $100 \text{ \AA} \pm 10 \text{ \AA}$  and purities >99.95%. The Hydrogen peroxide  $\text{H}_2\text{O}_2$  was purchase from Barcelona, Spain in 30% percent weight. Congo Red (CR),  $\text{C}_{32}\text{H}_{22}\text{N}_6\text{Na}_2\text{O}_6\text{S}_2$ , with formal weight equal to 696, were supplied by from Merck.

### Synthesis of MWNTs

The Few-wall carbon nanotubes FWNTs were prepared by chemical vapor deposition method CVDs as shown in Figure 1 with homemade tube furnaces.

Briefly, the tube furnace consist of two tubes which made from ceramic: the inner tubes with length 120cm and diameter 3cm when the precipitation was an accord. The outer tube was surround to the inner tube with 7cm in diameter and 100cm in length. The length of the thermal cable was 40cm, with an ability to control the temperature up to  $800^\circ\text{C}$  within  $\pm 2^\circ\text{C}$ . The carbon black was used to complete removing the air and the remains act as catalyst for growth CNTs. The black carbon were dissolved in 100 ml of ethanol then dropped inside inner tube. Before switching on the furnace, nitrogen gas was purged to remove the air from all the tube reaction with flow rate  $125\text{cm}^3/\text{min}$ . The furnace was switched on and heated to the reaction temperature. The synthesis was conducted at  $450^\circ\text{C}$  at atmospheric pressure, with a typical reaction time 30 min in a nitrogen atmosphere and a flow of  $125 \text{ cm}^3/\text{min}$ . When the furnace attained the desired temperature the  $\text{N}_2$  gas flow was reduced slowly to a rate of  $80 \text{ cm}^3/\text{min}$ . The  $\text{N}_2$  gas carried the sources of carbon when passing through a vaporizer of the mixture (1:1) methanol/propanol which evaporate at  $70^\circ\text{C}$ . After deposition, the furnace was switched off and allowed to cool down at room temperature under a continuous  $\text{N}_2$  flow. The CNTs that produces from this reaction was labeling FWNT450. The same conditions were done in preparing the second types of FWNTs only change the temperature to  $750^\circ\text{C}$  and labeling FWNT750. The product was collected to characterized then purify for adsorption process.

### Purification process

The process of purification was done as we reported on our previous work without treatment of 5%  $\text{HNO}_3$  [20]. Briefly, (125 mg) of as-prepared FWNT450 and FWNT750 were dispersed with an ultrasonic water bath in 150 ethanol alcohol for 10 min then using separation funnel, to re-distribution the aggregated particles. The lower part was separated and dispersion with 100 ml of hydrogen peroxide  $\text{H}_2\text{O}_2$  at  $15^\circ\text{C}$  for 12h. The solution was diluted by adding 100ml distilled water then allowed to reach room temperature. The mixture was heating gradually to  $50^\circ\text{C}$  for 30 minutes to complete dissociation most of the hydrogen peroxide. Finally, the sample was filtered and washed by distil-water and dried at  $100^\circ\text{C}$  for 12h.

## Results and discussion

### Characterization

The X-ray diffraction (XRD) patterns were done on a (Riga Rotaflex) (RU-200B) X-ray diffractometer using Cu K $\alpha$  radiation (wavelength 0.15405 nm) with a Ni filter. The tube current was 100 mA with a tube voltage of 40 kV. The  $2\theta$  angular regions between  $10^\circ$  and  $80^\circ$  were explored at a scan rate of  $5^\circ/\text{min}$ . For all the XRD analysis, the resolution in the  $2\theta$  scans was kept at  $0.02^\circ$ . Raman analysis was done by using Sentara infinity 1 Broker with using laser light at 530 nm, intensity 2m W for 5 laps per 2s and resolution equal to 3-5 cm. TEM images were performed in a Jeol JEM 2100 operating at 200 kV and equipped with a LaB6 filament, and an EDX Broker X-Flash 5030 detectors. The BET surface area was measured by using Micrometric Auto MATE 23. Thermalanalysis was conducted using a Thermal Gravimetric Analysis system (TGAQ 500 U20.13 Build 39) equipped with a high temperature furnace and a Type-S sample carrier. The TGA experiments were performed by heating from room temperature to  $900^\circ\text{C}$  at a heating rate of  $10^\circ\text{C}/\text{min}$  within  $50\text{ml}/\text{min}$  of dry flowing air.

The process of synthesized was done on the surface of silica covered with black carbon. As-prepared FWNTs, include impurities which mostly represent by amorphous carbon, graphite, and unconverted carbon. Actually, the predicted in impurities can be related to conditions of precipitation which made on the support without using a

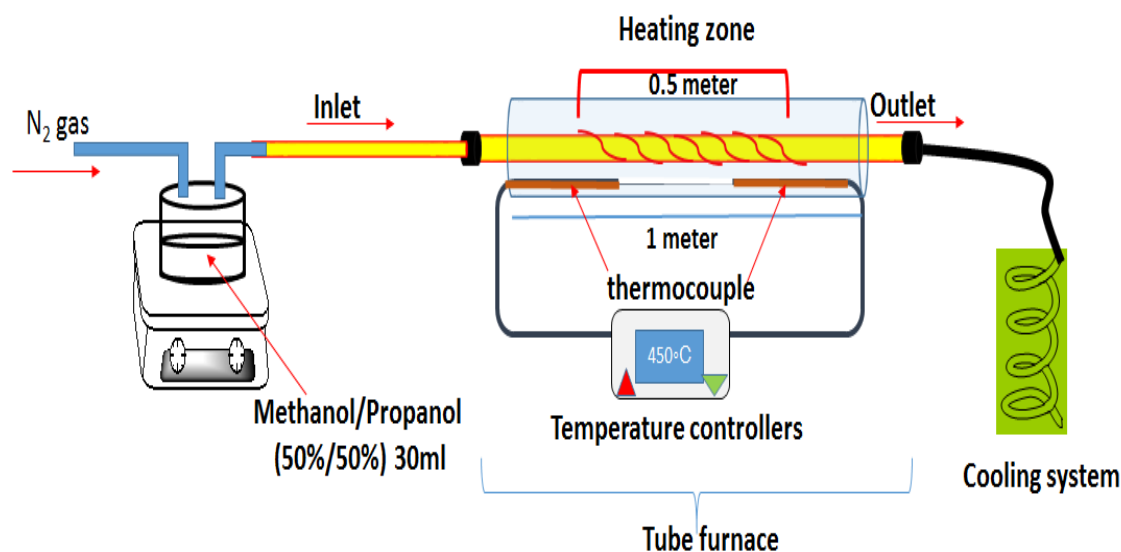


Figure 1. Schematic diagram of tube furnace homemade and reaction system for the synthesis of CNTs by CVD homemade reactor.

metals such as Fe, Mo, Co, or Ni. The XRD pattern in Figure 2, shows two characteristic peaks the first at  $2\theta \approx 25.48^\circ$  and  $25.16^\circ$ , while the second at  $43.11^\circ$  and  $43.78^\circ$  for FWNT450 and FWNT750 respectively. The two peaks can be attributed to the diffraction from the C(001) and C(002) planes of the CNTs which appears more width and shift to high  $2\theta$  with FWNT450, may relate to more disorder [20]. The second peaks at  $2\theta \approx 43^\circ$  were more intense with FWNT750 as compare with FWNT450 which refers to increase the crystalline with increasing temperature. The first peak can be used to calculate inter layer spacing when equal to 0.34 nm for FWNT750 and 0.35 nm for FWNT450.

The Raman spectroscopy in Figure 3, shows the D band, which refers to defect style that required to scatter flexible in order to maintain momentum including  $sp^2$  and  $sp^3$  amorphous carbon. This

style is usually located between  $1250$  and  $1450\text{ cm}^{-1}$  which appear in  $1343$  and  $1362\text{ cm}^{-1}$  for FWNT750 and FWNT450 respectively [21]. The G band were observed at  $1596$  and  $1580\text{ cm}^{-1}$  for FWNT750 and FWNT450 respectively which variances in intensity with D band [22]. The intensity of D to G bands ( $I_D/I_G$ ), equal to 1.34 for FWNT450 while that refer to reasonable crystalline quality of FWNT750 when shows 0.76. Increase the value of  $I_D/I_G$  refers to the distortion on the surfaces of FWNTs that may relate to a low temperature of precipitation [23]. The third weak peaks appear at  $2963\text{ cm}^{-1}$  can be related to overlap with the two peaks G and D on tubular structure which very clear with FWNT750 [24].

Transmission electron microscopy TEM gives a more accurate image of synthesized tubular structure. Figure 3 shows the TEM image

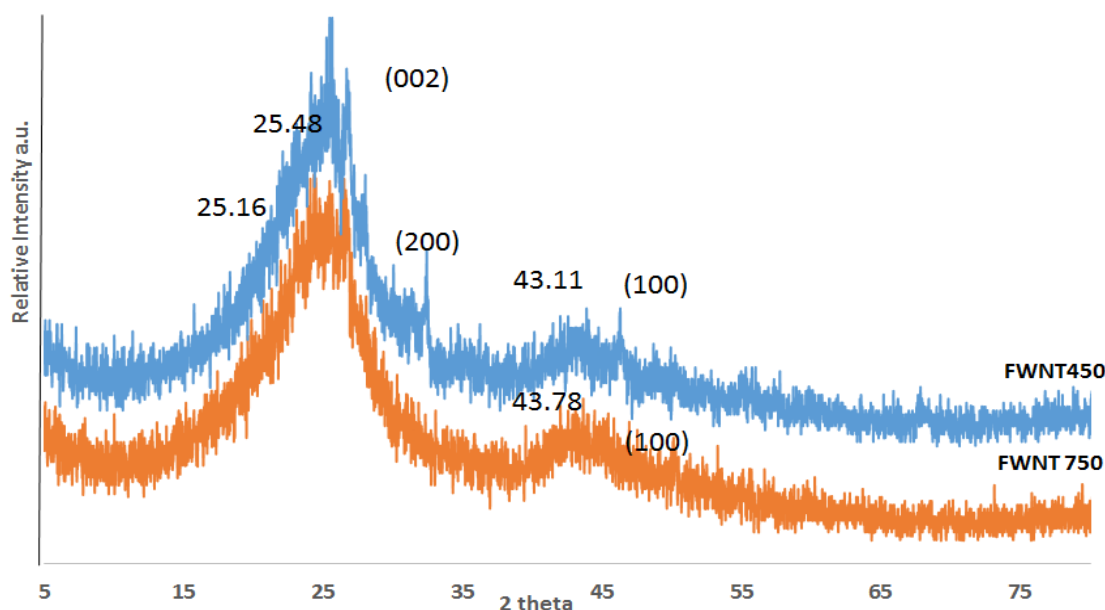


Figure 2. XRD pattern for two types of CNTs which is: FWNT450 and FWNT750 before purification.

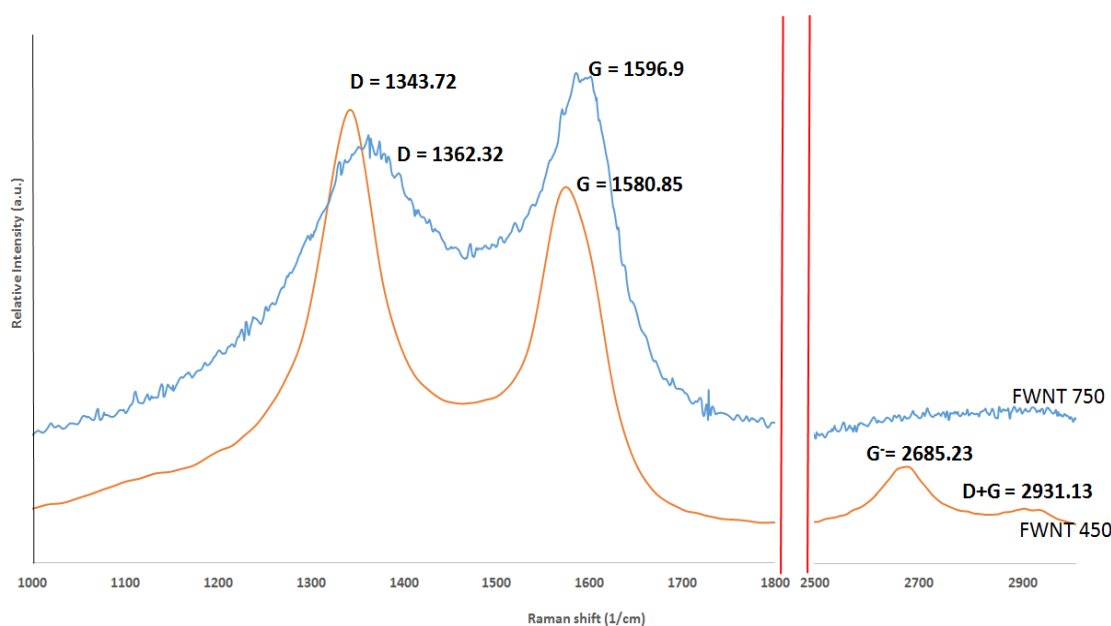


Figure 3. Raman spectroscopy for as prepared FWNT450 and FWNT750.

of FWNT450, the nanotubes have a range of inner diameters varying from (~ 4.21-6.28) nm and outer diameters about (7.49 - 11.80) nm. The thickness of tubes was 3.28-5.52 nm which refers to 2-4 shells with less regular carbon nanotubes with a long reach to 2  $\mu\text{m}$ . The TEM images in Figure 4 and Figure 5, shows many particles that can be related to unconverted and amorphous carbon with silica.

TEM image of Figure 5 for FWNT450 shows many distortion on different regains of tubular structure. The distortion which pointed in Figure 5 refer to defect and deformation that forming the tubular structure which increases with decreases temperature of precipitation [16,22].

The condition of synthesized FWNT750 includes high temperature as comparing to FWNT450 which caused more regular for precipitation.

The image of TEM in Fig 6 shows a tubular structure of outer diameter 7.56-10.48 nm and inner diameter 3.78-5.71 nm. The thickness of tubes was arranged 3.78-4.77 nm which refers to 3 shells of carbon nanotubes with a length of regular tubes reach to 2  $\mu\text{m}$ .

The samples were characterized using, Brusquer, Emmett and Teller BET to measure the specific surface area of the materials. The surface area was 279 and 191  $\text{m}^2/\text{g}$  for MWNT450 and MWNT750 respectively. The variance in the value of surface area can be related to the variance in the nature of surface as shown in Figure 4 and Figure 6.

The best methods to indicate for this interaction is TGA when the temperature at which carbon nonmaterial is oxidized is an index of their stability [25]. From Figure 7 and Figure8 which explains the

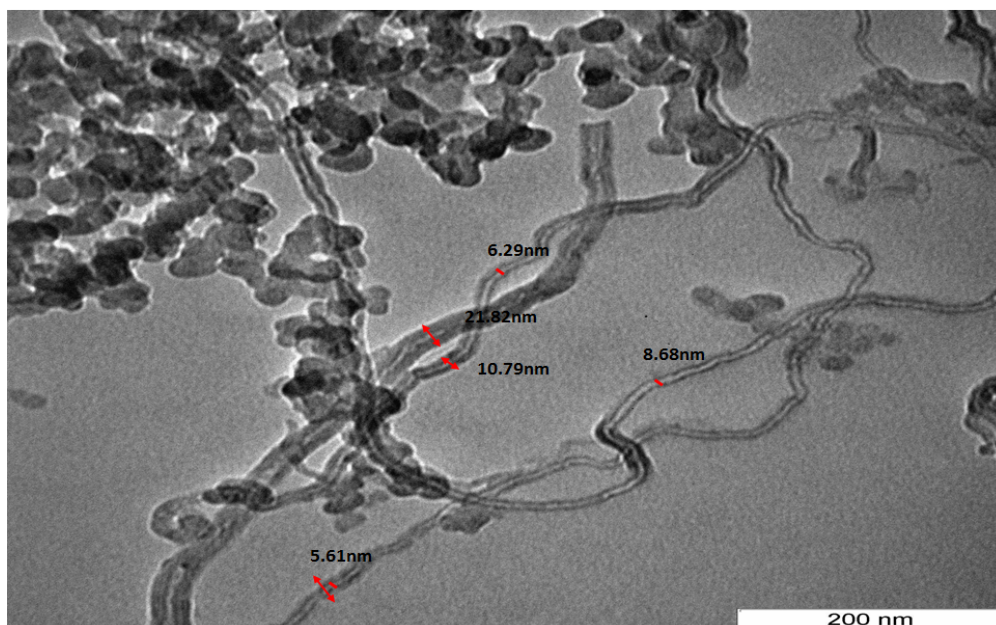


Figure 4. TEM images of as prepared FWNT450 from Methanol/Propanol (1:1) at 450°C.

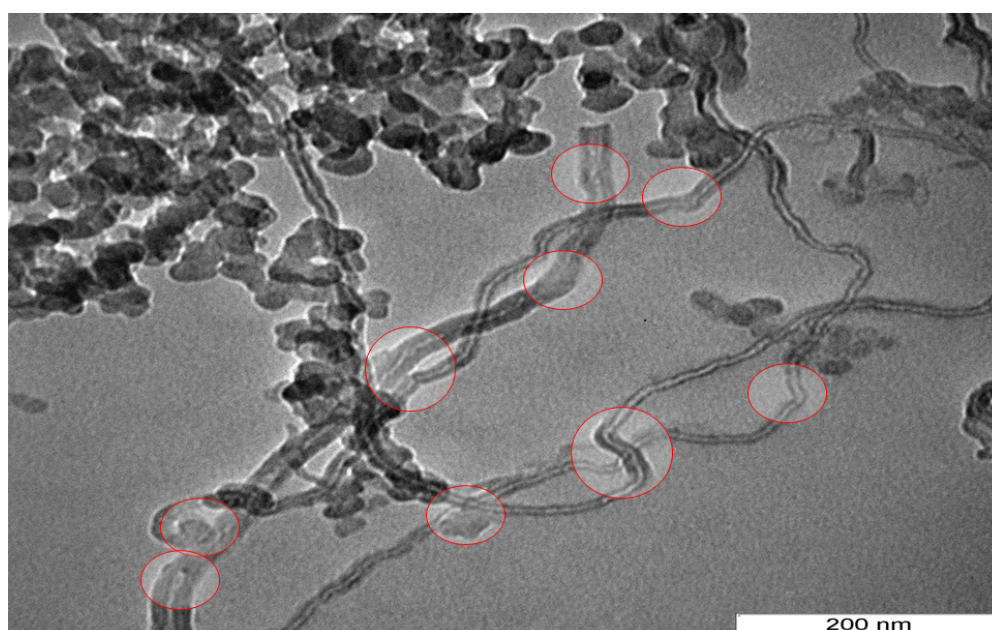


Figure 5. TEM images for synthesized FWNT450 shows many defect and deformation on the surface.



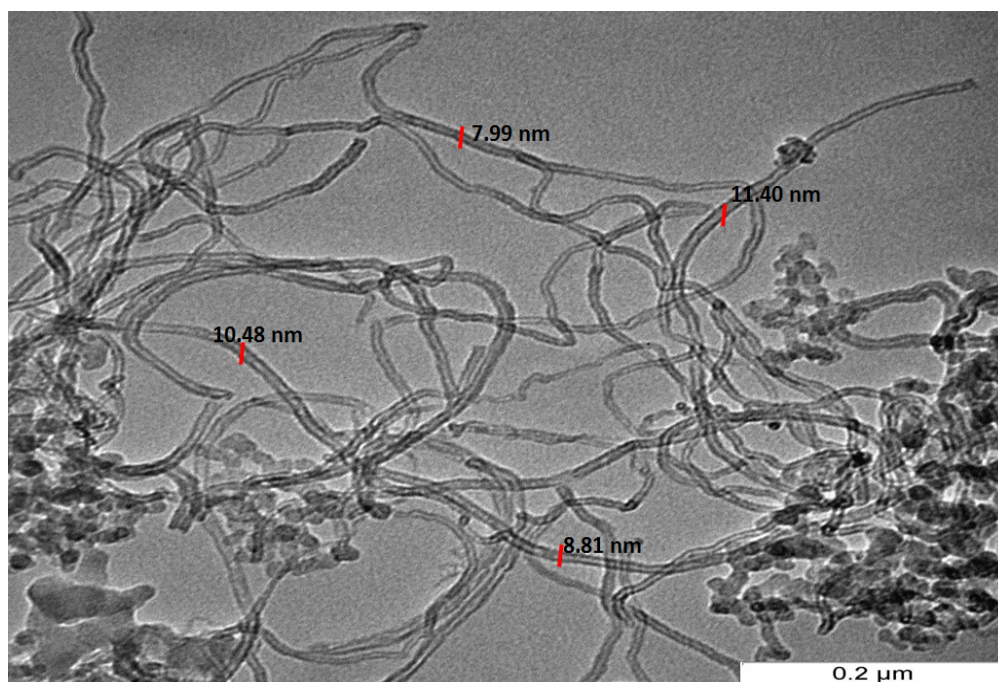


Figure 6. TEM images of as prepared FWNT750.

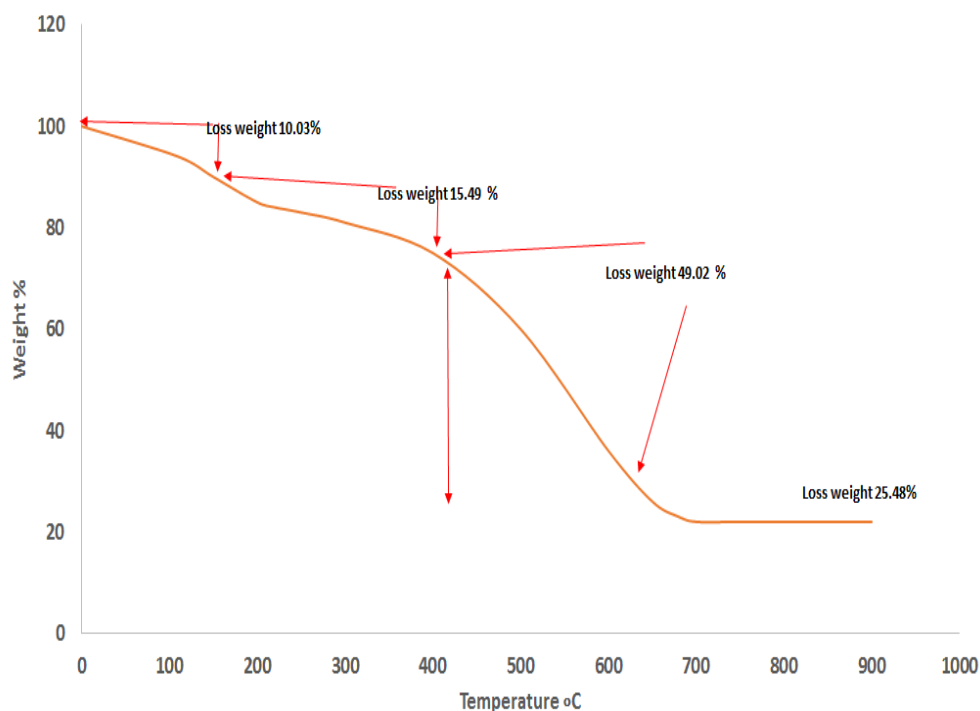


Figure 7. TGA of as-synthesized FWNT450.

identification of synthesized carbon nanotubes with black carbon before purification. The TGA of FWNT450 includes three steps as shown in Figure 7: the first at  $T < 100^{\circ}\text{C}$ , there is 10.03% of mass loss that can be attributed to the evaporation of water [26]. The second steps represent by losing 15.49% on range 100-300°C which related to amorphous carbon which appears after more than 150°C [27]. The last steps after 430°C to 500°C refer to decomposition of synthesized FWNT450 [28-29]. While the remaining materials after decomposition

all the sample represents 21.99% mostly attributed to SiC produce on CVD by reacting the carbon with the support [30]. The ratios of FWNT450 reach to about 49.02%.

Figure 8 shows TGA of FWNT750 which also includes three steps: the first at  $T < 100^{\circ}\text{C}$  when 4.18% loss for adsorption water [26]. The second part from 180 until 470°C shows slow oxidation steps causing loss 8.34% from the sample which refers to unconverted carbon and

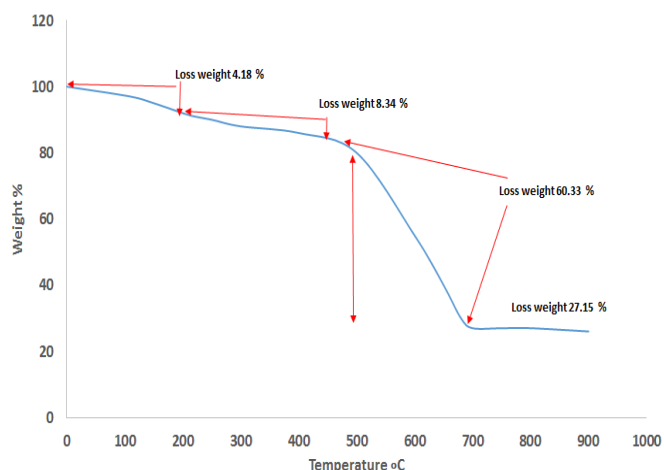


Figure 8. TGA of as-synthesized FWNT750.

amorphous carbon. The ratios 57.67% refer to FWNT750 when shows rapid oxidation in the part of 500-590°C leaving 31.81% of the residue SiC [29,30]. According to TGA analysis, it can be seen the ratios of amorphous carbon for FWNT450 were more than FWNT750 while thermal stability with the yield of product of FWNT750 which the best as compare with FWNT450 [31].

Luiz *et al.* [32] reported that both defect and deformation increase the active site on the surface then make the probabilities to make connections with different species easier. Zhang *et al.* [33] reported that the regular surface of tubular structure reduces the active site and decreases the surface area. Fernando *et al.* Agreement with Mitri *et al.* [15] and Raja R *et al.* [16] when reported that increase temperature of synthesis CNTs Reduce the defect and deformation which mostly decreases the surface area and active sites [1]. Synthesis of carbon nanotubes with support or catalyst influence directly in purities and types of carbon nanotubes [31]. The influence appears due to the interaction between a catalyst or the support material and the particle size of catalyst [32,33]. This reasons influence directly in the number of the wall of product and increased the density of it due to higher density of catalyst and support as comparing with carbon. Furthermore, defect-free CNTs represent highly desirable to limit the application which applied by controlling the conditions of synthesized [33].

**Adsorption test**

Measuring the activity of the FWNT450 and FWNT750 was tested by adsorption 40ppm of Congo Red. The typical adsorption experiments of 40 ppm Congo Red with 60 mg of FWNT450 and FWNT750 in aqueous phases. The pH equals to 5 at 298.15K which needed for  $\lambda_{max}$  equal to 495 nm with using a magnetic mixer at 350 rpm. The required amount of the adsorbent where suspend in 100 cm<sup>3</sup> of an aqueous solution of Congo Red; at various times 2 ml taken from the reaction vessel, and filtered to remove the particles by centrifuge at 4,000 rpm for 10 minutes in an 800 B centrifuge. The Congo Red absorbency was measured at 495 nm, using a Shimadzu Cary 100Bio UV – visible spectrophotometer. The activity was tested by measuring the percent of removal and applied the pseudo-first-order kinetics which represents by equation 1 and 2 respectively [34]:

$$\% \text{ Removal} = [(C_i - C_0)/C_0] * 100 \tag{1}$$

$$\ln(C_0/C_t) = k_1 * t \tag{2}$$

Where  $C_0$  refer to the initial concentration in the bulk solution before starting the adsorption process,  $C_t$  refers to concentration of Congo Red after t min of adsorption. The  $k_1$  is the adsorption rate constant. A plot of  $\ln(C_0/C_t)$  versus t for Congo Red adsorption with FWNT450 and FWNT750 is presented in Figure 9. The value of  $k_1$  can be obtained directly from the slope of the respective linear curves in the Figure 10.

From Figure 9 and Figure 10, the Removal of Congo Red by FWNT450 increased 10% and the rate of adsorption was increased 25% as compare to FWNT750. This result attributed to defect and distortion which increasing the active sites and surface area then caused increasing the adsorbent amount of Congo Red. Many kinds of literature concern with studies the adsorption which explained by function groups or interaction of  $\pi$  bonding with different acid or base materials. In this work, the adsorption accrued due increase the surface area with active site causing FWNT450 which more active than FWNT750.

**Conclusion**

Few-walled CNTs were prepared by CVD method from a binary mixture of methanol/propanol (1:1) at 450°C and 750°C under an N<sub>2</sub>

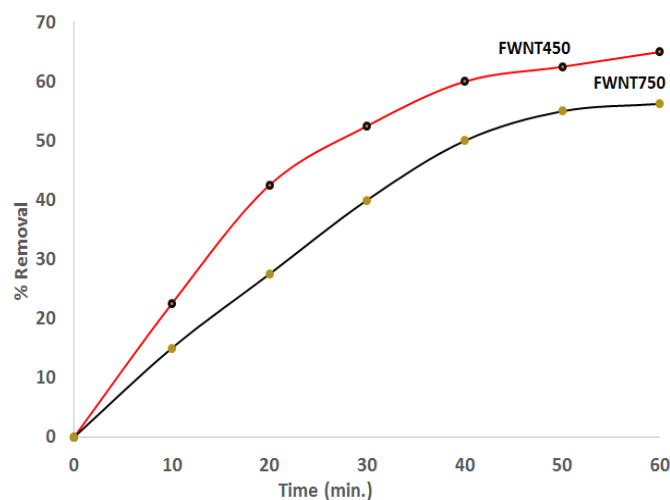


Figure 9. The compare for adsorption 40ppm Congo Red with 60 mg of FWNT450 and FWNT750.

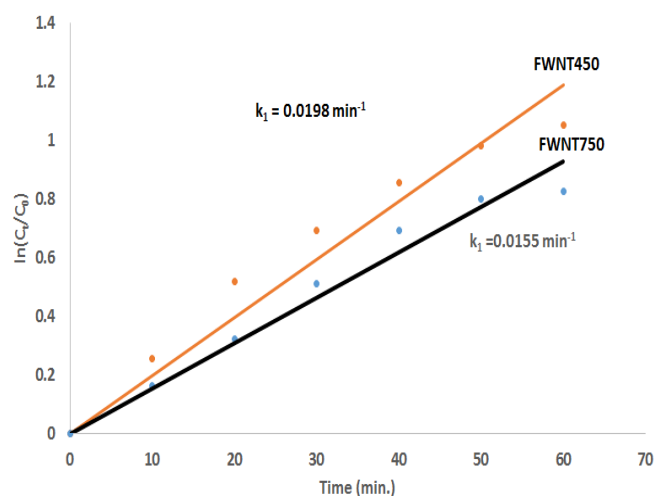


Figure 10. plotting  $\ln(C_t/C_0)$  vs time for adsorption 40ppm Congo Red with FWNT450 and FWNT750.

atmosphere on silica and black carbon as support. The process were applied by homemade tube furnace which succeeded to synthesized two types of FWNTs in short time and low cost. The images of TEM shows that raising the temperature from 450°C to 750°C prevent grown the side branching and eliminate the defect or distortion on the surface of the tubular structure. The advantage of the present synthesis technique is synthesized nano-adsorbent materials by simple conditions. The adsorption activity for FWNT450 represent by the conditions of synthesis which forming many distortions on the surface which increase the active site and surface area.

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