

## Preparation of Carbon Nanotube by Pulse Laser Ablation of Graphite Target in Deionized Water

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### ABSTRACT

In the present work, carbon nanotube was synthesis by pulse laser ablation of graphite target in deionize-water. Fourier Transform Infrared Spectroscopy (FTIR), UV-Visible Spectrophotometer and Transmission electron Microscopy (TEM) were used to study chemical bonding, optical absorption, particle size, structure and morphology of the carbon nanotube. The FTIR absorption peaks at 2092.6  $\text{cm}^{-1}$ , 2150 and 1641.3  $\text{cm}^{-1}$  is due to the  $\text{C} \equiv \text{C}$  and  $\text{C} = \text{C}$  stretching vibration bond represents, which suggest the formation of carbon nanoparticles suspend in this suspension, UV absorption peaks coincide with the electronic transitions corresponding to linear hydrogen – capped polyynes ( $\text{C}_{n+1}\text{H}_2$ ), polyynes are important precursor molecular components in the formation of carbon nanotubes. The TEM show the aggregation of the carbon nanoparticles with size ranges from (9.4 – 70.5 nm), the structure and morphology of the nanotube can be observed with diameters (12.5 -25) nm and length (500 -708.3) nm.

**Keywords:** Carbon Nanotube, Laser Ablation in Liquid, Nanostructure

### تحضير انابيب الكربون النانوية بواسطة الاستئصال بالليزر النبضي لهدف الكرافيت في الماء اللاأيوني

#### الخلاصة

في هذا البحث تم تحضير انابيب الكربون النانوية بواسطة الاستئصال بالليزر النبضي لهدف الكرافيت في الماء اللاأيوني , تم استخدام مطياف تحويلات فورير تحت الحمراء و مطياف الأشعة فوق البنفسجية – المرئية و المجهر الإلكتروني النافذ لدراسة الاواصر الكيميائية و طيف الامتصاص و حجم الجسيمات و تركيب و طوبوغرافية انابيب الكربون النانوية. قمع الامتصاص للأشعة تحت الحمراء عند 2092.6 و 2150 و 1641.3  $\text{سم}^{-1}$  تدل على  $\text{C} = \text{C}$  ,  $\text{C} \equiv \text{C}$  على التوالي . شكلت الاواصر جسيمات الكربون النانوية العالقة داخل المذيب. قمع امتصاص الأشعة فوق البنفسجية مع الانتقالات الإلكترونية تشير الى وجود جزيئات خطية للهيدروجين – بولين والتي تعتبر المفتاح او السلائف الجزيئية في تشكيل انابيب الكربون النانوية. يظهر المجهر الإلكتروني النافذ تكتل

جسيمات الكربون النانوية مع حجم يتراوح ما بين (9.4 – 70.5 nm) ويمكن ملاحظة تركيب و طوبغرافية انابيب الكربون النانوية بقطر يتراوح (12.5 -25 nm) وبطول (500 -708.3 nm) .

## INTRODUCTION

Laser ablation (LA) in liquids presents a unique and fast method to synthesize nanosized objects [1] such as nanoparticles (NP), nanowires and nanotubes directly in suspensions that can be further studied, used or processed into a final material whose functionality requires the presence of the new nanostructure. Via this technique, high energy laser beam, first, heats up the target surface, which is immersed in liquid, up to the melting and even boiling point. Then metallic vapor is generated in the plasma plume immediately. After absorption of latest part of the laser pulse, the plume expands adiabatically; and finally, the condensation occurs, leading to synthesis of nanoparticles [3]. During the condensation, first, nucleation takes place; then fine nuclei collide and form nanosized hard agglomerates as a result of strong covalent or ionic bonds. These hard agglomerates can also stick together by weak Vander Waes or covalent-ionic bonds and bring about soft agglomerates, which can be broken to hard ones by agitation [5]. Only in a short period of time, typically about a few microseconds, do all these steps above happen, and thus nanosized particles are formed [3, 4].

Liquid phase – pulse laser ablation (LP-PLA) technique has been used to produce a variety of materials, including diamond-like carbon films from liquid aromatic hydrocarbons [6], nanocrystals of carbon nitride by ablating graphite in ammonia solution [1] and nanometer-sized particles of Ti, Ag, Au, Si and TiC [8]. Recently, Wang *et al.* [9] Converted hexagonal boron nitride crystals into cubic boron nitride crystals using this method. Nanocrystalline diamond has also been produced by LP-PLA using a graphite target along with water or acetone as the liquid medium [10 and 11]. Interest in these approaches ranges from the possibility of obtaining long chain carbon nanowires (CNWs), thus reaching a new allotropic form of carbon, to a step towards the understanding of the formation of other carbon nano-phases such as carbon nanotubes, fullerenes, carbon onions, etc.

When a graphite target is irradiated in liquid phase, Polyynes, linear carbon chains with alternating triple and single sp - hybridized C–C bonds terminated by atoms or groups (conventionally polyynes represent the class of molecules H

(–C≡C–)<sub>m</sub> H, with (integer m), have attracted considerable interest in astrophysics and nanoscience [12], and are important precursor molecular components in the formation of fullerenes and carbon nanotubes [13]. Including Nano- wires with controllable conductivity [14]. Recently, hybrid Nano-materials have been developed in which polyynes have been inserted into single wall carbon nanotubes [15], and Zhao *et al.*, reported the experimental fabrication of monatomic carbon linear chain which was shown to be inside double-walled carbon nanotubes of 0.7 nm in diameter [16], and their one-dimensional electronic structures have attracted considerable attention due to its size-dependent band gap [17], and nonlinear optical properties [18]. In addition, CNTs have been intensively studied due to their potential technical applications, including field emission displays, hydrogen storage media, sensors, nanometer-sized semiconductor devices, and so on, [19, and 20]. A linear carbon chain with pure sp hybridization is expected to hold the promise for atomic scale field emitting, [21 and 22] and other electronic

devices,[23 and 24] and, therefore, enrich further the characteristics and functions of CNTs[25].

Liquid-phase laser ablation has been used to produce CNTs and polyynes; this method has some advantages. The cost is comparatively low, because expensive instruments such as a vacuum apparatus are unnecessary. The recovery of the product is higher than that of laser ablation in the gas phase. Moreover, ablation surroundings can be easily changed by replacing solvent. This technique is not yet adequate for industrial application, but provides an alternative method to produce CNTs. It has been found that the synthesis in liquid phase bears excellent chemical flexibility and synthetic tenability at normal pressure.

The purpose of the present work is a novel method to prepare long chain carbines and CNTs is reported under normal temperature and pressure by pulsed laser ablation of a graphite target in deionized water at 1064 nm and study the effect of laser energy, laser pulses and irradiation time on CNTs and polyynes production by using FTIR spectroscopy, UV-Vis spectrophotometer, respectively and characterize the particle size, structure and morphology using TEM.

## EXPERIMENTAL

Linear-chain polyynes molecules and carbon nanotubes were produced by laser ablation of a pure graphite pellet that was cut from a graphite rod (99.9 % from national spectroscopic electrodes Co. diameter = 5 mm) was used in a glass Petri dish container filled with 5 ml of deionized water. The Petri dish was rinsed with ethanol cleaner prior to preparation. The target was immersed in deionized water to a depth of around 2 mm below the water surface during laser ablation. The target was rotated constantly during laser ablation for uniform ablation, ensuring that the nanoparticles that formed dispersed uniformly into the deionized water and minimize the target aging effect and to give some stirring effect.

The graphite target was irradiated vertically by an Nd: YAG laser (HUAFEI, 1064nm, 1HZ). The laser energy used in this experiment at range from (20, 80 and 200 mJ/pulse) and the laser pulse ranged from (25-100). The laser beam was loosely focused using lens with focal length of (12 mm) that equal to distance between the target and laser.

The optical properties of the nascent polyynes suspension were examined at room temperature in a quartz cell with 1 cm optical path. By a UV-Vis absorption spectrophotometer (SHIMADZU) operating in the wavelength range of (200-1000 nm), the chemical bonding studied by Fourier Transform Infrared Spectroscopy (FTIR) (8400S, SHIMADZU) were also taken by drying drops of CNPs colloids on NaOH cell and Transmission electron microscopy (TEM) (type CM10 pw6020, Philips-Germany) were used to study the particle size and the morphology, by depositing the CNPs colloids onto copper mesh coated with an amorphous carbon film

## RESULT AND DISCUSSION

### • Ftir spectra

Figure (1) shows the FTIR spectrums of carbon nanoparticles suspensions prepared by laser ablation of graphite target in deionized – water at laser energy (20, 80 and 200 mJ/pulse) with 25 laser pulses. From all figure, notice that the bond for all suspensions is between (3000 to 3600  $\text{cm}^{-1}$ ) is due to the O – H

stretching vibration. The peak at  $2092.6\text{ cm}^{-1}$  and  $\sim 2150$  is due to the  $\text{C} \equiv \text{C}$  stretching vibration bond, while  $\text{C} = \text{C}$  stretching vibration bond appear at  $\sim 1641.3\text{ cm}^{-1}$ , suggests the formation carbon nanoparticles suspend in this solvent. The peak between  $(800 - 1300\text{ cm}^{-1})$  is due to the  $\text{C} - \text{O}$  stretching bonded [26]. According to the results of figure, the relative intensity of FTIR absorption peak is change with change laser energy while the positions and width of observed peak are well constant for the most part in each spectrum, it is inferred that the  $\text{C} \equiv \text{C}$  and  $\text{C} = \text{C}$  bonds was formed by laser ablation of graphite in deionize – water which referred to formation carbon nanoparticles suspend in this solvent [27].

Figure (2) shows the FTIR spectra of carbon nanoparticles suspension preparation at laser energy (80 mJ) with different laser pulse (25 and 100). Also the same vibration bonded of  $\text{C} \equiv \text{C}$  and  $\text{C} = \text{C}$  stretching vibration while the peak absorption of carbon suspension prepared for 100 laser pulse higher than peak absorption at 25 pulse, this due to increased carbon nanoparticles concentration ,which cause scattering of laser light and reduce the absorption of solution and increased the transmission spectrum.

• **The UV-Visible spectra**

The effect of number of laser pulse on the formation of carbon nanoparticles is very important, Figure (3) shown UV-Visible absorption spectra of suspensions of CNPs prepared by laser ablation of a graphite target in deionize water at different laser pulse at range from (25 to 100pulse) and 80mJ, These spectra commonly exhibit many sharp bands and peaks [28 and 29] in the UV region. Show in Table (1), but each line has different absorbance value in each point of the wavelength. This indicates that the carbon nanoparticles produced have different size of distribution. Very small (or short) polyynes ( $n < 4$ ) are difficult to observe due to the weak intensity of the absorption peak at  $200.0\text{ nm}$ . The absorption behavior as a function of polyyne length is obtained by semi-empirical calculations of the electronic absorption of H-polyynes and dicyanopolyynes [30].

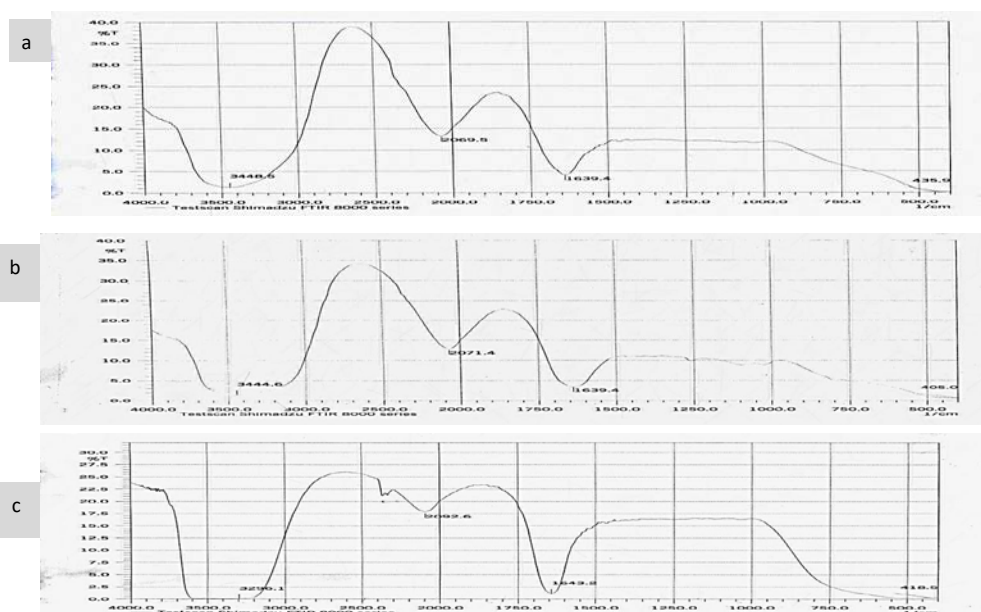
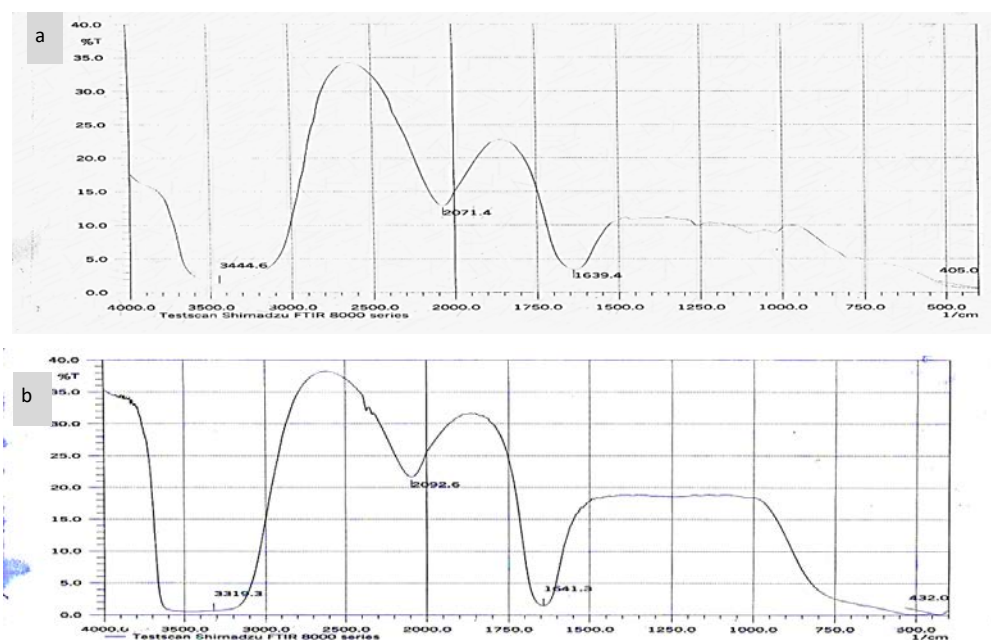


Figure (1) FTIR spectrum of CNPs suspensions at different laser energy

a) CNPs at 20 mJ b) CNPs at 80 mJ c) CNPs at 200 mJ.



**Figure (2) FTIR spectrum of CNPs suspensions at different laser pulse  
a) 25 pulse b) 100 pulse.**

The effects of number of laser pulse on all the peak intensities of polyynes at wavelengths are displayed in Figure (4), which reflects the concentration of ( $C_8H_2$ ) in suspension. The absorbance of polyynes increase with increased number of laser pulse but the absorbance was drastically reduced after 25 pulses and then increases. This peak shifts is due to the fact that strong scattering of light occurs when vapor nano-bubbles were formed at the time of leading edge of laser pulse and increase the concentration of CNPs [31]. It is observed that the optical absorbance of polyynes( $C_{n+1}H_2$ ) at wavelengths increases slightly with increasing the no. of laser pulse .this spectral change indicates that the abundance of CNPs is enhanced more under irradiation of laser.

Figure (5) demonstrates the influence of the number of laser pulse on nanoparticle concentration. It was observed that the increase number of laser pulse between 25 and 100 produce high particle concentration, due to increased radiation absorbed by the material. The increase of number laser pulses leads to increase the overlapping between these pulses which lead to increase in a local temperature. A high overlap between pulses, interaction of the laser beam with the previously ablated nanoparticles and the previously generated cavitation bubble could be the principal mechanism preventing the material from higher ablation. Laser ablation in liquids generates a cavitations' gas bubble that lasts around 300  $\mu s$  using Nd: YAG nanosecond laser irradiation of ( $36 J/cm^2$ )laser fluence [32 - 34]. This

cavitation bubble contains primary nanoparticles of extremely high local concentration that can scatter, reflect, or absorb the subsequent laser pulse.

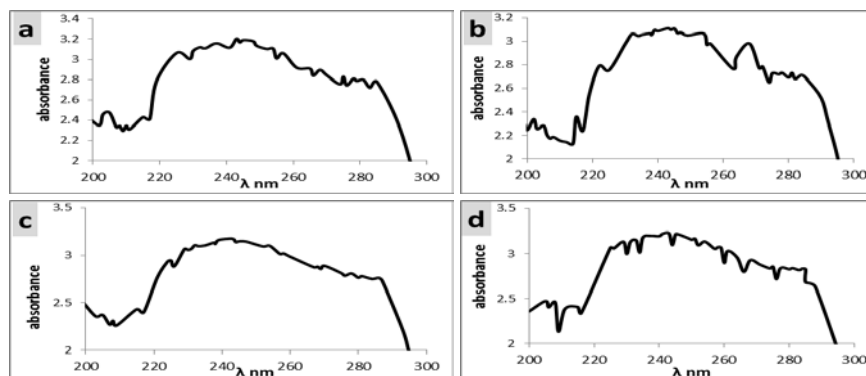


Figure (3) UV absorption spectrum of CNPs suspensions at different Laser pulse a) 25 pulse b) 50 Pulse c) 75 pulse d) 100 pulse.

Table (1) shows the polyynes at deionize water Suspension prepared at different laser pulse and different laser energy.

Peak at wavelength (nm)	polyynes
215.0	C4H2
208.0	C6H2
225.0	C7H2
205.0	C8H2
232.0	C9H2
237.6	C10H2
243.0	C11H2
275.0	C12H2
245.0	C13H2
278.0	C14H2
254.0	C15H2
268.0	C16H2
281.0	C18H2

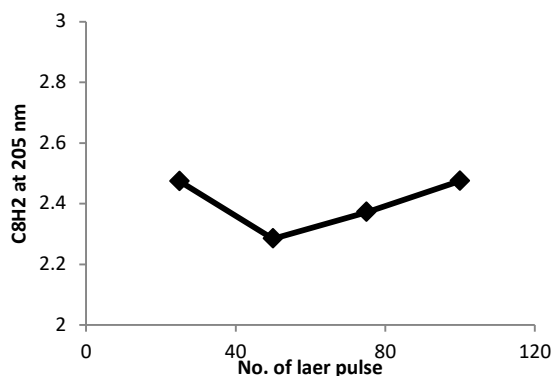
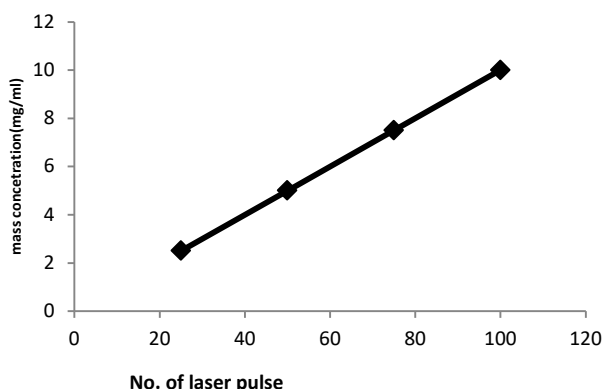


Figure (4) The effects of number of laser pulse on the peak

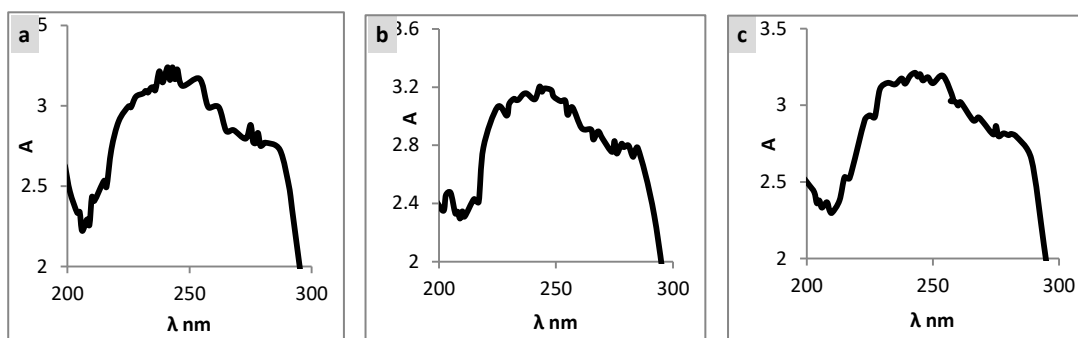
**Intensities of polyynes( $C_8H_2$ ) at wavelengths.**



**Figure (5) Carbon nanoparticles concentration as a function of the number of laser pulse.**

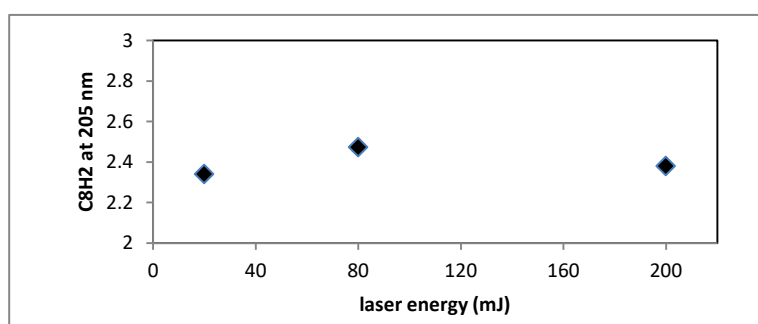
Figure (6) shows UV-Visible absorption spectra of suspensions of CNPs prepared at different laser energy range from (20, 80 and 200 mJ/pulse) and 25 laser pulse, these spectra commonly exhibit many sharp bands in the UV region. The observed bands coincide well with those of a series of hydrogen – capped polyynes. Notice that the suspensions contain polyynes with  $n= 4, 6, 7, 8, 9, 10, 11, 12, 13,14,15,16$  and 18 [26 and27], the all sample spectra show almost similar peaks, shows in Table (1). but each line has different absorbance value in each point of the wavelength. This indicates that the carbon nanoparticles produced have different size of distribution. The narrower the spectrum of peak indicates the more homogenous size distribution. If the height of the peak is calculated from the based line of spectrum, spectrum (80 and 200mJ) shows highest absorbance that indicates the highest number of CNPs in solution. Spectrum (20 mJ) show lower absorbance most probably that is because the spectrum has more number of big particles in the solution that reflect much more light than the spectrum (80,100,160and 200 mJ).

The increasing in the absorption intensity, accompanied by a slight change in band width and maximum wavelength, when increasing in the laser energy. This enhancement in intensity can be explained by the increase in the concentration of carbon nanoparticles formed in solution during the ablation process. The height and width of the absorption spectra were found also to be dependent upon the laser energy.



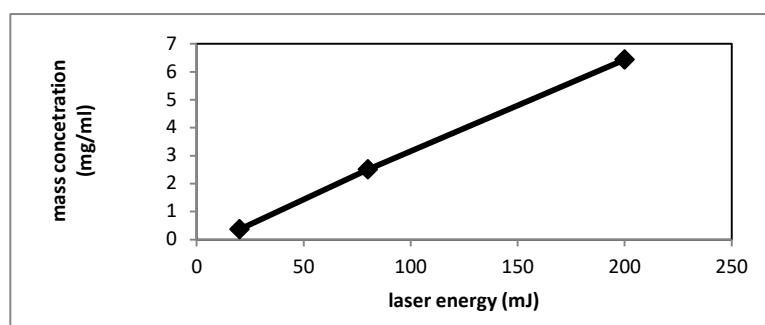
**Figure (6) UV-Visible absorption spectrum of CNPs suspensions at different laser energy a) 20 mJb) 80 mJc) 200 mJ.**

The effects of laser energy on all the peak intensities of polyynes at wavelengths are displayed in Figure (7), which reflects the concentration of ( $C_8H_2$ ) in suspension. The absorbance of polyynes increase with increase laser energy .illustrates the relative concentration of polyynes ( $\blacklozenge$ ), ( $C_{n+1}H_2$ ), which were estimated from the absorbance data at wavelengths, as a function of laser energy. At first glance, polyynes show turned in this absorption intensity as laser energy increases. Nerveless, the slight difference in the slope may possibly imply that polyynes are preferably produced at higher laser energies. This in line with the result of Figure (7), which shows the effect of laser energy on growth of large polyynes ( $n > 20$ ).



**Figure (7) the effects of laser energy on the peak intensities of Polyynes ( $C_8H_2$ ) at wavelengths.**

Figure (8) demonstrates the influence of the laser pulse energy on nanoparticle concentration, in the range (20-200 mJ) nanoparticle concentration increased almost linearly with pulse energy.

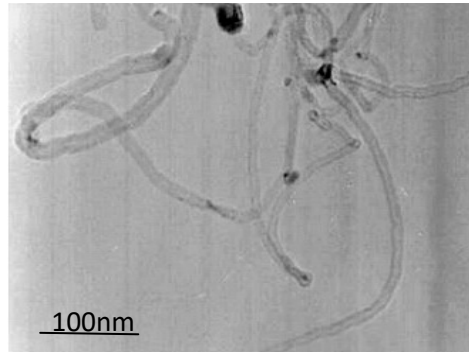


**Figure (8) Carbon nanoparticles concentration as a function of the applied laser pulse energy.**

• **Morphologic and structural characterization**

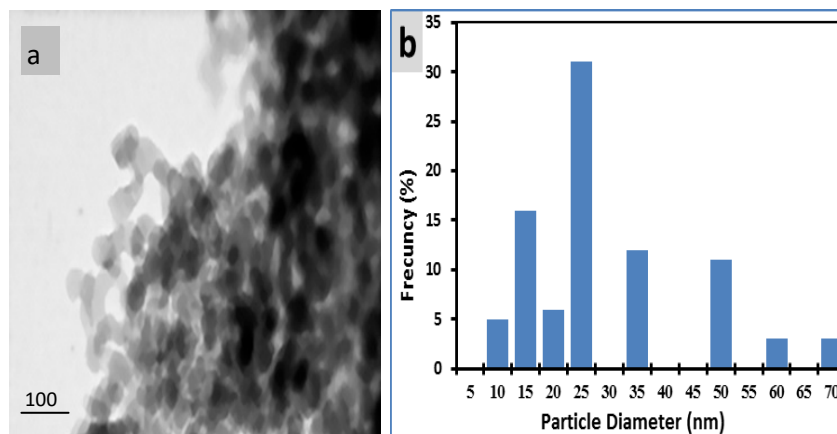


TEM images of the CNPs suspensions are illustrated in Figure (9), the Figure shows the effect of laser pulse energy on the formation of carbon nanotubes, the CNTs are uniform diameters, some of the CNTs aggregated to gather and some carbon nanoparticles with spherical geometry are contained in the middle of these CNTs. These nanotubes have diameter ranging (12.5 – 25 nm) with length ranging (500 – 708.3 nm).



**Figure (9) TEM image with manufacture (130X) of CNTs prepared by pulsed-laser ablation of graphite target at solid-liquid interface (deionized water) at laser irradiation with 25 pulses and energy of 80mJ/pulse.**

While Figure (10) shows TEM image of carbon nanoparticles. It was found that the carbon nanoparticles were in nearly spherical shape with an average diameter of around 34.5nm. In fact, the size distribution of carbon nanoparticles were in a range from approximately 9.4 nm to 70.5 nm and no individual particle higher than 70.5 nm in size was found.



**Figure (10) a) TEM image of carbon nanoparticles  
b) The size distribution of CNPs.**

## CONCLUSIONS

CNTs were synthesized at solid-liquid interface by pulsed laser ablation of graphite target in deionized water was developed. FTIR absorption peak referred to formation carbon nanoparticles suspended in this solvent. UV absorption peaks coincide with the electronic transitions corresponding to linear-chain polyynes the peak intensities increased when polyynes were produced different laser energy, the formation rates of polyynes increased with increased number of laser pulse. TEM result, shows CNPs with size ranging 9.4 – 70.5 nm, while CNTs were observed with diameter (12.5 -25 nm) and length (500-708.3 nm).

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