Laser Ablation Method for Synthesizing Different Types of Carbon Nanostructures in Presence of Graphite Target

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ABSTRACT— In this research, an attempt is made to approach a specific type of carbon, such as graphene or carbon nanotubes by using pulsed laser ablation technique in deionized water environment with changing the laser factors such as wavelength and fluence. Nd:YAG laser with two wavelengths of 1064 and 532 nm and three fluence of 0.8, 1 and 1.2 J/cm^2 were selected that number of pulses was 5000 with a frequency of 10 Hz to be irradiated on the graphite target at about 10 minutes. The medium was distilled water. Graphite was located in the 40 ml of distilled water.

The effects of wavelength and fluence of the laser have been experimentally investigated on types of carbon characteristics with different analysis such as Raman scattering spectrum, FE-SEM images, UV–Vis-NIR spectrum and X-ray diffraction (XRD). By using the mentioned analysis, the type of synthesized nano carbon is studied.

This study evaluates the effects of the pulse energy and laser wavelength on properties of synthesized carbon nanoparticle in laser ablation method in medium of distilled water.

KEYWORDS: pulsed laser ablation, carbon nanoparticles, graphite, graphene nanosheets

I. INTRODUCTION

There have been an increasingly focus on the Carbon Nano-particles (NPs) in the literature throughout last decade because of their substantial structural and electronic characteristics. They can be effectively used in electrochemical capacitors, single electron devices, magnetic storage materials, point source field emitters and nanotechnology. Additionally, lubrication products may be another potential field utilizing Nano-particles. Their applications include, but not limited to, textile production, biomedicine, electronics and etc. On the other hand, some conventional Carbon-based nanostructures can be named as graphene, carbonic quantum dots and Carbon nanotubes (CNTs).

Moreover, The NPs may be loaded on functional substrates such as Graphene to enhance their function [2,3].

Carbon nanotubes are composed of hollow cylinders of graphite plates. They are found in two basic forms of multi-wall nanotubes (MWNTs) and single-wall (SWNTs) ones.

It is obvious that SWNTs are formed from single nanotubes of graphene, while MWNTs consist of various concentric nanotubes of graphene which are fitted inside one another [5,6].

Due to the fact that graphene is the only two-dimensional structure known so far, it has various applications. Therefore, materials based on graphene are referred to as strategic Nano-materials because of their wide technological applications such as composites,

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sensors, super-capacitors, batteries and solar cells. Hence, high-quality fabrication of graphene Nano-sheets in industrial scales in a cost-effective and yet simple technique is considered to be ideal [7,8]. Additionally, graphene is considered as the desired substrate over all other species because of its properties such as optical transparency, mechanical strength, high surface area and good electrical and thermal conductivity [9].

There are more than eight methods to synthesize different types of carbon nano particles, which three common ones are: 1) Pulsed laser ablation (PLA), 2) Chemical Vapor Deposition (CVD) and finally 3) Arc-discharge. Reviewing previous literature shows that the chemical vapor deposition and arc discharge have been mostly adopted to compose MWCNTs. Different CVD methods like radiofrequency-enhanced, microwave-enhanced, and plasma-enhanced CVD have been introduced in similar researches [11].

Pulsed laser ablation (PLA) which is well-known for its great advantages over other methods, such as utilizing cheap and easy equipments for controlling the atmosphere of ablation, having simple experimental setup, and the fact that the produced material size can be controlled in this method by changing different characteristics like laser's wavelength, fluence, and pulse duration, the pH and temperature of solution and etc., have made it interestingly attractive to be considered as an efficient and fast producing method for nanoparticles [12].

In this technique, the target is immersed in a liquid and then the laser is applied to it. Next step is devoted to generating shock waves. As a result, the produced plasma expands and gradually cools followed by producing and expanding a cavitation bubble which is finally collapsed by the liquid and finally the NPs are released in the liquid. In addition, the target, whose composition defines the produced NPs' composition, used in PLA is drowned into a solution.

Using graphite in liquid environment and applying pulsed laser ablation (PLA) is a method for producing different type of carbon nanoparticles.

Several robust tools such as spot size, temperature, laser wavelength, intensity, pulse width and the liquid environment of ablation have been introduced to control the process of laser ablation and its final product.The product of ablation is affected by the liquid environment in two ways: 1) Controlling the plasma plume pressure on the surface of the target, which is directly proportional to the density of the ablation liquid. Therefore, the produced Nano-structures' morphology, size and structure could be controlled.2) The natural characteristics of the liquid environment like dispersion and polarity can strongly influence the products' aggregation. Accordingly, the laser ablation method is one of the most advantageous techniques of controlling the final product's structure, size and morphology due to its ability to adjust the laser wavelength and fluenc, the spot size, the pulse width and, last but not least, the ablation liquid environment, which is an important factor to determine the final product's characteristics, and its temperature [16].

In this work, carbon nanoparticles are synthesized by using laser ablation. Different parameters of laser ablation method such as fluences and wavelength was used as variables. The type of synthesized carbon nano particles is investigated using different analytical instruments.

II. EXPERIMENTAL SETUP

In this study, Pulse Laser Ablation (PLA) technique has been adopted to produce carbon nanostructures utilizing the fundamental and second harmonics of an Nd:YAG laser which has been operated at two wavelengths of 1064 and 532 nm. A 1-cm-thick high purity graphite target (99.9%) is located on the bottom of irradiation container which is, in turn, filled with 40 mL of distilled water with a height of 0.8 mm above the target. Various liquids have been reported to be adopted as the environment of the pulsed laser ablation method for producing different types of carbon nano particles [7]. Here distilled water was used as

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medium of PLA container which is shown in the schematic setup of the Fig. 1.

It worth mentioning that before the experiments start, all the equipment including the graphite plate were cleaned ultrasonically in deionized water and ethanol. Then, a second harmonic pulse of a 10-Hz Nd:YAG laser was used with two different wavelengths of 1064 and 532 nm to ablate the graphite target at about 10 minutes. Besides, producing carbon nanostructures in the liquid environment has consumed 5000 laser pulses. The fluences used for the laser pulses were 0.8, 1 and 1.2 J/cm^2 with a diameter of 6 mm. In addition, a 40-mm focal length lens helps the laser output to be focused on the surface of graphite target. Next, the cylindrical vessel gets full of deionized water which is considered to work as environment for ablation to get the carbon nanostructure ready in distilled water. Other important details about preparing the samples are presented in Table 1.

Table 1. Laser characteristics of samples produced by PLA

Sample number	1	2	3	4	5	6
Laser fluence (J/cm^2)	0.8	1	1.2	0.8	1	1.2
Laser wavelength (nm)	1064			532		

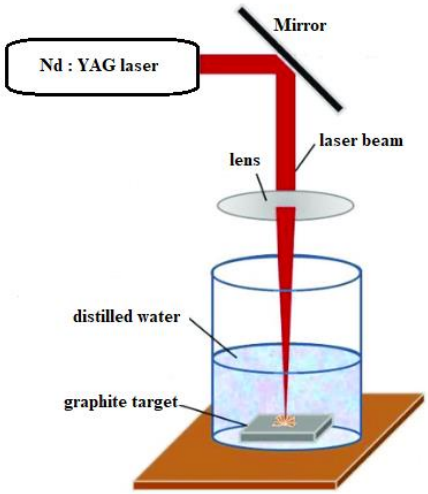

Fig. 1. Schematic pulse laser ablation setup.

To determine the characteristics of nanostructure products, different analytical techniques has been conducted. Also, a UV–Vis-NIR spectrophotometer is employed to examine the optical properties of the samples and for recording the spectra, samples are placed in 10-mm path length quartz cells with reference to the correspond solution. It's worth mentioning that the morphology of samples have been probed by Field Emission Scanning Electron Microscope (FESEM). Finally, the Raman spectroscopy was used to determine the quality, structure and the amount of carbon nanostructure in the suspension; and the crystalline structure of the carbon nanostructures was analyzed by X-ray diffraction (XRD) [7,10,11,13,14,29]. Raman spectra plays very important role for determination the type of produced nano structure.

III. Results and Discussion

In pulsed laser ablation (PLA) method several forms of carbon-based structures like carbon spheres, diamond-like carbons, fullerene molecules are expected to be produced besides graphene nanosheets after laser irradiation. In this experiment carbon nanostructures are fabricated by pulsed laser ablation of graphite target in deionized water [18].

A. *SEM Images*

To estimate the particle size and check the morphology of the resulted carbon nanoparticles, the Scanning electron microscopy (SEM) images are employed. Finally, SEM images confirm that increment of the laser pulse fluence will decrease the size of produced nanoparticles [19].

The surface morphology of the nanostructures are depicted in Figs. 2 and 3 utilizing emission scanning electron microscopy.

The results confirm the transparency of graphene nanosheets' morphology under all experimental conditions. As can be seen from the micrographs, for the samples at 1064 & 532 nm, the dominant morphology is that of spherical carbon nanoparticles and the graphene sheets. Moreover, it can be concluded from the results that wavelength and fluence of laser are able to remarkably change the carbon nanostructures average size and the number of carbon nanoparticles. For example, the carbon

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nanostructures average size has enlarged for the 532 nm samples [7].

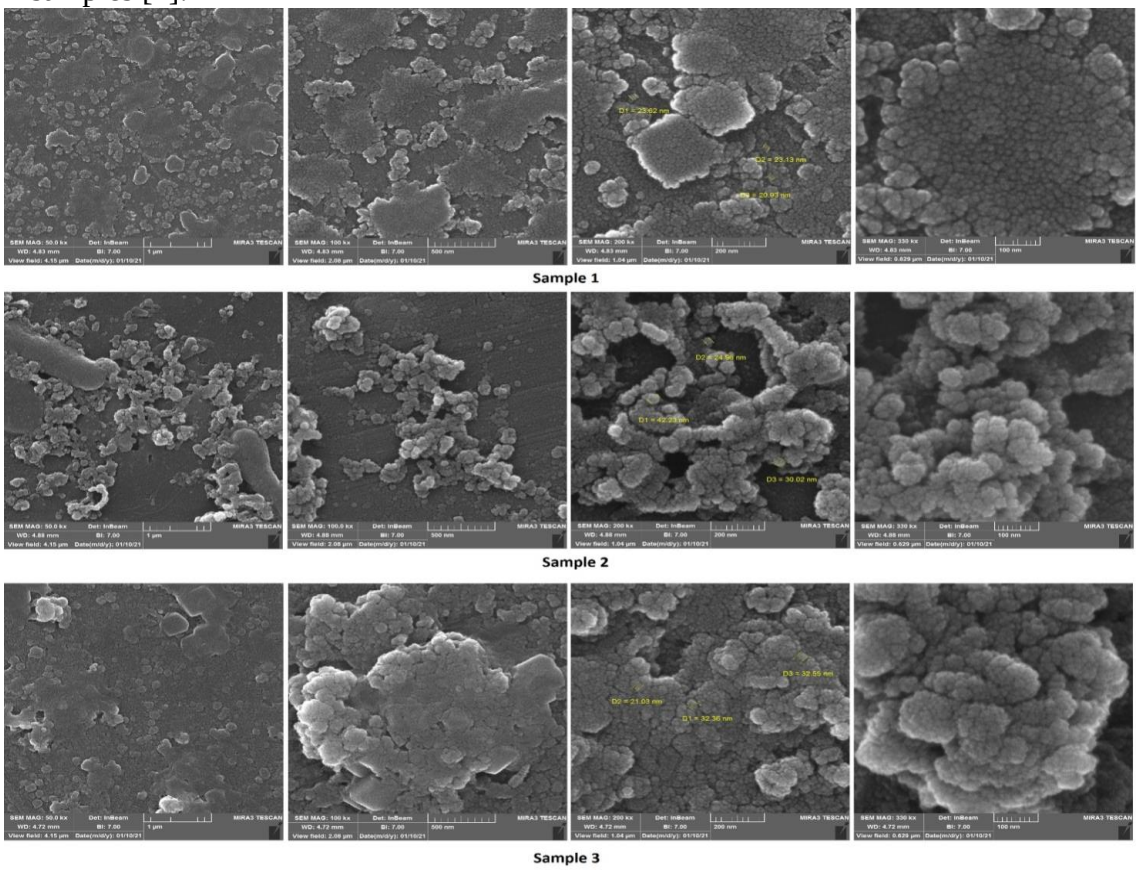

Fig. 2. SEM micrographs of samples 1, 2 and 3 in different magnifications

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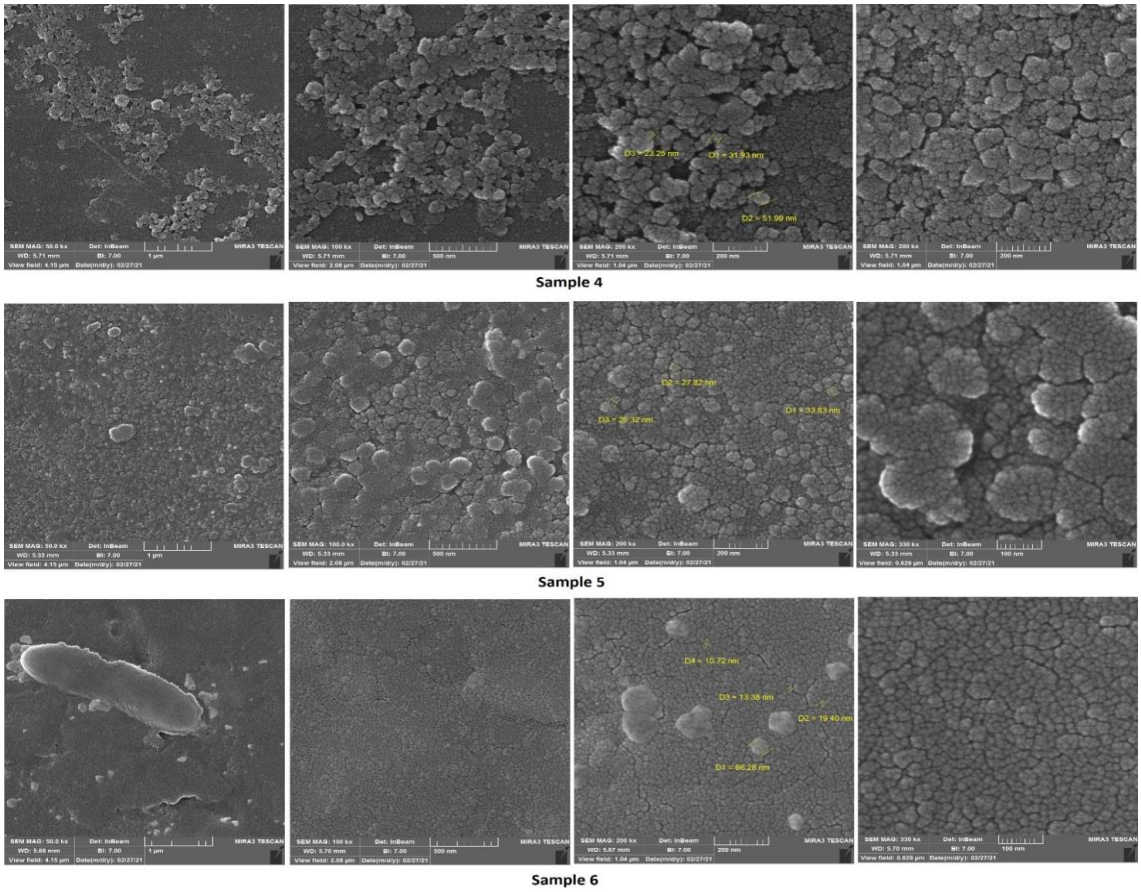

Fig. 3. SEMmicrographs of samples 4,5 and 6 in different magnifications

Moreover, there have been other carbon nanostructures produced in the ablation environment besides the graphene nanosheets. Average size of carbon nanoparticles for the samples at 1064 nm was around 28 nm and for the samples at 532 nm was around 31 nm.

B. *Raman Spectrum*

Raman Spectroscopy has been widely used, as a powerful and non-destructive diagnostic tool, to identify the quality and the structure of the materials based on carbon. Fig.4 shows the Raman spectra of the samples in the range of 600–3400 cm^{-1}. It's worth mentioning that all the experiments have been performed on dried drops of the suspensions on glass substrates. Table 2 presents the detailed information of the peaks appeared in Fig. 4.

Table 2 the position of the samples' Raman peak

Sample	D peak position (cm^{-1})	G peak position (cm^{-1})	2D peak position (cm^{-1})	I_D/I_G
Sample 1				
Sample 2	1367	1593	2865	0.85
Sample 3	1383	1603	2945	0.86
Sample 4	1094	1372		0.79
Sample 5	1082	1603		0.67
Sample 6	1082			

To explain more, it should be considered that the obtained G band in Raman spectra represents the E_{2g} vibrational modes of the aromatic domains while the D band indicates the breathing modes of the graphitic domains. On the same side, the G band is affiliated to the doubly degenerated (TO and LO) phonon mode (E_{2g} symmetry) at the center of the Brillouin zone and is created Raman scattering's first order process. While, the D band can be attributed to the TO phonons which are located about the Brillouin zone's K point that is issued from a defect/disorder in the SP2 carbon network. However, the 2D band is defined as the second order of the D band resulted from a two-phonon lattice vibrational process which need not to be activated through proximity to a defect unlike the D one. As it is seen in Table 2 and Fig 4, for sample 2 and 3, D band appeared in 1367 and 1383 and G band appeared in 1593

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and 1603. Also, 2D band is obvious in these two samples.

In similar research (F. Kazeimzadeh *et al.*) [6] D band of Raman scattering spectra was around between 1250 and 1350 cm^{-1}, G band was between 1550 and 1600 cm^{-1} and 2D band was around 2500 and 2700 cm^{-1} which are closer to our results in samples 2 and 3 and confirm them.

In other research (E. Solati *et al.*) [7] results of Raman analysis were in the range of 1100–3000 cm^{-1} that like our work graphene was achieved.

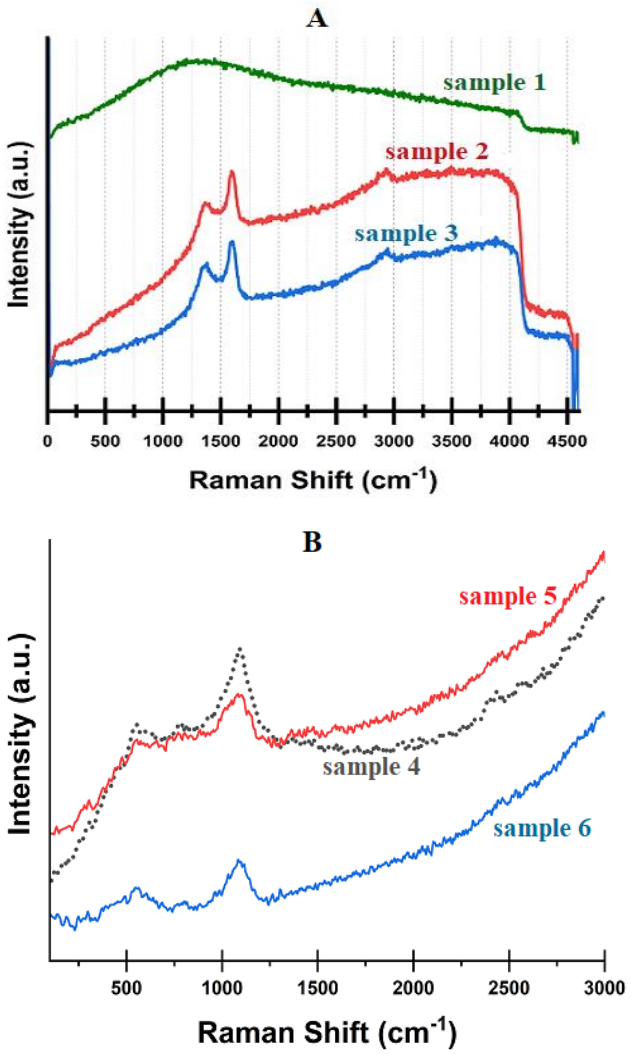

Fig. 4. Raman scattering spectra of the samples **A)** 1 to 3 prepared using wavelengths of 1064 nm **B)** 4 to 6 prepared using wavelengths of 532 nm

In addition, being oppositely proportional to the graphitic material's SP^2 clusters, the relation of D and G bands (I_D/I_G) has widely been utilized to define the level of disorders. Furthermore, the following equation is used to calculate the graphene nanosheets' SP^2 clusters' in-plane crystallite:

$$L_a\,(nm) = \left(2.24\times10^{-10}\right)\frac{\lambda^4}{\left(I_D/I_G\right)} \qquad (1)$$

where I_D/I_G is the intensity ratio of the D and G bands, λ represents the excitation wavelength of Raman measurement and *La* indicates the average crystallite size of the SP2 clusters.

Eventually, it can be realized from the Raman spectra that the graphene nanosheets of the samples prepared by a laser fluence at 1064 nm have the best quality among all samples.

In similar research (by F. Kazeimzadeh *et al.*) D-band and G-band were very close to our results that mean in sample 2 and 3 we reach to better quality graphene [6].

C. *XRD Analysis*

In order to investigate the morphology and the crystal structures like carbon nanoparticles and graphene nanosheets, the XRD analysis is employed. [7].

Therefore, silicon substrate is prepared to hold the suspension of the dried-at-room-temperature samples to form dried films. Finally, the XRD measurement and results can be seen in fig. 5.[14].

As the Fig. 5 indicates, the peaks of the samples 1, 2, 3 (produced at 1064 nm) can be addressed at about 2θ=26.5°, 2θ=26.5° and 2θ=29.5°, respectively.

The XRD pattern of samples 4, 5, 6 (produced at 532 nm) shows a peak at about 2θ =27°.

There are also two tiny peaks, having too weak intensity, at 2θ=42.5° and 2θ=54.5° in the XRD pattern of samples 1,2,3 which are formed because of the lattice structure of graphite target.

In similar researches (E. Vaghri *et al.*) [11] and (R. Hameed *et al.*) [12] and (N. Tabatabaie *et al.*) [20] reflection peaks were at 2θ=32.1°,

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2θ=26.6°, and 2θ=26.4°, respectively, which related to graphene structure that is close to our result.

Being a two-dimensional material helps the graphene to result in no significant X-ray diffraction for pure graphene [7].

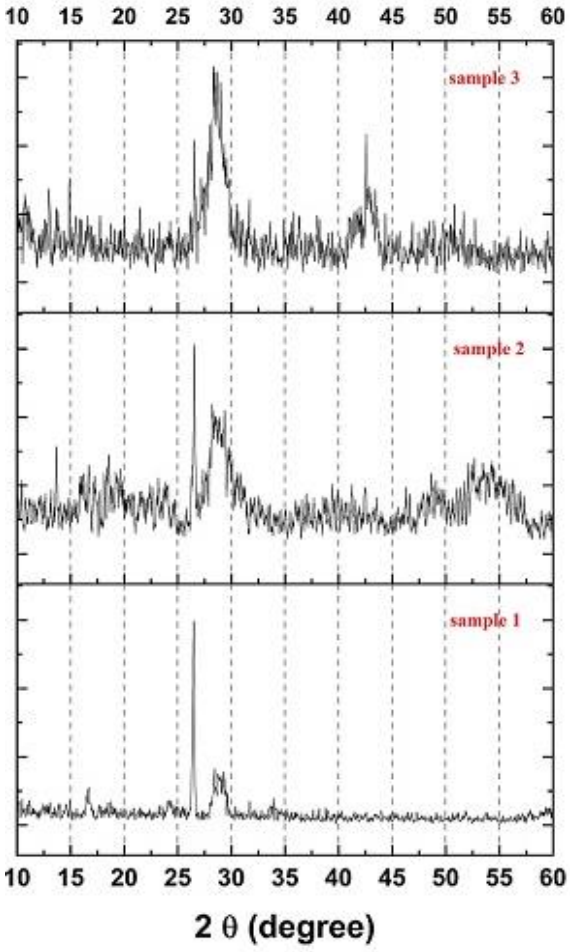

Fig. 5. X-ray diffraction of carbon nanoparticles and graphite target for samples **A)** 1 to 3 prepared using wavelengths of 1064 nm **B)** 4 to 6 prepared using wavelengths of 532 nm.

Resultingly, the peaks, whose intensity is not that strong, appearing in the XRD of the samples of the current study are attributed to the graphene nanosheets as well as carbon nanostructures which exist in the suspensions.

Eventually, as a result of their low-intensity peak, the produced nanostructures at 1064 nm are so close to graphene structures. On the other hand, it is deducted that the decrement of wavelength of the laser brings about ablating atomic carbons from graphite target.

Therefore, the carbon nanoparticles and other carbonic structures should have been produced in the suspensions [8,10].

As a result, no XRD peaks are expected to be seen for monolayer graphene; while, the few-layer graphene results in very weak peaks [14].

D. *UV–vis Analysis*

Considering distilled water absorption as the base line, the measurement of PLA-prepared carbon nanostructures in liquid water as medium is performed by a UV–vis spectrometer at the wavelength range of 200–1100 nm. The UV–Vis–NIR absorption spectra of carbon nanostructures for samples 1-6 are shown in Fig. 6. The absorption peaks of carbon can normally be seen in the range of 180-280 nm [13].

As can be seen in the UV–Vis–NIR spectra of samples prepared in deionized water at 1064 nm there is an absorption peak at 219 nm. Moreover, the fact that the absorption peak intensity is low for these samples may be because of the fewer particle nanostructure existence in these suspensions. On the other hand, the sharp peak of these samples proves the distribution of the narrow-sized carbon nanoparticles in them [7].

Additionally, an absorption peak appears in around 240 nm in the samples prepared using laser pulses of 532 nm which can be because of the π–π* transition of C=C band. Conversely, there is no sharp absorption peak in the spectra

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of samples 3 to 6 that might be because of the appearance of some surface resonance (SPR) peaks at different energies which, in turn, proves the existence of different-size carbon nanoparticles overlapping each other [8,10].

It can be seen that the unmatched laser fluences make almost the same intensity of the absorption peaks.

Reviewing the results of the experiment reveals that the graphene nanosheets as well as the carbon nanoparticles which are parts of the suspensions cause higher peak in the 1064 nm samples compared to the 532 nm ones.

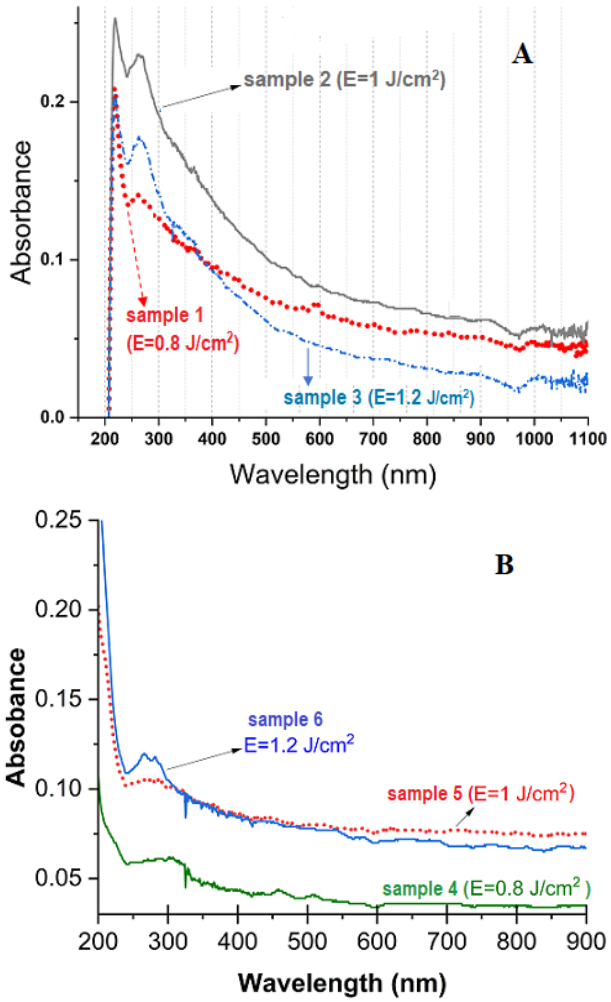

Fig.6 Absorption spectra of samples **A)**1-3 prepared using wavelengths of 1064 nm **B)** 4-6 prepared using wavelengths of 532 nm

IV. Conclusion

Pulsed laser ablation in liquids has been widely used as a method to produce ultrapure nanomaterials [13]. In this article, utilizing an Nd:YAG laser with two wavelengths of 1064 and 532 nm and three fluence of 0.8, 1 and 1.2 J/cm^2 on the graphite target in water and focusing its fundamental and second harmonic, some carbon nanostructures like carbon nanoparticles and graphene nanosheets have been fabricated. The laser fluence and wavelength has been set to change the distribution of the carbon nanoparticles and graphene. The produced carbon nanostructures has been in the form of transparent sheets which is like that the graphene morphology.

On the other hand, several graphene transparent sheets as well as carbon nanoparticles has been detected in the morphology of samples produced in this study. Additionally, the lower wavelength of the laser, the higher absorption peak intensity. Moreover, the results of the pulsed laser ablation technique indicate that the laser wavelength determines the quantity of the produced nanostructure which may be a result of the fact that the large wavelengths are strongly absorbed into the ablation plasma.
The Raman spectra reveals that the best quality of graphene nanosheets among all samples is achieved in the laser fluence of 1 J/cm^2 at 1064 nm.
Finally, analysis results show that the produced nanostructures are very close to structure of graphene.

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