

Impact of Laser Fluence on the Formation of T-Nb₂O₅ Nanostructure: A Study in a Liquid Environment

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ABSTRACT

In this work, different laser fluencies ranges (8.1–17.8 J/cm²) and Nd: YAG of 1064 nm wavelength were studied and analyzed. Energy band gaps acquired ranged from 4.2 eV to 3.9 eV. X-ray diffraction (XRD) results revealed the formation of an orthorhombic (T-Nb₂O₅) niobium pentoxide nanostructure. The grain size of the Nb₂O₅ nanoparticles ranged from approximately 58.2 nm to 244.6 nm. TEM images showed spherical particles whose density increased with increasing laser fluence. Raman and Fourier-transform infrared (FTIR) spectra exhibited peaks indicating the formation of T phase-Nb₂O₅ nanomaterial.

Keywords: Liquid-pulse laser ablation, Optical properties, Raman spectra, FTIR

1. INTRODUCTION

Niobium pentoxide (Nb₂O₅) or niobium [1, 2] is a substantial n-type transition metal oxide semiconductor with unique properties. Thus, it has been applied to electrochromic devices, and sensors and develops highly efficient solar cells, [3-5]. This material has drawn great attention as a combination of different compounds, such as lithium niobite and barium, used in optical modulators and waveguides [6-8]. It is the most thermodynamically stable metal oxide featuring chemical inertness and a low cytotoxicity [9, 10]. The charge state of Nb₂O₅ is (5+) and has a lower electrical conductivity compared to other niobium oxides [11]. Besides, Nb₂O₅ absorbs Ultra Violet light with a 385nm wavelength or less and visible light to achieve high photocatalytic efficiency [12, 13].

Niobia can either happen in an amorphous state or one of various crystalline polymorphs. In general, Nb₂O₅ can come in a powder-like structure of white color or a single crystal transparent form. The physical properties of the Nb₂O₅ can be ultimately changed according to its polymorph and the composition parameters and synthesizing method [14-16].

Nb₂O₅ has been prepared using various methods, such as metal oxidation in air, hydrolyzation of alkali niobates, niobium alkoxides [17], chemical reaction of hydrofluoric acid and ammonia, pulsed laser deposition [18, 19], reactive

radio frequency magnetron sputtering [20], atomic layer deposition [21, 22], sol-gel [23], and other methods.

Joya et al. (2017)[24] fabricated an Nb₂O₅ orthorhombic structure by employing the sol-gel method, and SEM revealed unique structures (skeleton Nb₂O₅). Later, Wang et al. [25] prepared Nb₂O₅ nanowires using plasma-enhanced chemical vapor deposition. XRD data showed an orthorhombic T- Nb₂O₅ structure, Dai et al.(2020)[26] prepared Nb₂O₅ nanofibers used the template, XRD revealed an orthorhombic Nb₂O₅ with different range band gaps. A monoclinic Nb₂O₅ was prepared by Fakhri et al. (2021)[27] they employed different conditions using a pulsed laser deposition system.

Material properties can be varied by tuning preparation conditions. The effect of laser energy on the properties of Nb₂O₅ in liquid is not extensively studied yet. This work aims to characterize the unique properties of Nb₂O₅ nanoparticles obtained by laser ablation in liquid by using a metal plate target.

2. MATERIAL AND METHOD

Nb₂O₅ nanoparticles were prepared under certain conditions and parameters. A high purity (about 99.999) Niobium plate manufactured by Sigma Aldrich was used with de-ionized water and Nd: YAG laser to achieve

nanoparticle preparation. Sample preparation process was maintained by submerging the niobium target into the de-ionized water and ablated by laser of parameters: 1 Hz, 150 pulse, and fluence ranges from 8.1 J/cm² to 17.8 J/cm². A Shimadzu 1800 spectrophotometer double-beam UV-VIS device was utilized to study the Nb₂O₅ optical properties.

FTIR spectroscopy was studied using BRUKER-7613 to achieve the chemical bonds of prepared materials. A SUNSHINE-V2-86 device was used to obtain the Raman spectrum. An X-ray diffraction system from (Shimadzu) with 0.15406 nm wavelength was employed to determine the structural characteristics of Nb₂O₅. The sample was scanned from 2θ of 15° to 50°. Niobia morphology, such as size, distribution, and particle shape, was investigated using a Titan 80-300 HRTEM device. For chemical composition, the stoichiometry and element percentages in the sample were investigated using inspect S50 (FEI Company/Netherlands).

3. RESULTS AND DISCUSSION:

The X-ray diffraction pattern of the Nb₂O₅ samples is shown in Figure (1). The X-ray diffraction revealed several peaks centered at different angles (2θ): 16.8°, 22.8°, 35.8°, 42.8°, 47.4°, and 48.4° which they are correspond to the planes (1 3 0), (0 0 1), (1 0 1), (1 1 3 0), (0 0 2) and (1 1 0). It was shown that the peaks belong to the orthorhombic structure of the T-Nb₂O₅ as consistence with the (00-030-0873) card. The diffraction peaks at 2θ = 30.6° belong to the pure Nb as tabulated in (00-003-0905) JCPDS card and shown in other results [28-31].

The crystallite structure of the prepared Nb₂O₅ showed a significant enhancement in terms of laser fluency to reach

12.09 J/cm² with an evident reduction in the niobium diffraction peak of that fluency. As laser fluency increased to 17.82 J/cm², the diffraction peaks of the prepared sample became higher and more obvious with prominent development in the pure metal formation. Scherrer's formula was utilized to estimate the size of the particle of the prepared samples [32-34]

$$D = k\lambda / (\beta \cos \theta) \quad (1)$$

Where D is the particle size in nanometers, K is the shape factor, λ is 0.15406 nm (X-ray wavelength), β is spectrum broadening at the Full-Width Half-Maximum (FWHM), θ is Bragg angle, and finally, δ is dislocation density which can be calculated using the equation 2 [35-37]:

$$\delta = 1 / D_{XRD}^2 \quad (2)$$

It is worth mentioning that the shape (K) factor is about 0.9 and this value changes concerning the crystallite shape. The Micro-strains were obtained using the following formula [38-40]:

$$\eta = \beta / 4 \tan \theta \quad (3)$$

Table (1) presents the grain size of the Nb₂O₅ nanoparticles at different laser fluencies. It can be recognized that the grain size is directly related to laser fluence due to the increase in the amount of the ablated material. This phenomenon led to particle agglomeration, which agrees with the TEM and UV-Vis results.

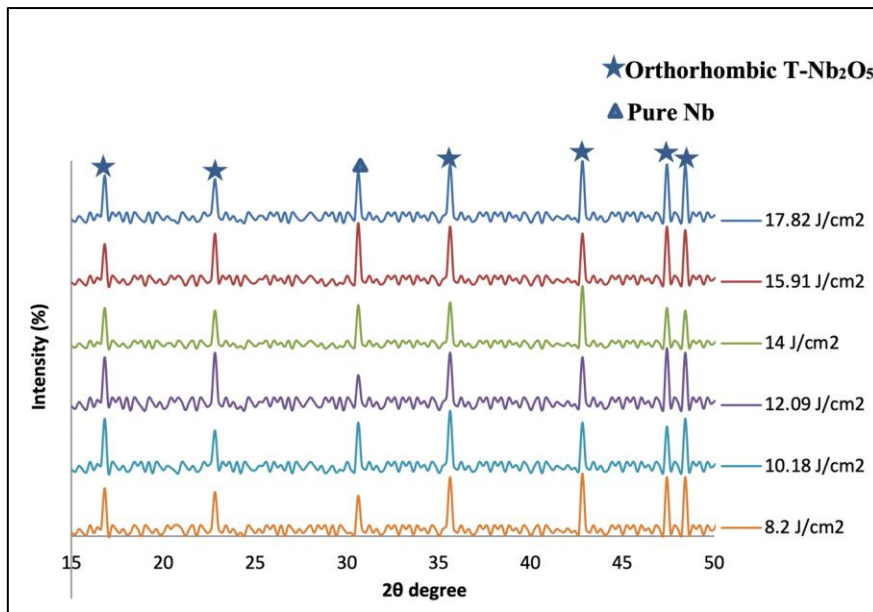


Figure 1. X-ray diffraction patterns of Nb₂O₅ samples using 150 pulses at different ranges of Nd-Yag laser fluencies.

Table 1 Grain size for different laser fluencies

2 θ .	FWHM.	Miller indices	Grain size(nm)	Dislocation density(δ)	Microstrains
16.8°	0.141	1 3 0	58.245	0.294	5.2
22.8°	0.131	0 0 1	62.121	0.259	6.6
35.8°	0.069	1 0 1	114.559	0.076	5.5
42.8°	0.054	1 13 0	143.144	0.048	5.2
47.4°	0.045	0 0 2	169.194	0.034	4.8
48.4°	0.031	0 0 1	244.664	0.016	3.4

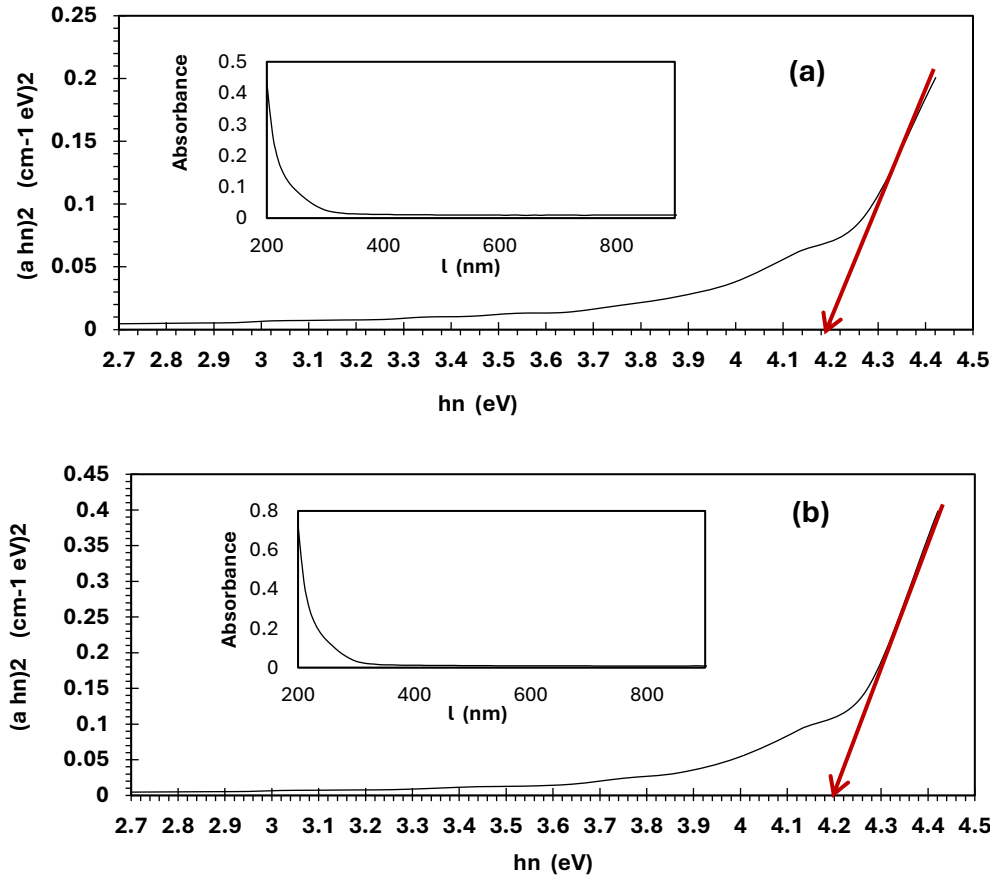
The energy bandgap and the absorption spectrum are presented in Figure (2). They are graphed as a function of the wavelength of Nb₂O₅ Nanomaterials. A clear red shift in the absorbance spectrum with laser fluence could be recognized. The obtained results indicate that the concentration of the ablated material is ultimately developed. Besides, the energy gap and laser fluence values were found to be inversely related.

This can be back to the fact that the size of the prepared nanoparticles is large enough compared to the exciton Bohr radius which results in a reduction in energy bandgap value due to the weakness confinement as shown in Table (2) [41-44]. These results strongly agree with the grain size obtained using XRD.

The estimation of the energy bandgap was conducted using Tauc's plot. It indicates that the incident photon energy to be on the horizontal axes (abscissa) and $(\alpha h\nu)^2$ is inordinate. The value of the energy bandgap was determined by the absorption edge for the direct interband transition and the obtained from equation 4 given below [45-47]:

$$\alpha h\nu = c(h\nu - E_g)^{1/2} \quad (4)$$

Equation (4) parameters are defined as: α is the absorption coefficient, h is Plank's constant, ν is the incident photon frequency, and finally c is the direct transition constant.



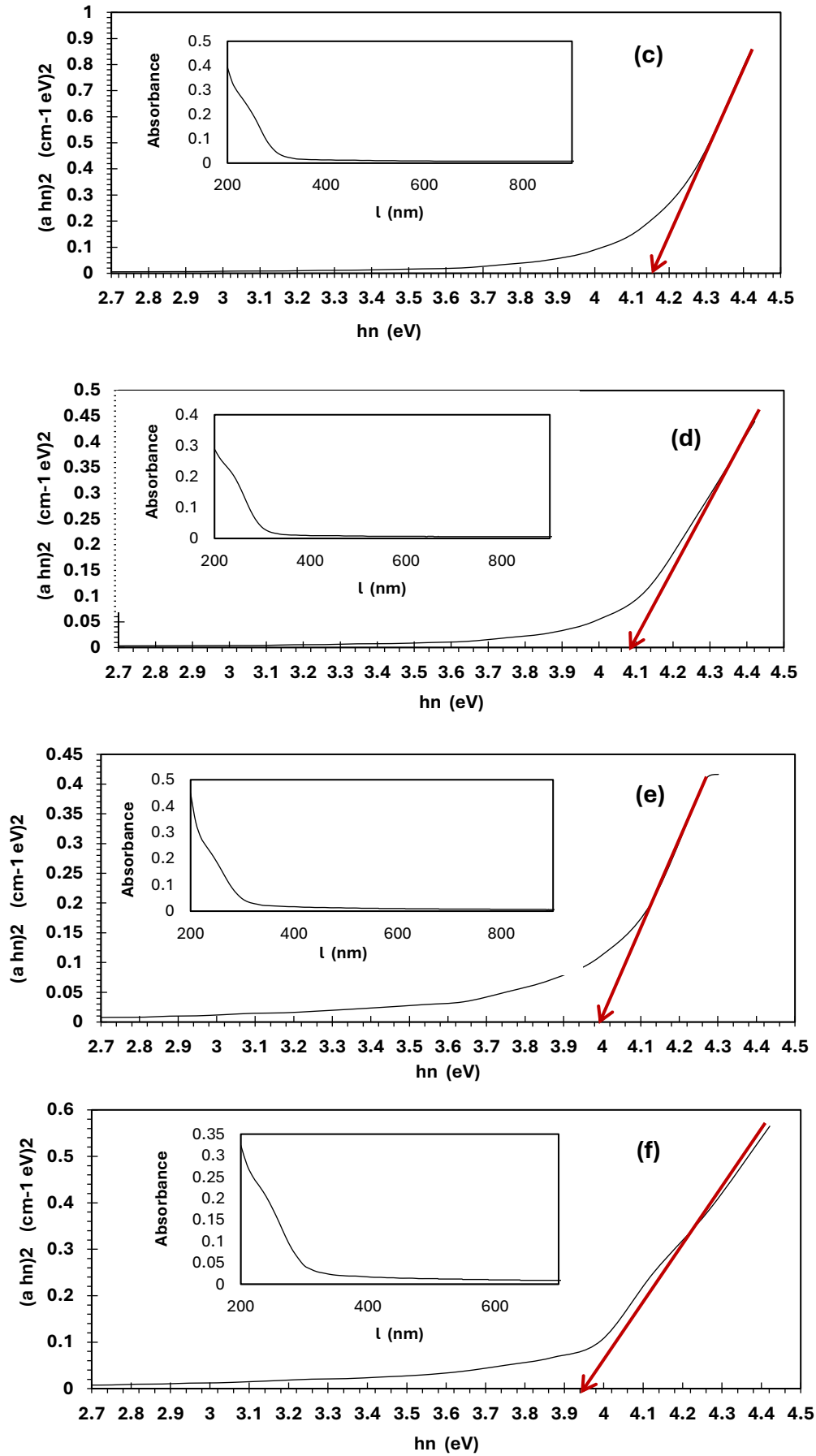


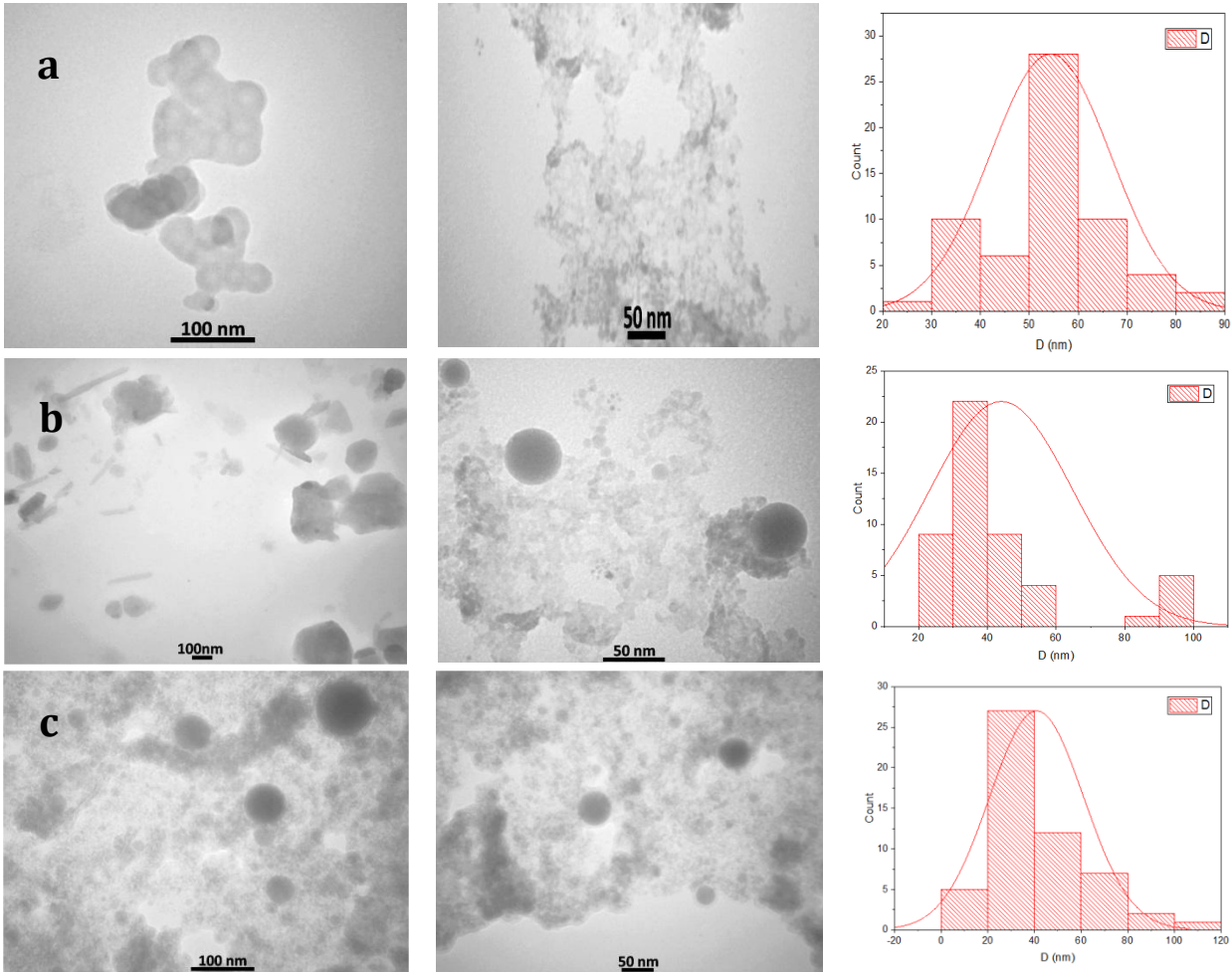
Figure 2. Absorption and bandgap energy plots of the prepared at various (a) 8.2 J/cm², (b) 10.18 J/cm², (c) 12.09 J/cm², (d) 14 J/cm², (e) 15.91 J/cm², and (f) 17.82 J/cm².

Table 2 Energy bandgap obtained at different laser fluencies

Laser Fluence (J/cm ²)	Energy Bandgap (eV)
8.2	4.2
10.18	4.19
12.09	4.15
14	4.08
15.91	4
17.82	3.93

The spherical nanoparticles can be shown clearly in Figure (3) using Transmission Electron Microscopy images for samples prepared at different fluencies. Low concentrations of small, spherical-shaped particles are recognized at low laser fluence. Whereas, increasing the value of the laser fluence shows an increasing the particle size due to a high aggregation rate of high-concentration small fragments. Similar results are found in the literature referenced at [48-50].

Image J. software was used to perform TEM imaging histogram. Table (3) lists and illustrates the histogram curves of the average particle size. Its values were found to decrease with increasing laser fluence due to the large absorbed amount of energy. This phenomenon led to a temperature rise, after which the particles started [49].



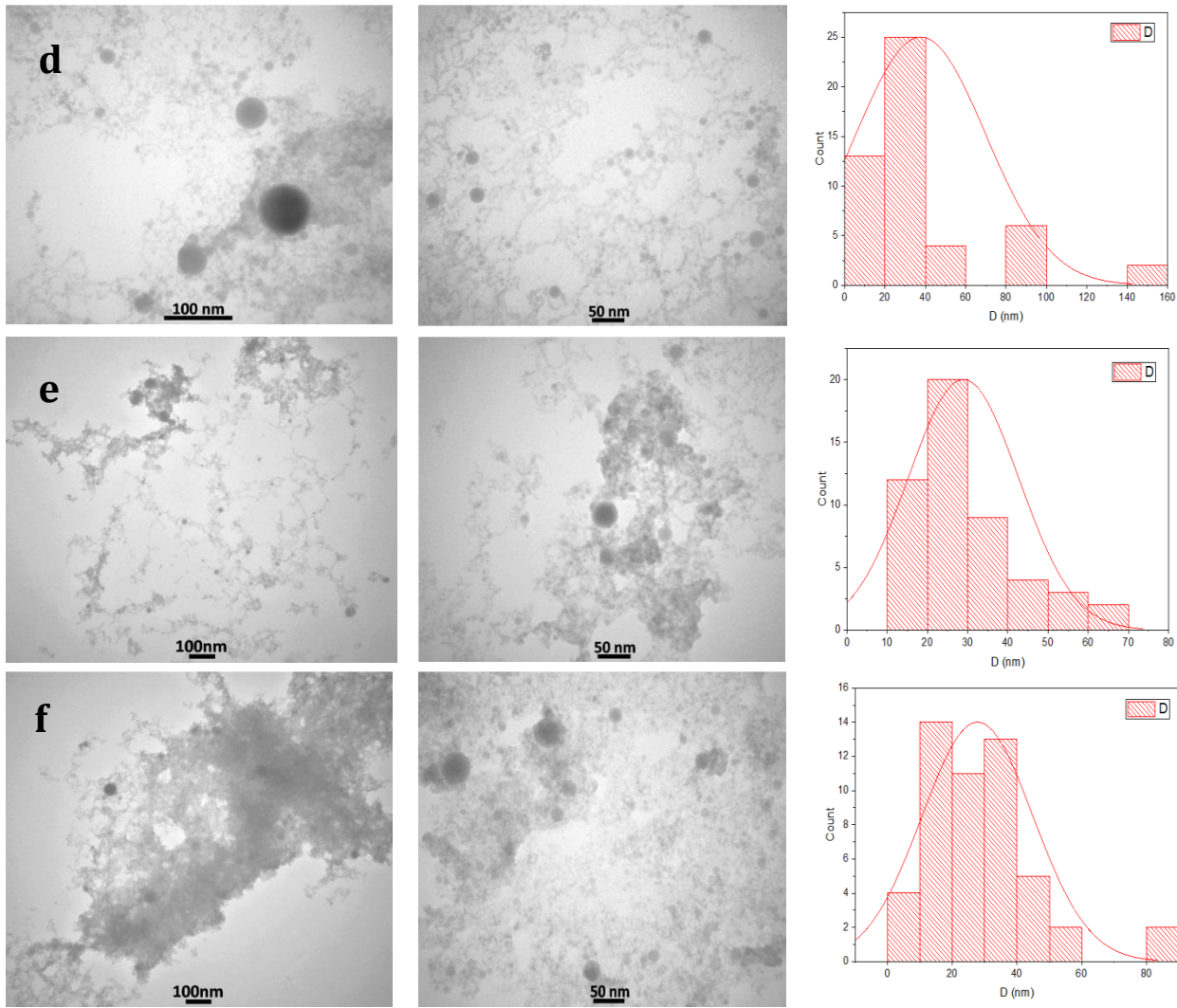


Figure 3. TEM images of the prepared samples at (a)8.2 J/cm² (b)10.18 J/cm² (c)12.09 J/cm² (d) 14 J/cm² (e)15.91 J/cm² (f)17.82 J/cm².

Table 3 Lists the average particle size in nanometers results are different laser fluences

Laser Fluence (J/cm ²)	Average particle size (nm)
8.2	55
10.18	45
12.09	42
14	40
15.91	30
17.82	28

According to previous results, the optimum results were achieved at a laser fluence value of 12.09 J/cm². Further measurements were conducted to understand the chemical behaviors of the prepared sample. Figure (4) shows the EDX image shows the optimal structural properties obtained by

sample preparation at 12.09 J/cm² laser fluence. The appearance of Niobium, Oxygen, and both Carbon and Silicon are confirmed by the results obtained. The stoichiometry of Nb₂O₅ was approximately equal to 66.02%.

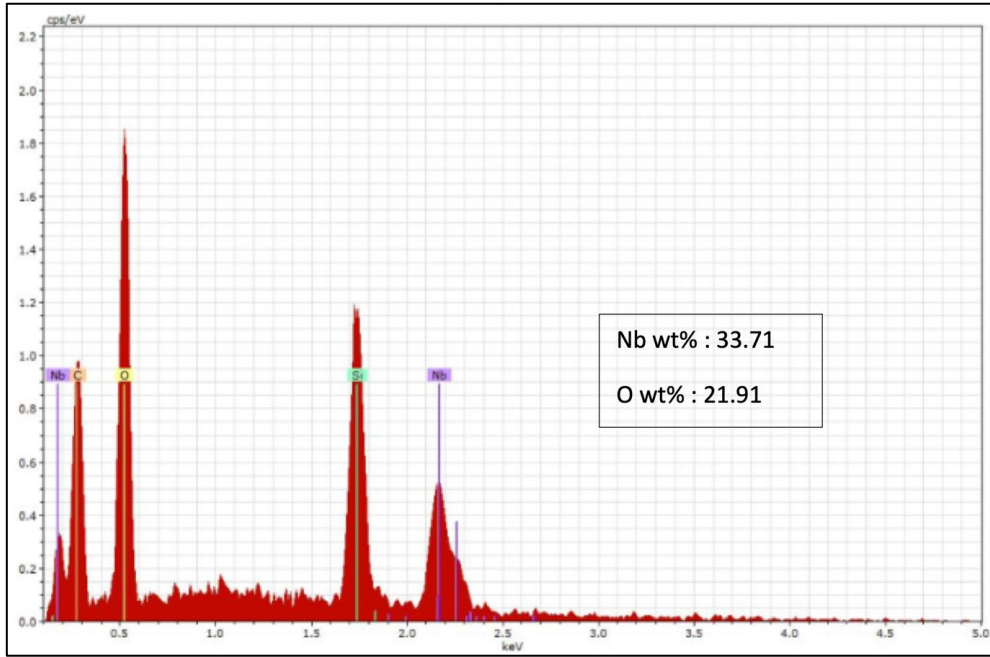


Figure 4. The EDX spectra of the prepared Nb₂O₅ at 12.09 J/cm².

Surface chemical bonds of Nb₂O₅ were investigated using FTIR spectroscopy. Figure (5) displays the FTIR transmission spectra of the Nb₂O₅ nanoparticles synthesized using 12.09 J/cm² laser fluence and 150 laser pulse. This fluence showed the best chemical bonds of Nb₂O₅. The FTIR spectra confirmed the existence of Nb₂O₅ and water.

Figure (5) shows the FTIR image of the prepared sample at 12.09 J/cm². The FTIR image depicts four transmission percentage peaks belonging to different wavenumbers.

The first peak belongs to the stretching vibration mode of the “Nb-O-Nb” which occurred at 713.6 cm⁻¹. This peak commonly indicates the formation of the Nb₂O₅ and its T-Phase as stated in works references in [51-54]. The second peak is assigned to the bending vibration of the H₂O and the weak asymmetric band of the OH group which occurred at 1631.7 cm⁻¹ as shown in other work [55, 56]. Finally, the peaks 2113.9 cm⁻¹ and 3255.8 cm⁻¹ are related to the pure niobium and the OH stretching vibration of Nb-OH, respectively [57-59].

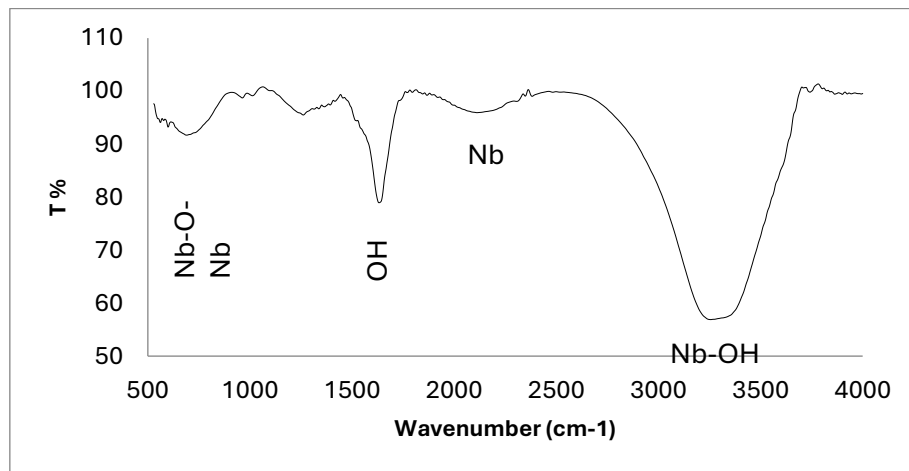


Figure 5. The FTIR of the prepared Nb₂O₅ at 12.09 J/cm².

According to the chemical bonds and the IR resonance peaks of the colloidal Nb₂O₅ are listed in Table (4). The locations of

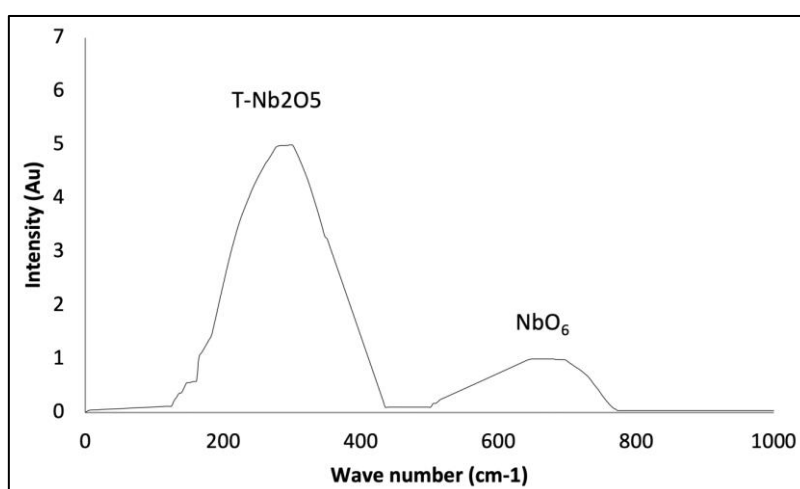
the observed peaks are completely agreed with works in literature listed in the same table.

Table 4 Represents the obtained chemical bonds and their position at 12.09 J/cm² laser fluence

Obtained bond value (cm ⁻¹)	Assignment	Vibration mode	Reference
713.6	Nb-O-Nb	Stretching	[51-54], [59, 60]
1631.7	O-H	Bending	[52, 55, and 61-63]
2113.98	Pure Nb		[57]
3255.84	Nb-OH	Stretching	[55, 62]

Raman spectroscopy is a very important non-destructive technique to characterize the chemical composition of Nb₂O₅. Figure (6) illustrates the Raman spectrum of Nb₂O₅ samples prepared using 12.09 J/cm² laser fluence and 150 pulse. This fluence provided the best Raman peaks, which represent the best chemical composition of Nb₂O₅. Raman

spectrum of Nb₂O₅ Nanoparticles shows a peak 298.45 cm⁻¹, which indicates orthorhombic T-Nb₂O₅. The obtained results are coincide with those obtained from FTIR and XRD tests. The small peak occurred at 649.15 cm⁻¹ corresponds to the NbO₆ stretching modes of the typical polyhedra of the orthorhombic Nb₂O₅ crystalline structure [61-63]

**Figure 6.** Raman spectra of the prepared Nb₂O₅ sample using 1064 nm Nd:YAG at 12.09 J/cm² with 150 pulse.

Details regarding the chemical bonds and their corresponding locations of the Raman peaks are listed in Table (5). The values of the observed peaks and their

corresponding positions are highly agreed with FTIR and the results found in the literature which are mentioned in the table.

Table 5 Raman peaks for Nb₂O₅ nanoparticles corresponding chemical bonds

Peaks cm ⁻¹	Peak Assigned to	Reference
298.45	T-Nb ₂ O ₅ (orthorhombic structure)	[62]
649.15	Stretching modes of NbO ₆ polyhedral typical of orthorhombic structure	

4. CONCLUSIONS

In conclusion, spherical Nb₂O₅ nanoparticles were successfully synthesized using PLA. T-Nb₂O₅ structure was obtained, as confirmed using different measurements. Laser fluence influenced the properties of the prepared Nb₂O₅ nanostructured material in a liquid environment. The optimum laser fluence to prepare orthorhombic crystalline materials was 12.09 J/cm².

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