

Synthesis of Nb₂O₅ Nanoparticle by Liquid Phase Laser Ablation Method

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ABSTRACT

Niobium pentoxide (Nb₂O₅) nanoparticles were synthesized by laser ablation in water employing 1064nm, 12.09 J/cm² laser fluence and 150 pulses. X-ray pattern showed intense peaks of orthorhombic T-Nb₂O₅ and the grain sizes ranged from (58.245 -244.664nm) while dislocation density was (0.294—0.016nm) and micro strains were (5.2-3.4nm). The optical properties showed an absorption edge at (309nm) and the energy band gap was (4.15eV). TEM image showed spherical agglomerated particles with moderate concentration, while the average particles size was about (40nm). FESEM images also showed small spherical particles aggregated forming larger sized particles and the average particles size was less than (30nm). EDX image confirmed the existence of niobium and oxygen elements and the stoichiometry of niobium pentoxide was 66.03%. AFM image showed vertically oriented (pyramid shaped) particles with average particle size (9nm) while roughness was (3.558nm) and RMS roughness was (4.194nm). FTIR results and Raman spectroscopy showed a peak referring to the Nb-O-Nb bond which indicates the formation of the Nb₂O₅ complex.

Keywords: Liquid-pulsed laser ablation, morphological properties, optical properties, structural properties

1. INTRODUCTION

Laser ablation in liquid is environmentally friendly and chemically clean method to produce nano-scale colloids. Those colloids are very useful in many areas like solar nanofluids, optical limiting devices and so on[1-3]. The surface of the target absorbs the photons coming from the laser beam leading to heating and photo-ionization. Then plasma is generated in the area between the surface of the target and the surrounding fluid, which is called a "laser induced plasma". This eventually leads to a shock wave expanding the plasma to a state with elevated temperature and pressure and higher density[4-6]. This process is schematically showed in the Figure 1.

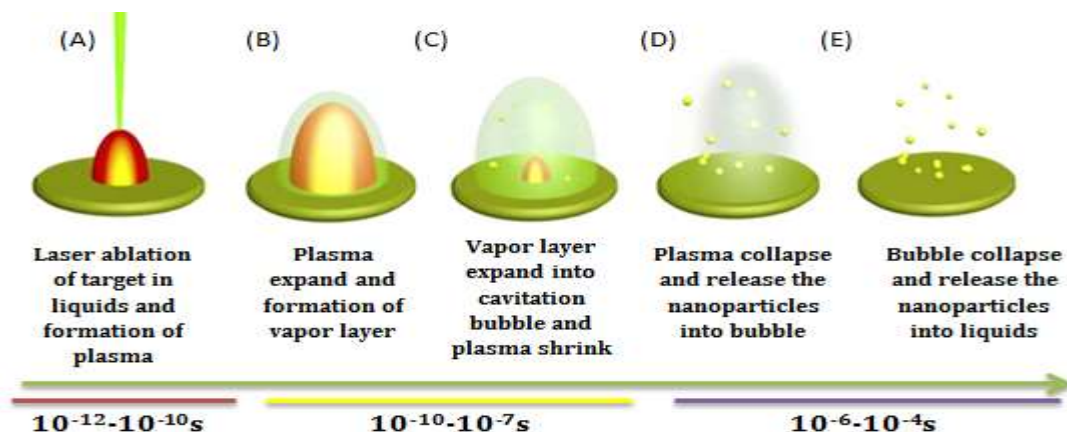


Figure 1. The process and time sequence of PLAL.

Niobium pentoxide is a substantial semiconductor metal oxide with outstanding photocatalyst, gas sensing and biosensing activity. Niobium oxide has diverse stoichiometries, niobia [7-9] is the most thermodynamically stable metal oxide which is chemically inert and low cytotoxicity. Various approaches have been utilized for manufacturing Nb₂O₅ nanostructures, such as thermal oxidation, pulsed laser deposition, anodic oxidation, plasma oxidation and the hydrothermal method and others[10-12].

Osmando F. Lopez et al[13] prepared spherical agglomerated niobium pentoxide nanoparticles by the use of oxidant peroxide method, and showed a significant improvement in morphology by increasing synthesis temperature. Later Gilberto F. et al[14] fabricated Nb₂O₅ nanoparticles by using microwave-assisted method. The band gaps obtained were about 3.1 and 2.9 eV. Nagaraju P. et al [15] prepared orthorhombic-Nb₂O₅ structure and then combined it with graphene as decoration for properties enhancement. In 2020 Liang D. et.al[16] fabricated a pseudohexagonal phase of the Nb₂O₅ nanopillars using the PLAL method followed by the hydrothermal treatment for enhancing the crystallization of the Nb₂O₅ nanoparticles. While Makram et.al [17 prepared Nb₂O₅ nanostructures using the PLD method and obtained a monoclinic phase. In addition he studied the effect of annealing temperature and how it can affect the outcome of the nanostructure phase.

This work aimed to synthesis of niobium pentoxide nano-particles acquired employing ablation of metal target in liquid using laser beam as a tool.

2. EXPERIMENTAL WORK

A plate of Niobium with high purity, deionized water, 1064 nm Nd:YAG laser were utilized in preparation of niobium pentoxide nanoparticles. Samples were fabricated by immersing the Nb metal/plate (99.99 purity) from (Sigma-aldrich) into deionized water and then ablated by Nd:YAG laser 1Hz, 150 pulse and 12.07 J/cm² laser fluence.

The setup of the experiment consisted of Nd:YAG laser using the 1064 nm wavelength, 1 HZ, 150 laser pulses and 12.09 J/cm² laser fluence. A glass container filled with 3 ml having the Nb target in the bottom, this container is 8 cm apart from the laser head. The experiment took place in the laboratories of the university of technology/Baghdad at 10:30 AM. The setup of the experiment is shown in Figure 2.



Figure 2. The setup of the experiment.

For structural analysis the X-ray diffract meter of (Shimadzu 6000) type was employed using 0.15406 nm wavelength from cu- α radiation source. Grain size was calculated using Scherrer's equation[10]

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

where, D is the grain size (nm), K is the dimensionless shape factor, about 0.9 and varies with crystallite shape, λ x-ray wavelength, β is the broadening at half the maximum intensity (FWHM), θ is Bragg's angle. Dislocation density (δ) was calculated using the equation [11]

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

Microstrains was calculated using the equation [12]

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

For optical investigation, the UV-VIS spectrophotometer model (Shimadzu, 1800) was used. Tauc's plot was used for band gap estimation. The following equation can be used for direct inter band transition[13]

$$(\alpha h\nu)^2 = c(h\nu - E_g) \quad (4)$$

Where, α = absorption coefficient, ν = frequency of the incident photon, c = constant.

The chemical bond formation of Nb₂O₅ was insured using Fourier Transform-Infrared Spectroscopy employing (BRUKER-7613) device. Bonding structure was analyzed by a (SUNSHINE-V2-86) for the measurements of Raman spectrum of Nb₂O₅. For morphology analysis, the (Titan 80-300 HRTEM) device was employed to investigate Niobia morphology such as particles shape, size and distribution.

The FESEM (ARYA Electron Optic) device equipped with an Energy Dispersive X-ray (EDX) was used for surface morphology, particle size, and stoichiometry and element percentages in the sample.

3. RESULTS AND DISCUSSION

Figure 3 shows XRD pattern for niobium pentoxide nanoparticles prepared by using (150) laser pulses and 12.09 J/cm². The figure shows multiple diffraction peaks located at diffraction angles of 16.8°, 22.8°, 35.8°, 42.8°, 47.4° and 48.4° that corresponds to (1 3 0), (0 0 1), (1 0 1), (1 13 0), (0 0 2) and (1 1 0) diffraction planes respectively. These peaks belong to the orthorhombic T-Nb₂O₅ structure based on JCPDS card number (00-030-0873), while peaks located at diffraction angle $2\theta = 30.6^\circ$ belong to pure nobium according to JCPDS card number (00-003-0905) [14-17]. While, in 2020 Liang et.al [8] prepared a pseudohexagonal TT-Nb₂O₅ nanopillars using the same preparation method.

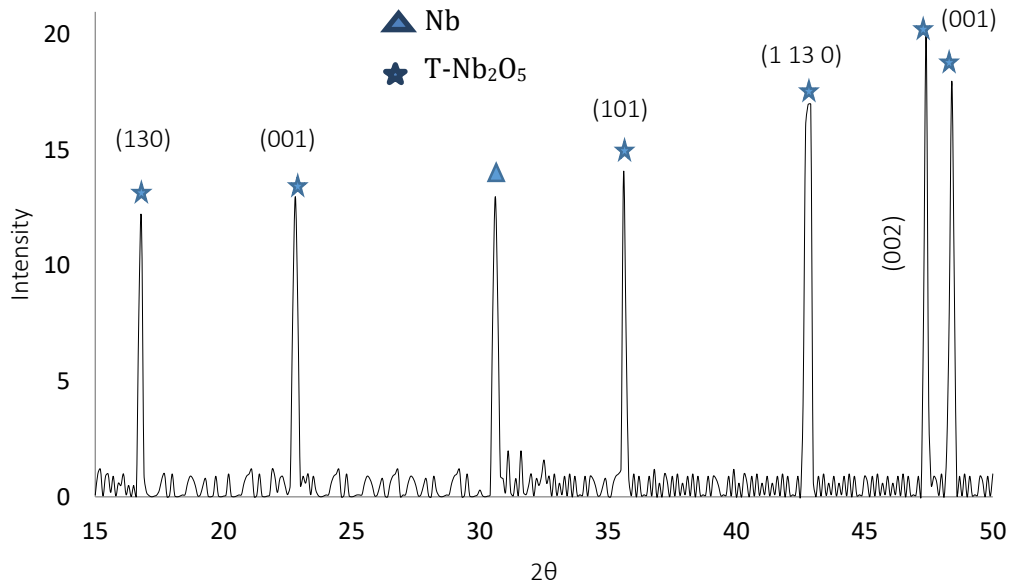


Figure 3. XRD profile of Nb₂O₅ nanoparticles for T-Nb₂O₅ and pure Nb.

Table 1 depicts that grain size increased while dislocation density and microstrains decreased as 2θ increases. In general, the micro-strain represents the root mean square (RMS) of the variations in the lattice parameters across the sample. It has been found that the lattice parameters inversely related with crystallite size, as well as the lattice strain increases as crystallite-size increase.

Table 1 Grain size, dislocation density and microstrains for Nb₂O₅ nanoparticles

2θ	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 1 3 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 lattice constants of T-Nb₂O₅ were calculated manually by using the Cramer-Cohen method in accordance with data from JCPDS file of the orthorhombic structure. This calculation declares that the values of the lattice constants are almost the same as the data of the JCPDS of the T-Nb₂O₅ nanostructures. The lattice constants were calculated based upon five peaks.

The orthorhombic phase lattice constants are calculated using the following equations:

$$\lambda = 2d \sin \theta \quad (5)$$

$$\frac{1}{d^2} = \frac{h^2}{a^2} + \frac{k^2}{b^2} + \frac{l^2}{c^2} \quad (6)$$

$$\left. \begin{aligned} \Sigma \alpha \sin^2 \theta &= C \Sigma \alpha^2 + B \Sigma \alpha \gamma + A \Sigma \alpha \delta \\ \Sigma \gamma \sin^2 \theta &= C \Sigma \alpha \gamma + B \Sigma \gamma^2 + A \Sigma \gamma \delta \\ \Sigma \delta \sin^2 \theta &= C \Sigma \alpha \delta + B \Sigma \gamma \delta + A \Sigma \delta^2 \end{aligned} \right\} \quad (7)$$

Where d is the interplanar spacing, a,b and c are the lattice constants, h,k and l is the plane indices, λ is the X-ray wavelength (1.54056 Angstrom), θ is the diffraction angle, $\alpha = h^2 + k^2 + l^2$, $\delta = 10 \sin^2 2\theta$, $\gamma = l^2$, A,B and C are numerators. Table 2 contains a detailed analysis of the data for the lattice constants of the orthorhombic phase of the crystal structure.

Table 2 Analysis of lattice constants for the orthorhombic crystal structure

2 θ	θ	hkl	Sin θ	Sin ² θ
16.8	8.1	130	0.14	0.019
22.8	11.4	001	0.19	0.039
35.8	17.9	101	0.3	0.094
47.4	23.7	002	0.4	0.16
48.4	24.2	001	0.41	0.17

Table 2 (Continued)

α	α^2	γ	γ^2	δ	δ^2
10	100	0	0	0.778	0.605
1	1	1	1	0.150	0.0225
2	4	1	1	0.342	0.116
4	16	4	16	0.541	0.292
1	1	1	1	0.559	0.312
$\Sigma \alpha = 18$	$\Sigma \alpha^2 = 122$	$\Sigma \gamma = 7$	$\Sigma \gamma^2 = 19$	$\Sigma \delta = 2.37$	$\Sigma \delta^2 = 1.347$

Table 2 (Continued)

$\alpha \sin^2 \theta$	$\alpha \delta$	$\gamma \sin^2 \theta$	$\alpha \gamma$	$\gamma \delta$	$\delta \sin^2 \theta$	$\alpha \delta$
0.19	7.78	0	0	0	0.0147	7.78
0.039	0.150	0.039	1	0.150	0.005	0.150
0.188	0.684	0.094	2	0.342	0.032	0.684
0.64	2.164	0.64	16	2.164	0.086	2.164
0.17	0.559	0.17	1	0.559	0.09	0.559
$\Sigma \alpha \sin^2 \theta = 1.227$	$\Sigma \alpha \delta = 10.653$	$\Sigma \gamma \sin^2 \theta = 0.943$	$\Sigma \alpha \gamma = 20$	$\Sigma \gamma \delta = 3.215$	$\Sigma \delta \sin^2 \theta = 0.232$	$\Sigma \alpha \delta = 11.337$

By substituting the values of the matrix we get:

$$1.227 = 122 C + 18 B + 11.337 A$$

$$0.943 = 20 C + 19 B + 3.15 A$$

$$0.23 = 11.337 C + 3.215 B + 1.347 A$$

By solving the matrix we can obtain the values of A, B and C and by using the following equations we can get the values of the lattice constants:

$$\left. \begin{aligned} A &= \frac{\lambda^2}{4a^2} \\ B &= \frac{\lambda^2}{4b^2} \\ C &= \frac{\lambda^2}{3c^2} \end{aligned} \right\} \quad (8)$$

The above mentioned formulas give the values of the lattice constants:

$$a = 5.9583 \text{ \AA}$$

$$b = 8.9325 \text{ \AA}$$

$$c = 8.8421 \text{ \AA}$$

these results are nearly consisted with the orthorhombic lattice constants.

Figure 4 displays the absorption spectrum and the energy gap of Nb₂O₅. The figure shows the absorbance peak was located in the UV region of the spectrum at almost 200 nm while the absorbance edge was located in 309nm. Additionally, the related band gap was about 4.15eV which agree with other reported works [18-21].

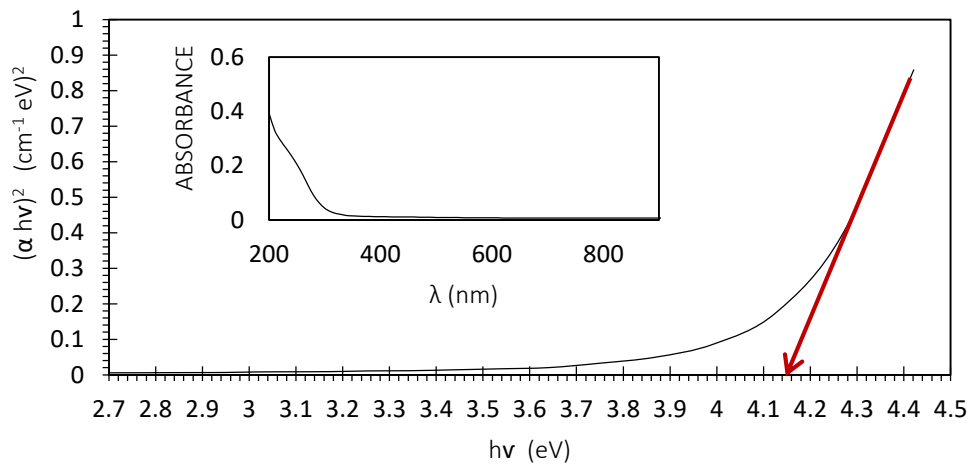


Figure 4. E_g and the absorption spectrum of Nb₂O₅.

Figure 5 shows TEM image and average particle size histogram with spherical shape nanoparticles. TEM images show small agglomerated spherical particles with moderate concentration (this was estimated by measuring the weight of the target before and after the ablation process and then calculating the mass concentration of Nb₂O₅ nanoparticles by using the equation (m₁-m₂/liquid volume)). This result is consistent with other reported work[22]. The histogram for TEM image was obtained by using ImageJ software. The histogram shows an average particle size about 40nm.

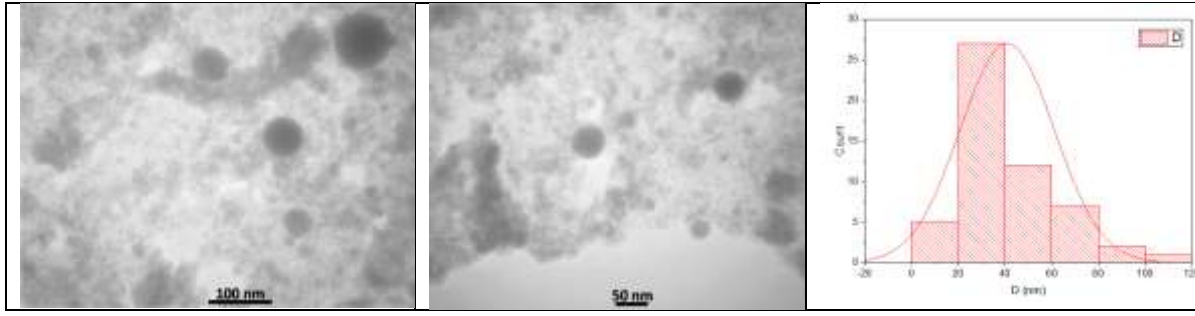


Figure 5. TEM and particle size histogram of Nb₂O₅.

Figure 6 shows FESEM images and histogram for Nb₂O₅ nanoparticles. The figure shows that the morphology of Nb₂O₅ nanoparticles relies on laser fluence and on the environment of the experiment. Particles are spherical and agglomerated, this result agrees with other reported works [23]. The morphology basically depends on the characteristics of the generated plasma on the surface of the target which can create localized high temperature and high pressure plasma plumes due to the interaction of the laser with the target [24, 25].

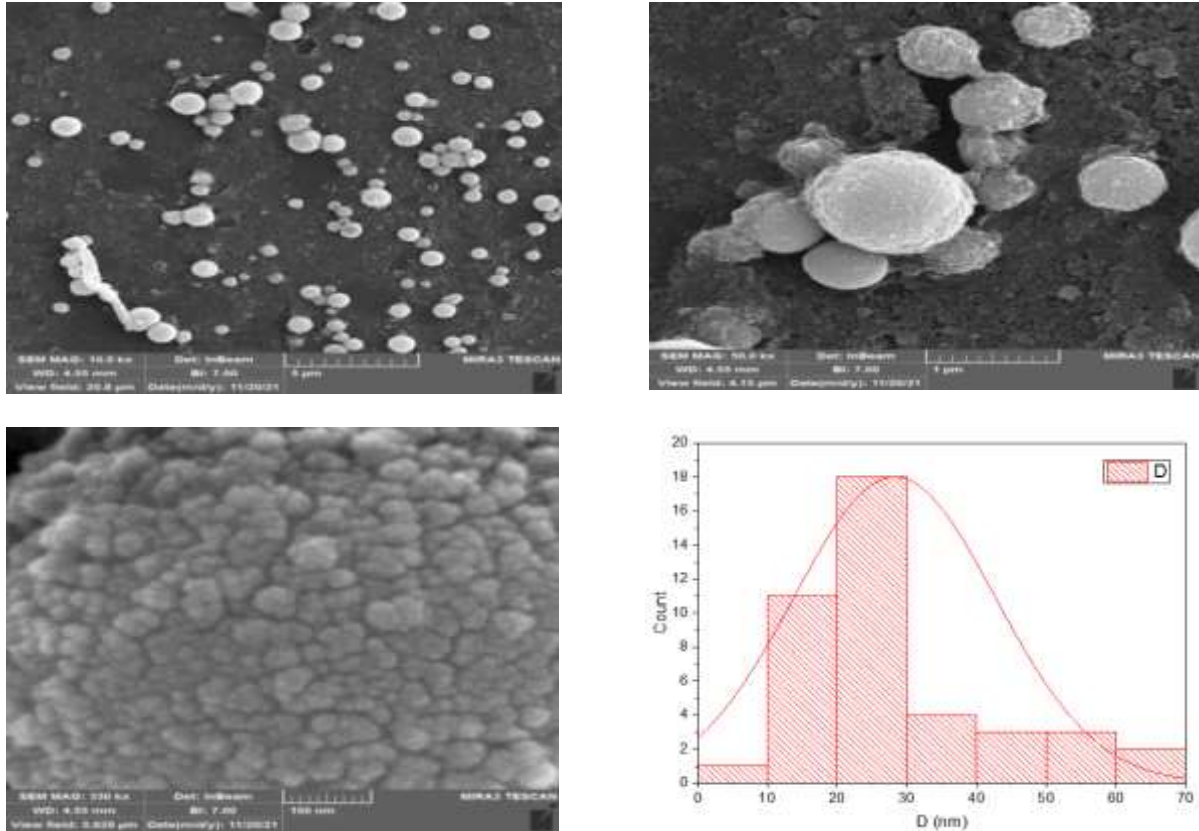


Figure 6. FESEM with particle size histogram of Nb₂O₅ nanoparticles.

The histogram for FESEM images was obtained by using ImageJ software. The histogram shows an average particle size less than 30nm. Figure 7 shows EDX image for Nb₂O₅ nanoparticles. The spectrum confirms the presence of Niobium, Oxygen, the presence of Silicon and Carbon elements may relate to used substrate. The stoichiometry of Nb₂O₅ was about 66.03%. Table 3 presents the purity and stoichiometry of Nb₂O₅ nanoparticles and other elements.

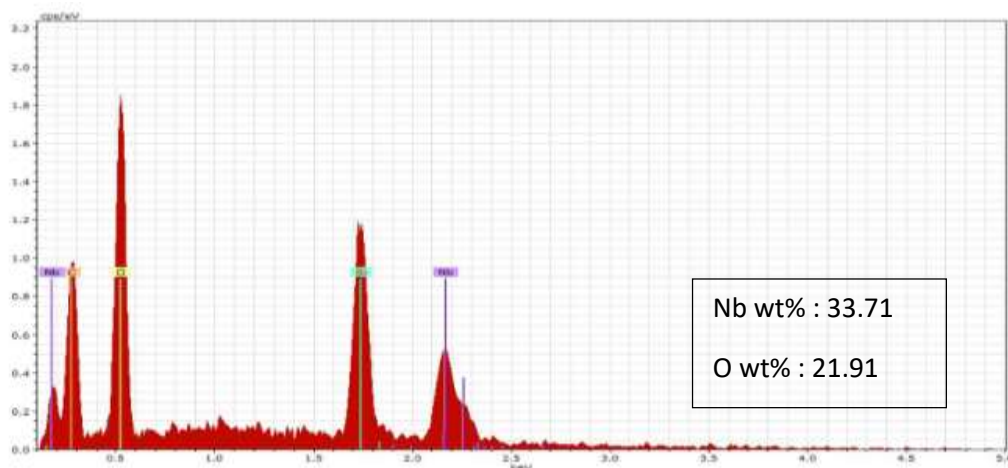


Figure 7. EDX spectra for Nb₂O₅ nanoparticles.

Table 3 Purity and stoichiometry of Nb, O and Nb₂O₅ nanoparticles

Nb wt%	O wt%	Nb ₂ O ₅ Stoichiometry %
33.71	21.91	66.03

Figure 8 shows topographical image of the surfaces and average size histograms of Nb₂O₅. AFM results showed vertically oriented particles (pyramid shaped) distributed all over the surface which indicates a good crystallinity [26], while average particle size was about 9nm, Roughness (the average of the measured peaks and valleys) was 3.558nm, lastly RMS roughness (is the root mean square of the measured peaks and valleys [27]) was 4.194nm. These results are consisted with other reported work [28].

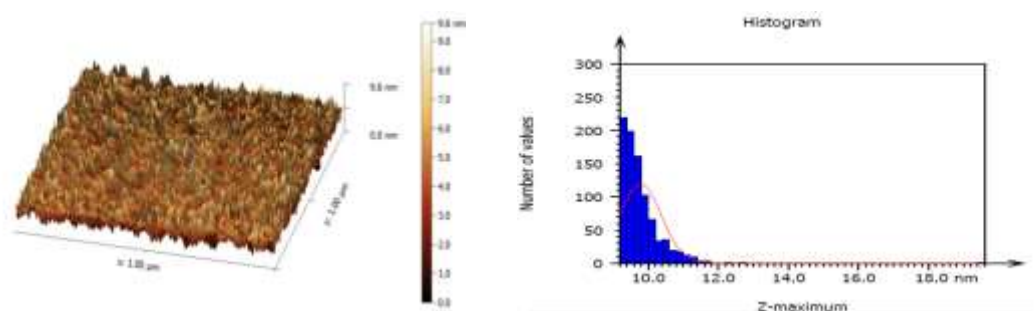


Figure 8. AFM image for Nb₂O₅ nanoparticles.

From Figure 9 the peak 713.6 cm⁻¹ refers to the Nb-O-Nb stretching vibration mode which indicates the formation of niobium oxide complex also this peak indicates the formation of T-Nb₂O₅ structure [29] and this result agrees strongly with XRD result and Raman spectrum. The peak 1631.7 cm⁻¹ is attributed to the bending vibration of H₂O molecule and the weak asymmetric band presence of OH group [30]. While the peak 2113.98 cm⁻¹ is attributed to pure niobium Nb [31]. Lastly, the peak 3255.84 cm⁻¹ is assigned to the OH stretching vibration of Nb-OH[31]. Chemical bonds and their IR resonance positions in Nb₂O₅ Nanoparticles colloidal are presented in Table 4. These peaks are in good agreement with other reported works mentioned in the table.

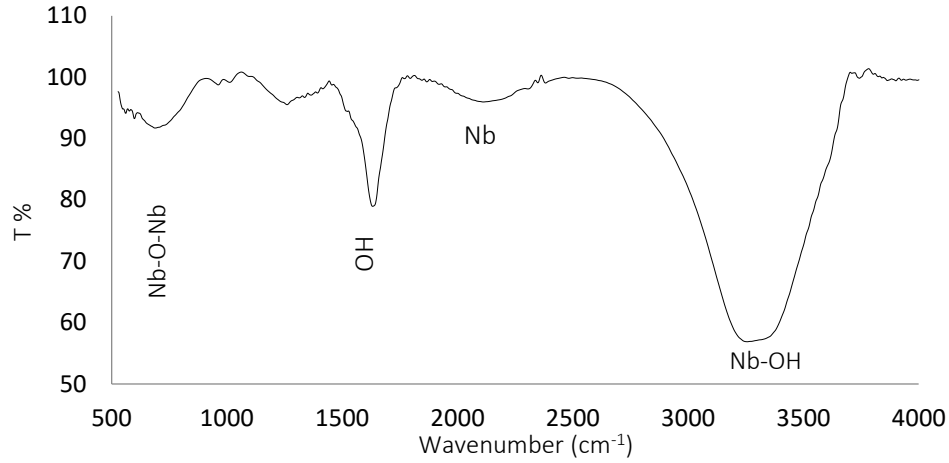


Figure 9. FTIR spectrum of Nb₂O₅ nanoparticles.

Table 4 Chemical bonds and their positions

Obtained bond value(cm ⁻¹)	Assignment	Vibration mode	Reference
713.6	Nb-O-Nb	stretching	[32-35]
1631.7	O-H	bending	[30, 34, 36, 37]
2113.98	Pure Nb		[31]
3255.84	Nb-OH	stretching	[30, 36]

Figure 10 shows the Raman spectrum of Nb₂O₅ Nanoparticles. The Raman spectrum contains the peak 298.45 cm⁻¹ which is related to the orthorhombic T-Nb₂O₅ [38] which mainly agrees with FTIR and XRD results. while the peak 649.15 cm⁻¹ is related to stretching modes of NbO₆ polyhedral typical of the orthorhombic Nb₂O₅ crystalline structure [37].

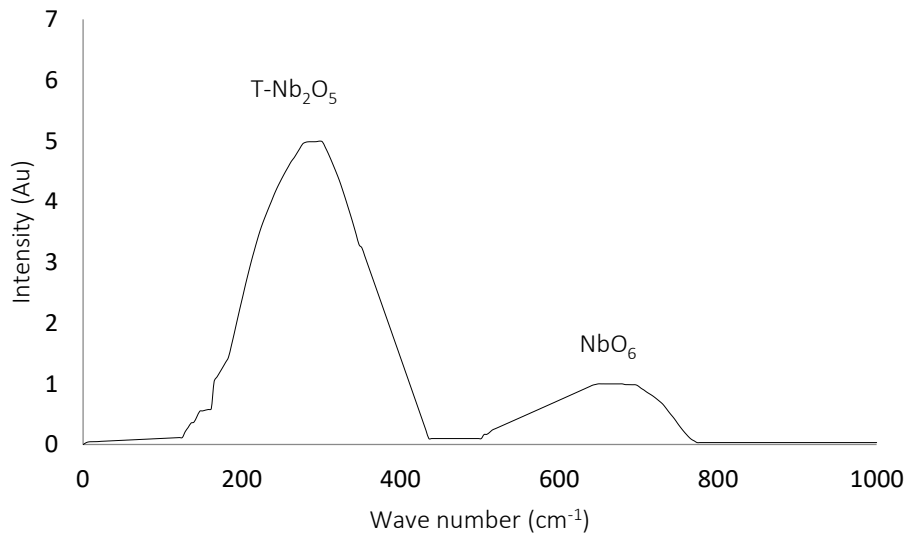


Figure 10. Raman spectrum of Nb₂O₅ nanoparticles.

4. CONCLUSION

In summary, this paper demonstrates a successful method for the preparation of Nb₂O₅ nanoparticles by laser ablation of Nb plate target in deionized water at room temperature. The UV-Visible spectrum shows maximum absorbance around UV region and the energy gap was around 4.15 eV. This method was useful in the formation of spherical particles as shown in SEM and TEM results. XRD, FTIR, Raman spectroscopy confirmed the formation of orthorhombic T-Nb₂O₅ structure. XRD measurements showed that the grain size of Nb₂O₅ nanoparticles increased from 58.245 nm to 244.664 nm as 2θ increased. While the dislocation density reduced from 0.294 to 0.016 nm. Lastly the microstrains reduced from 5.2 to 3.4 nm. TEM images showed small agglomerated particles with average particle size about 40 nm. While FESEM images showed small spherical fragments agglomerated together forming larger sized particles with average size about 30 nm. In addition, EDX showed that the stoichiometry of Nb₂O₅ nanostructures was about 66.03 %. AFM results showed vertically oriented particles with an average particle size about 9nm, the roughness was 3.558nm and the RMS roughness was 4.194 nm.

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