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Zinc Oxide Nanoparticles-Poly(vinyl) Alcohol Thin Film for Generation Q-Switched Erbium-Doped Fiber Laser

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ABSTRACT

The nonlinear optical property of Zinc oxide (ZnO) nanoparticles thin film synthesized by the solution casting method was investigated from the continuous wave to the Q-switching using an erbium-doped fiber laser cavity. Our laser experiment shows that ZnO nanoparticles embedded with polyvinyl alcohol (PVA) allow for the generation of a stable Q-switching laser pulse, centered at 1560.08nm with a 3-dB spectral bandwidth of 0.941 nm, a pulse duration of 3.53 µs and a high pulse energy of 171 nJ. Our finding suggests that ZnO nanoparticles thin film can be a useful nonlinear optical material for laser photonics devices such as passive q-switching for applications involving high energy.

1. Introduction

Saturable absorbers (SAs) or non-linear modulators are used in order to provide self-amplitude modulation for passive lasers [1]. The evolution of real SA began in 1964 by using coloured glass or dyes before advancing into 2-dimensional (2D) material [2]. The first incorporation of 2D material in fiber laser started in 2009 with graphene which is known for its zero-bandgap property that provides a wideband optical operation [3]. Over a decade later, graphene along with its 2D counterparts including topological insulators (TIs), transition metal dichalcogenides (TMDs), and MXene [4,5].

The recent advancements in fiber laser has opened opportunities for transition metal oxide (TMO) to be used as an SA. Transition metal oxide is attracting attention for their large optical nonlinearity, thermal stability and robustness. It has also been widely accepted that the same electrons, those originally connected to the d orbitals of the transition-metal ions, are in charge of not only the magnetic properties but also the electrical and low-energy optical capabilities [6].

Zinc Oxide (ZnO), a TMO semiconductor that exhibits an efficient blue-green emission has an advantage of a low-temperature band-gap which is around 3.437 eV with a free exciton occurring at

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3.372 eV giving binding energy of approximately 65 meV which is shorter compared to other TMOs [7,8]. Besides having a wide band gap which is beneficial in optical applications, it is also more resistant to radiation damage [9]. Furthermore, in nanoparticles form, it is easily a better alternative for the environment due to its biocompatibility, nontoxicity and pollution-free nature [10-13], proven by its capability to act as a medium for drugs and cosmetics formulations [14-15].

The generation of pulse in fiber laser by using ZnO as the SA started in 2016 when Ahmad *et. al.,* [16] fabricated a ZnO nanoparticles thin film by mixing ZnO powder from Sigma Aldrich with a particles size average of 20-50 nm into silane and ethanol. The thickness of the film was reported to be 0.15 ± 0.1 nm with a saturation intensity and modulation depth of 0.016MW cm^{-2} and 3.5%, respectively. The SA produced a Q-switched EDFL with a highest pulse energy of 47.9 nJ. ZnO based SA was also reported in 2017 by Aziz *et al.,* [17] producing a Q-switched pulse train with a repetition rate of 61.43 kHz and the shortest pulse width of 7.00 µs. To synthesize the ZnO powder, zinc nitrate hexahydrate with hexamethylenetetramine were dissolved in deionized (DI) water. The synthesis process includes stirring the solution for 5 minutes before placing it in the oven for 5 hours. The precipitate that forms after being heated for 5 hours was the ZnO powder. The ZnO powder was then dissolved in DI water through a centrifuge before mixing with polyvinyl alcohol (PVA) to form a film. The thickness of the film was measured to be approximately 50 µm. In the following year, Norizan *et al.,* [18,20] reported a method to fabricate a ZnO film by adding a ZnO powder (Alfa Aesar, 99.99% trace metal basis, 81.35 Mw) directly in the solution of polyvinyl alcohol (PVA). The ZnO- PVA mixture then undergoes sonication and centrifuges before decanted in a petri dish. The thickness was determined to be 22 µm [18]. The SA yielded a pulse width of 4.52 µs and a pulse energy of 31.6 nJ.

Therefore, this research aim is to provide a better performance and simple fabrication process of ZnO nanoparticles film to be used as saturable absorber.

2. Preparation and Characterization of ZnO NP/PVA Saturable Absorber

2.1 Preparation of ZnO NP/PVA SA

All the chemicals were used in the experiment without any further purifications. Initially, 25 mg ZnO NP (99.99 % trace metal basis, Alfa Aesar) with particles size of 40-100 nm was dispersed in 10 mL of ethanol (99.6 %, R&M Chemicals) and ultrasonicated for 3 hours to improve the dispersibility of the ZnO NP in the ethanol. The PVA solution was prepared by dissolving PVA powder (40000 MW, Sigma Aldrich) in 120 mL filtered DI water and stirred at a temperature of 145 °C until the powder completely dissolved. After that, 2.5 mL ZnO NP dispersion was added into 2.5 mL of PVA solution and slowly stirred for an hour without any aid of heat. To fabricate the free-standing ZnO NP/PVA SA, dry casting technique were employed by transferring the ZnO NP/PVA solution into a petri dish and placed inside the desiccator cabinet for 3 days. After 3 days, the film was slowly peeled and kept in a vacuum bag to avoid any contaminations. Figure 1. shows the step for the preparation of ZnO NP/PVA SA.

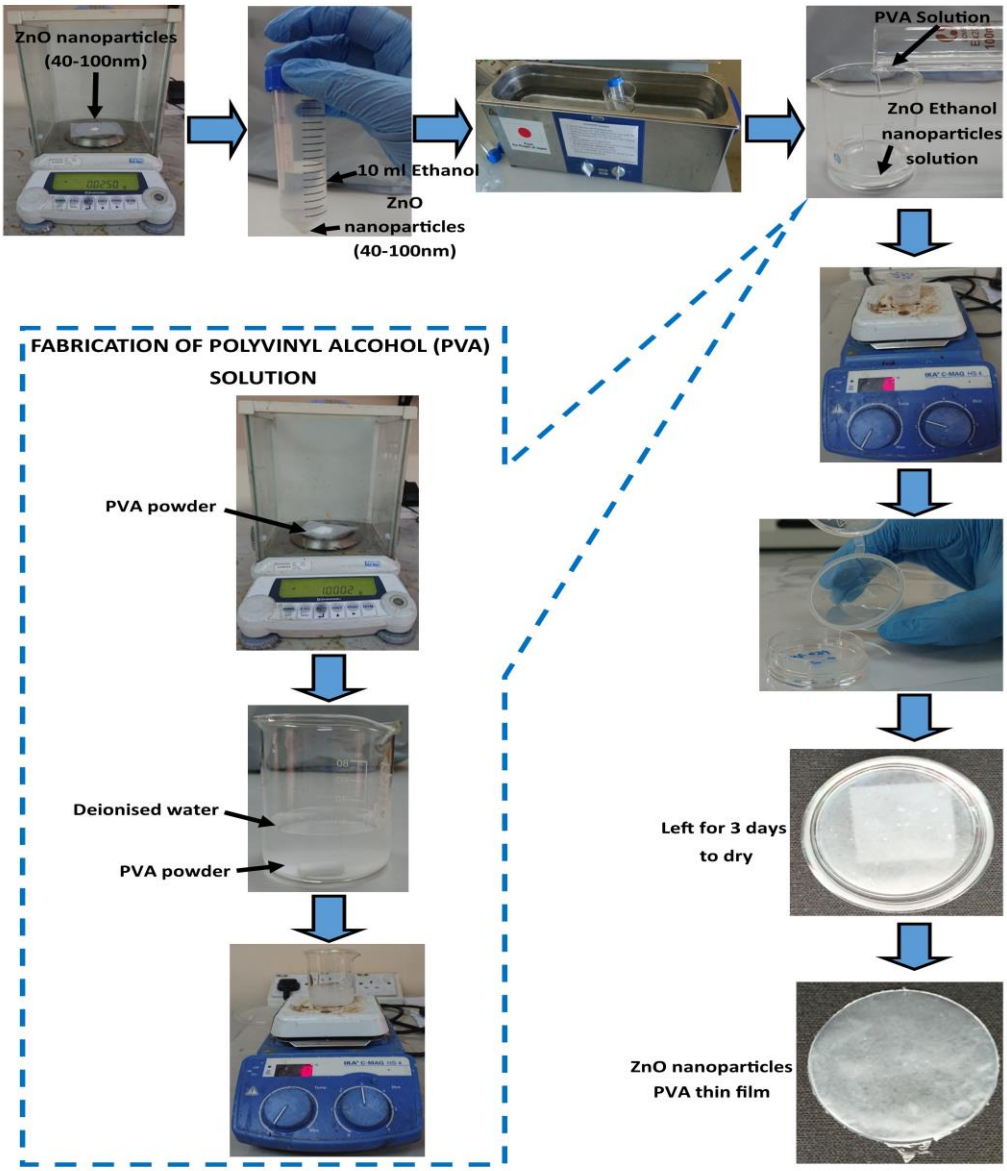

Fig. 1. Step by step preparation of ZnO NP/PVA film-based SA

2.2 Characterization of ZnO NP/PVA SA

The surface morphology of the ZnO NP/PVA film was obtained using field emission scanning electron microscopy (FESEM) (JEOL, JSM-7800F) as displayed in Figure 2(a). The ZnO NP/PVA film was cut into 5 mm × 5 mm and mounted on the aluminium stub using a conducting tape. The FESEM equipped with Schottky electron gun was fired on the sample and the morphology was displayed on the monitor. The 5 kX magnification image with accelerating voltage 1 kV shows that the ZnO NP was homogenously embedded inside the PVA polymer. Inset of the Figure 2(a) shows the physical appearance of ZnO NP/VA film deposited on a FC/PC fiber ferrule with 1 mm × 1 mm dimension, captured using a USB portable microscope. The ZnO NP/PVA film was carefully aligned using tweezers in order to make sure the film was exactly placed on the core of the fiber ferrule. Besides that, a small amount of index matching index gel was applied on the FC/PC to prevent the SA from moving during the experiment. Figure 2(b). shows the chemical elemental spectrum of ZnO NP/PVA detected using energy dispersive spectroscopy (EDS) through spot scanning technique. Three elements were

detected which was the zinc (Zn), oxygen (O) and carbon (C). The Zn element shows the highest percentage of 75.69 % at peak around 1 keV and followed by O element at around 0.5 keV indicating that the nanoparticles of ZnO is with high purity. The carbon element detected was due to the composition of PVA with chemical formula of $[CH_2CH(OH)]_n$. Figure 2(c) and Figure 2(d) shows the thickness profile of ZnO NP/PVA which was measured using 3D laser scanning microscope (Olympus, OLS4100). The thickness of 85.99 µm was measured by the difference in height of the ZnO NP/PVA and the microscope stage.

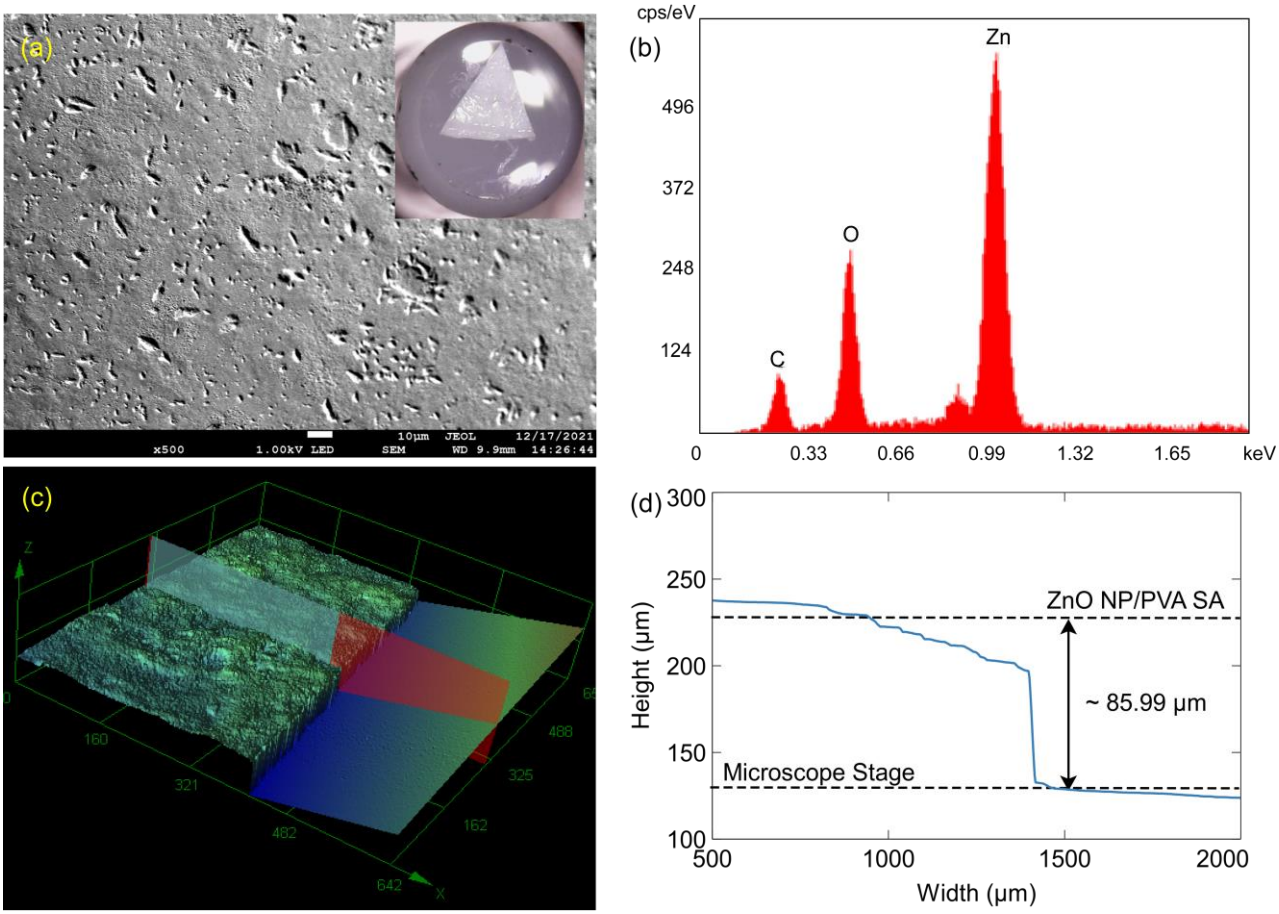

Fig. 2. ZnO NP/PVA film's (a) FESEM image (b) EDS spectrum (c) 3D thickness profile (d) Thickness measurement

2.3 Erbium-doped Fiber Laser Cavity

The schematic setup of the erbium-doped fiber laser (EDFL) cavity in ring configuration used in the experiment is shown in Figure 3. The laser cavity consists of a 2.8 m long erbium doped fibre (EDF) as the gain medium, a wavelength division multiplexer (WDM), an isolator, the fabricated ZnO nanoparticles-polymer SA and an 80/20 output coupler. A laser diode with a central wavelength of 980 nm was used as the pump source. The light was pumped to the gain medium via the WDM. The EDF used has a numerical aperture (NA) of 0.16 and Erbium ion absorption of 23 dB/m at 980 nm with a core and cladding diameters of 4 µm and 125 µm, respectively. To ensure unidirectional propagation of the oscillating laser in the ring laser cavity, a polarization independent isolator was used. The laser signal was coupled out using an 80:20 output coupler which keeps 80% of the light oscillating in the ring cavity for both spectral and temporal diagnostics. The output laser was tapped out from a 20 % port of the coupler. The spectral characteristic was measured using an optical

spectrum analyzer (OSA) with a spectral resolution of 0.02 nm while the temporal characteristics were measured using a 500 MHz oscilloscope and a 7.8 GHz radio-frequency (RF) spectrum analyser via a 1.2 GHz photodetector.

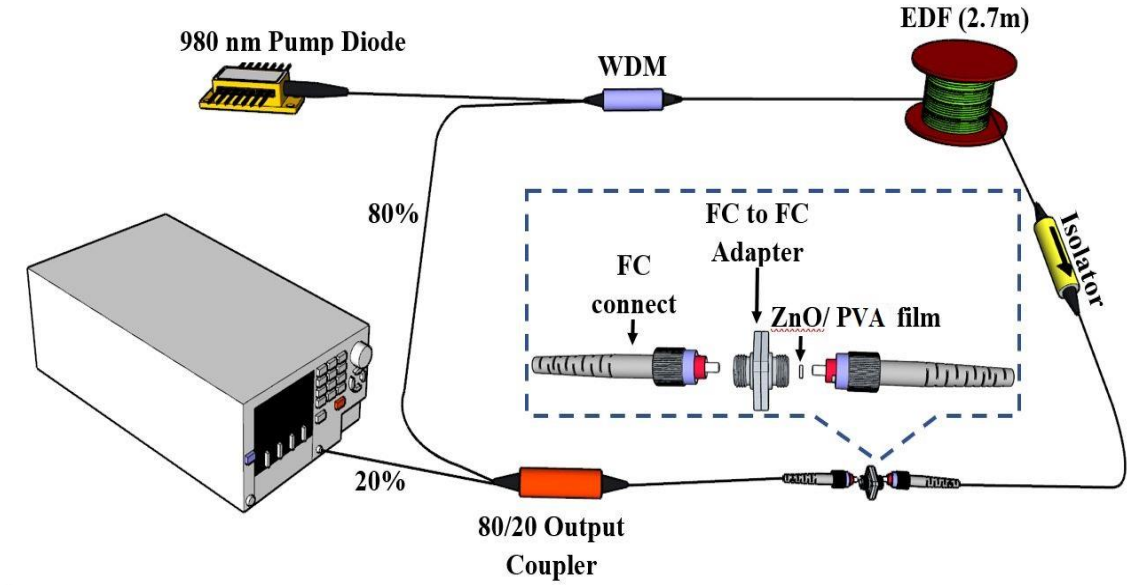

Fig. 3. Erbium-doped fiber laser set up the cavity

3. Results and Discussion

Upon the insertion of the ZnO NP- PVA-based SA in the EDFL cavity, a passive Q-switching commenced at the pump power of 29.41 mW which was kept stable until the maximum pump power of 157.01 mW. Figure 4 shows the output spectrum of the Q-switching laser at 1560.08 nm with a measured 3 dB bandwidth of 0.933 nm.

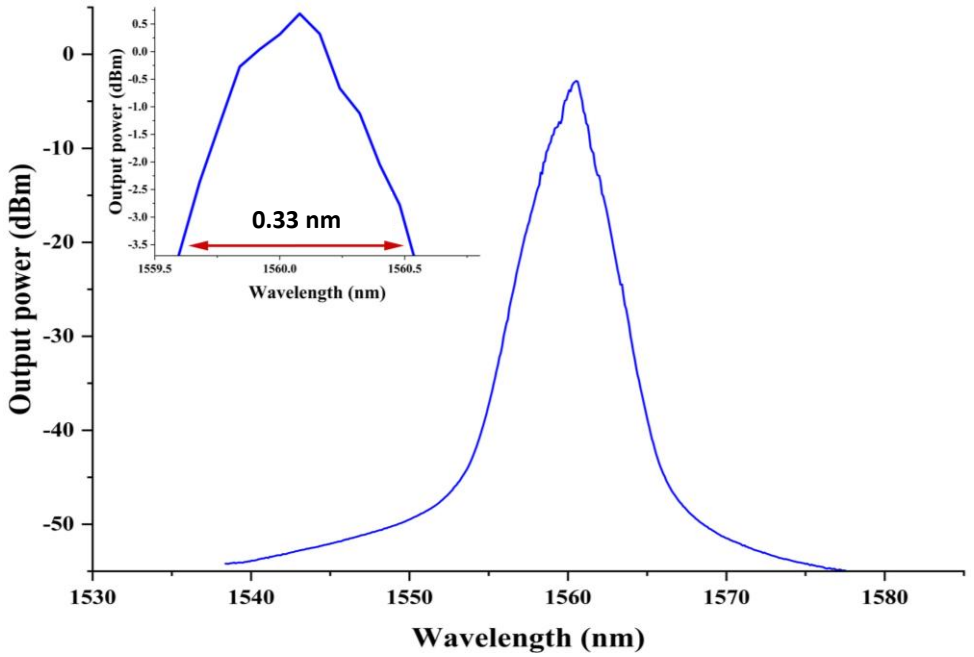

Fig. 4. Optical spectrum of the ZnO nanoparticles-PVA SA based Q-switched EDFL at 157.01 mW

The recorded typical pulse train of the ZnO NP-PVA SA-based Q-switched EDFL at the maximum input pump power is as shown in Figure 5. The repetition rate of around 30 kHz was recorded with pulse separation of 33.3 µs. The output pulse train was stable without any amplitude modulations, proving that during the Q-switching operation, no self-mode locking effect was present. The pulse has an almost symmetry shape with a pulse width of approximately 3.53 µs.

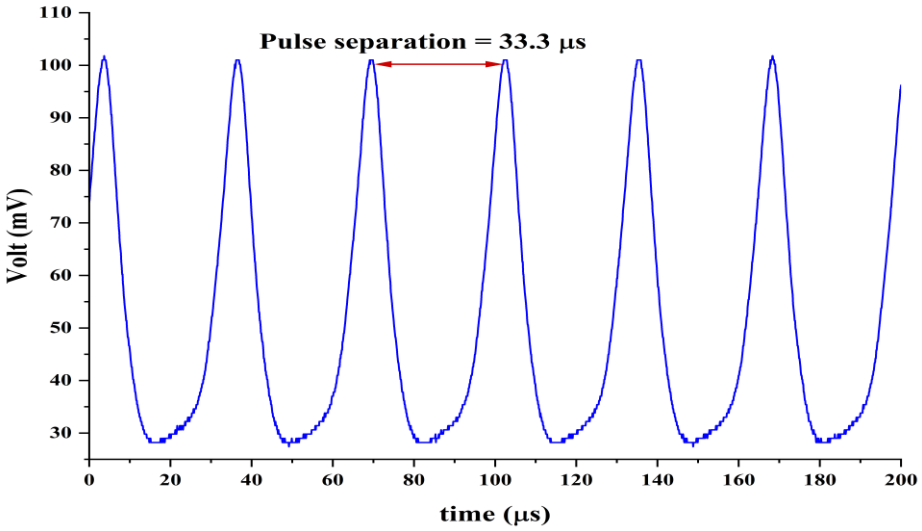

Fig. 5. Pulse train of ZnO nanoparticles-PVA SA based EDFL at 157.01 mW

The repetition rate and pulse duration as a function of pump power is plotted in Figure 6. Increasing the pump power from 29.41 mW to 157.01 mW causes the repetition rate to grow almost monotonously from 37.94 kHz to 75.79 kHz. The pulse repetition rate increased almost linearly with the pump power at the rate of ~0.48 kHz/mW. On the other hand, the pulse duration decreased from 11.32 µs to 3.53 µs with the increase of the pump power which is narrower than some of the reported works on ZnO based SA [17-20]. At lower pump power of less than 30 mW, the pulse duration dropped exponentially. At higher pump power (>25 mW) the pulse duration can be seen to reduce in a steadier rate while the repetition rate increase. As the pump power was steadily increased, more power circulated inside the laser cavity, thus hastening the saturation of SA. The smaller decrement of pulse duration from the pump power of 29.41 mW to 157.01 mW indicates the SA was becoming saturated with increasing light intensity. It is believed that the repetition rate can be further increased while the pulse duration can be smaller with higher pump power since the Q-switched operation remained stable throughout the experiment but it was limited by the maximum power the pump can provide.

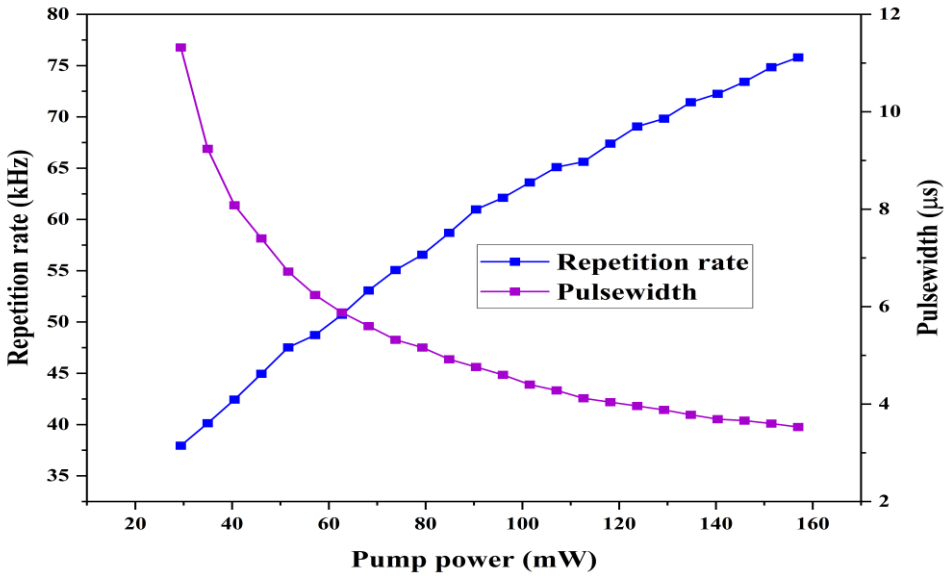

Fig. 6. Pulse width and repetition rate versus incident pump power

Figure 7 illustrates the instantaneous peak power and pulse width as a function of pump power of the Q-switched EDFL. The instantaneous peak power and pulse energy of the Q-switched laser were 45.654 mW and 171 nJ, respectively, at the maximum input pump power. Both of the parameters increased almost linearly with the increasing pump power, typical of a Q-switched laser. The obtained pulse energy is considerably higher than the works using ZnO based SA in the same region [16-19,21].

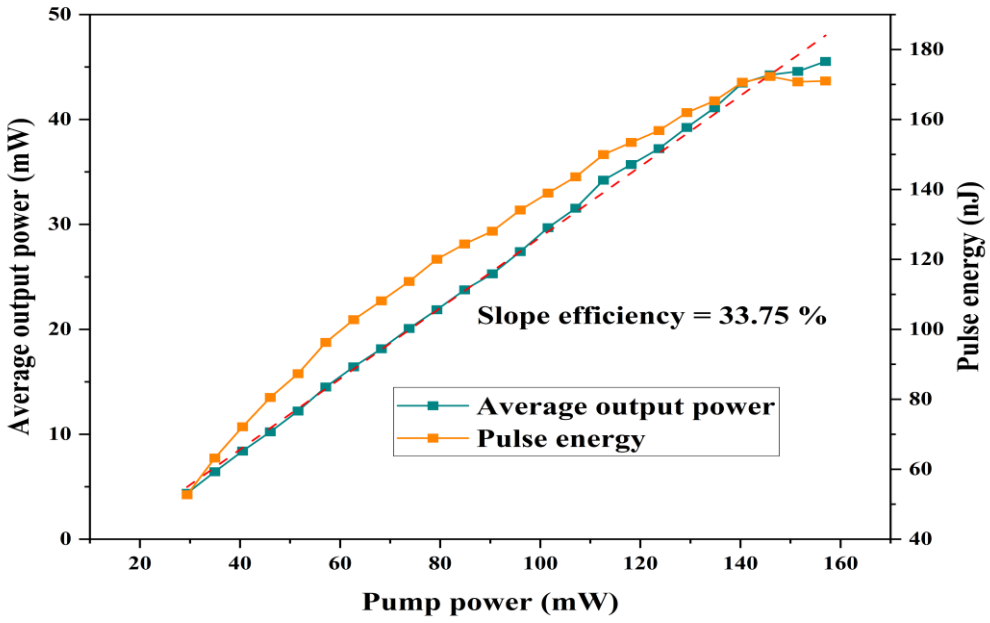

Fig. 7. Pulse energy and average output power versus incident pump power

To investigate the stability of our Q-switched pulse, the RF spectrum was obtained at the maximum pump power of 157.01 mW as shown in Figure 8. The RF spectrum shows the fundamental frequency of 75.79 kHz with a high signal to noise ratio (SNR) of ~40.44 dBm, indicating a very stable Q-switched operation.

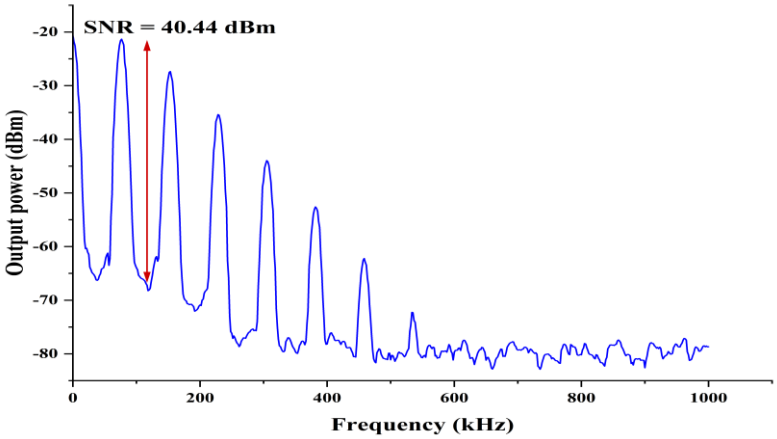

Fig. 8. RF spectrum of ZnO nanoparticles-PVA

The ZnO nanoparticles- PVA film has being left in the cavity for one hour with a maximum pump power of 157.01 mW to determine film durability and its performance. After one hour, the Q-switch pulse train was stable without any fluctuations. This proves that ZnO nanoparticles-PVA film able to sustain a high illumination of incident light at a pump power of approximately 157.01 mW. Above than that, could not be test due to the laser diode has reach maximum pump. Even though, there is higher pump power and shortest pulse width in the previous research of ZnO saturable absorber, this work shows a highest repetition rate and pulse energy. Table 1 shows the overall comparison of this work with previous research. ZnO nanoparticles-PVA in this work is easier to fabricate and its performance is better compared to previous research.

Table 1
ZnO saturable absorber performance comparison of EDF laser

Material and size	Fabrication method	Max. pump power (mW)	λ (nm)	Max. repetition rate (kHz)	Shortest pulse-width (μs)	Max. pulse energy (nJ)	SNR (dB)	Ref
ZnO NP-PEO (20-50 nm)	Hydrothermal method and solution casting	360	1560.9	69.4	1.68	39.9	42	Peck *et al.,* [10]
ZnO-PVA	Hydrothermal method and solution casting	77.9	1560.4	61.43	7.00	154.6	62	Cheah and Sum [11]
ZnO-PVA	Solution casting	134.8	1557.6	71.84	4.52	31.6	61	Hamrayev *et al.,* [12]
ZnO NP-PVA (40-100 nm)	Solution casting	157.01	1560.08	75.79	3.53	171.00	45.65	This work

4. Conclusions

A passively Q-switched EDFL is experimentally demonstrated using ZnO nanoparticles film-based SA as a Q-switcher. The Q-switching was successfully achieved with a maximum repetition rate of 75.79 kHz and a smallest pulsed width 3.53 μs. The ZnO nanoparticles-PVA SA based EDFL also produced a high pulse energy of 171 nJ. These results verify that the presented ZnO nanoparticles is a good candidate as a Q-switcher in the telecommunication region.

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