

**ORGANIC MATERIALS AS SATURABLE ABSORBER  
FOR PULSE GENERATION AT TELECOMMUNICATION  
WINDOW**

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**FACULTY OF ENGINEERING  
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# ORGANIC MATERIALS AS SATURABLE ABSORBER FOR PULSE GENERATION AT TELECOMMUNICATION WINDOW

## ABSTRACT

This thesis demonstrates several types of fiber lasers based on newly developed organic materials as saturable absorbers (SAs). We used these SAs in all-fiber based erbium-doped fiber laser (EDFL) ring cavity to produce Q-switched and mode-locked pulses at telecommunication window, at  $\sim 1560$  nm. Three different organic materials were used in this thesis: Bis[2-(4,6-difluorophenyl)pyridinato- $C^2,N$ ](picolinato)iridium(III) (FIrpic), bis(8-hydroxyquinoline)zinc ( $Znq_2$ ) and Tris(8-hydroxyquinoline)aluminum ( $Alq_3$ ). Finding new materials is still one of the defining characteristics of nonlinear optics field, as the rapid development of SAs has provided several new opportunities for nonlinear optics. Throughout this work, organic material showed a very interesting performance in terms of pulse duration, pulse energy, damage threshold, stability, spectral tunability, nonlinear response and proved to have a great potential in fiber laser technology. Organic materials are bio-compatible, environment-friendly, light-weight and mechanically flexible. The SA thin-films based on FIrpic,  $Znq_2$  and  $Alq_3$  in conjunction with polyvinyl alcohol (PVA) were successfully fabricated, in a simple, straight and low-cost process. A small piece of fabricated SA thin-film was inserted between two ferrules to form SA device. A set of measurements were taken to characterize the fabricated SAs, such as Fourier transform infrared (FTIR) spectrometer, optical absorption, linear and non-linear transmission. Different kinds of Q-switched fiber lasers with very high stability were successfully demonstrated using these materials. At first, FIrpic was used to develop a Q-switched laser with single- and dual-wavelength operation. Then,  $Znq_2$  was used to demonstrate Q-switching pulsing with fixed and tunable wavelength operation. The produced pulse width was decreased from  $6.6 \mu s$  to  $2.8 \mu s$ , while the repetition rate was increased from 45 kHz to 85 kHz. Finally,  $Alq_3$  was used as a Q-switcher to produce pulses at  $1.5 \mu m$  and  $1.0$

$\mu\text{m}$  regions. At  $1.5 \mu\text{m}$  region, the pulse width and repetition rate were tunable within  $1.2 \mu\text{s}$  to  $6.65 \mu\text{s}$  and  $31.65 \text{ kHz}$  to  $144.5 \text{ kHz}$ , respectively. At  $1 \mu\text{m}$  region,  $\text{Alq}_3$  produced high pulse energy and high peak power of  $0.8 \mu\text{J}$  and  $237.62 \text{ mW}$ , respectively, in two different setups. Stable ultrafast soliton laser operations were also achieved by using the developed SAs. At first, FIrpic was used to demonstrate a mode-locked laser with pulse width and repetition rate of  $120 \text{ ns}$  and  $3.43 \text{ MHz}$ , respectively. Similarly,  $\text{Znq}_2$  was also successful as a mode-locker, where it was used to produce a laser with a pulse width of  $1.46 \text{ ps}$  and a repetition rate of  $3.5 \text{ MHz}$ . Finally, a pulse width of  $820 \text{ fs}$  and a repetition rate of  $4.9 \text{ MHz}$  was achieved by using  $\text{Alq}_3$ .  $\text{Alq}_3$  produced mode-locking operation with single and multi-wavelength operation at  $1.0 \mu\text{m}$  with pulse width and repetition rate of  $5.96 \text{ ps}$  and  $6.84 \text{ MHz}$ , respectively. In conclusion, this work has successfully drawn attention to the potentials that organic materials have in fiber laser technology especially for Q-switching and mode-locking applications.

**Keywords:** Organic materials, Q-switched fiber laser, mode-locked fiber laser

# **BAHAN-BAHAN ORGANIK SEBAGAI PENYERAP BOLEH TEPU UNTUK PENJANAAN DENYUT PADA TETINGKAP TELEKOMUNIKASI**

## **ABSTRAK**

Disertasi ini mendemonstrasikan beberapa jenis laser gentian berasaskan penghasilan baru baham organic sebaga penyerap boleh tepu (SAs). Kami menggunakan SAs ini di dalam semua kaviti cincin gentian asas laser gentian ion erbium yang didopkan (EDFL) untuk menghasilkan denyut suis-q dan selakan –mod di tingkap telekomunikasi, pada ~ 1560 nm. Tiga bahan organic yang berlainan telah digunakan dalam disertasi ini; Bis[2-(4,6-difluorophenyl)pyridinato-C<sup>2</sup>,N](picolinato)iridium(III) (FIrpic), bis(8-hydroxyquinoline)zinc (Znq<sub>2</sub>) and Tris(8-hydroxyquinoline)aluminum (Alq<sub>3</sub>). Pencarian bahan baru masih lagi satu ciri-ciri khusus kepada bidang tak linear optik, pembangunan pesat SAs telah menyediakan banyak peluang baru untuk optik tak linear. Melalui kerja ini, bahan organik menunjukkan prestasi yang sangat bagus dalam terma tempoh denyut, denyut tenaga, ambang kerosakan, kestabilan, spektrum boleh tala, tindakbalas tak linear dan terbukti mempunyai potensi bagus dalam teknologi laser gentian. Bahan-bahan organik adalah serasi-bio, endah alam sekitar, ringan dan boleh suai mekanikal. Filem SA berasaskan pada bahan FIrpic, Znq<sub>2</sub> and Alq<sub>3</sub> berhubung dengan polivinil alcohol (PVA) telah berjaya difabrikkan, ringkas, lurus dan proses kos rendah. Satu bahagian kecil SA-filem yang telah difabrikkan telah dimasukkan diantara dua ferrule dalam turutan menghasilkan peranti SA. Satu set pengukuran telah diambil untuk mencirikan turutan SAs yang telah difabrikkan, seperti Transformasi Fourier Inframerah Spektroskopi (FTIR) Spektrometer, penyerapan optic, tranmisi linear dan tak linear. Berlainan jenis laser gentian suis-q dengan kestabilan yang tinggi telah Berjaya Berjaya didemostratkan menggunakan bahan-bahan ini. Mula-mula, FIrpic telah digunakan untuk menghasilkan laser suis-q dengan operasi jarak gelombang tunggal dan dual. Kemudian, Znq<sub>2</sub> telah digunakan untuk mendemost operasi denyut suis-q dengan tetap dan operasi jarak gelombang bola tala. Penghasilan denyut lebar

telah menurun dari 6.6  $\mu\text{s}$  ke 2.8  $\mu\text{s}$ , manakala kadar ulangan telah menaik dari 45 kHz ke 85 kHz. Akhir sekali, Alq<sub>3</sub> telah digunakan sebagai suis-q untuk menghasilkan denyut-denyut pada bahagian 1.5  $\mu\text{m}$  dan 1.0  $\mu\text{m}$ . Pada bahagian 1.5  $\mu\text{m}$ , masing-masing denyut lebar dan kadar ulangan telah boleh tala diantara 1.2 ke 6.65  $\mu\text{s}$  dan 31.65 sehingga 144.5 kHz. Pada bahagian 1  $\mu\text{m}$ , Alq<sub>3</sub> telah menghasilkan tenaga tinggi dan kuasa puncak yang tinggi iaitu 0.8  $\mu\text{J}$  dan 237.62 mW dalam dua tatacara yang berbeza. Operasi laser *Ultrafast Soliton* yang stabil juga tercapai dengan menggunakan pembangunan SAs. Pada mulanya, FIRpic telah digunakan untuk mendemonstrat laser selakan-mod dengan denyut lebar dan kadar ulangan 120 ns dan 3.43 MHz. Akhirnya, denyut lebar 820 fs dan kadar ulangan of 4.9 MHz telah tercapai dengan menggunakan Alq<sub>3</sub>. Alq<sub>3</sub> menghasilkan operasi selakan-mod tunggal dan pelbagai jarak gelombang pada operasi 1.0  $\mu\text{m}$  dengan masing-masing denyut lebar dan kadar ulangan 5.96 ps dan 6.84 MHz. Sebagai kesimpulan, kerja ini berjaya menarik perhatian kepada potensi bahan-bahan organik yang terdapat di dalam teknologi gentian laser terutama untuk aplikasi suis-q dan selakan-mod

**Kata kunci:** Bahan organik, laser gentian bersuis-Q, laser gentian selakan mod

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## LIST OF SYMBOLS AND ABBREVIATIONS

2D	: Two-dimensional
Alq <sub>3</sub>	: Tris(8-hydroxyquinoline)aluminum
BP	: Black Phosphorus
c	: Speed of light
CNTs	: Carbon Nanotubes
CVD	: chemical vapour deposition
CW	: Continues Wave
dB	: Decibel
DI	: Deionized Water
DWQS	: Dual-Wavelength Q-Switch
EDF	: Erbium-Doped Fiber
EDFA	: Erbium-Doped Fiber Amplifier
EDFL	: Erbium-Doped Fiber Laser
E <sub>g</sub>	: Optical Band Gap
FESEM	: Field Emission Scanning Microscopy
FIrpic	: Bis[2-(4,6-difluorophenyl) pyridinato-C <sup>2</sup> ,N] (picolinato)iridium(III)
FTIR	: Fourier-Transform Infrared Spectroscopy
FWHM	: Full-Width at Half Maximum
GVD	: Group Velocity Dispersion
HNLF	: Highly Nonlinear Fiber
<i>hν</i>	: Photon Energy
<i>I</i>	: Light intensity
ISO	: Optical Isolator

LASER	: Light Amplification by Stimulated Emission of Radiation
LD	: Laser Diode
LPE	: Liquid Phase Exfoliation
MWCNTs	: Multi-Walled Carbon Nanotubes
$n$	: Refractive index
NA	: Numerical Aperture
NLPs	: Noise-Like Pulses
$nm$	: Nanometer
NOLM	: Nonlinear Optical Loop Mirror
NPR	: Nonlinear Polarization Rotation
OC	: Optical Coupler
OSA	: Optical Spectrum Analyzer
OSC	: Oscilloscope
OTDM	: Optical Time Division Multiplexing
PC	: Polarization Controller
PCF	: Photonic Crystal Fiber
ps	: Picosecond
PVA	: Polyvinyl Alcohol
PZT	: Piezoelectric Transducer
RF	: Radio Frequency
RFSA	: RF Spectrum Analyzer
SA	: Saturable Absorber
SC	: Supercontinuum
$\text{Sech}^2$	: Secant hyperbolic
SEM	Scanning Electron Microscope
SESAMs	: Semiconductor Saturable Absorption Mirrors

SMF	: Single Mode Fiber
SNR	: Signal to Noise Ratio
SPM	: Self-Phase Modulation
SWCNT	: Single-Walled Carbon Nanotubes
TBF	: Tunable Bandpass Filter
TBP	: Time-Bandwidth Product
TI	: Topological Insulator
TMD	: Transition Metal Dichalcogenide
WDM	: Wavelength Division Multiplexing
XPM	: Cross-Phase Modulation
YDF	: Ytterbium-Doped Fiber
YDFL	: Ytterbium-Doped Fiber Laser
Znq <sub>2</sub>	: bis(8-hydroxyquinoline)zinc

## CHAPTER 1: INTRODUCTION

### 1.1 Background and Motivation

Pulsed fiber lasers are extremely important in many fields in science and engineering technology. Fiber lasers have extensive applications in many areas such as telecommunications (O'Mahony, Simeonidou, Hunter, & Tzanakaki, 2001), signal processing (Ghelfi et al., 2014; Khilo et al., 2012), optical frequency measurements (Jones et al., 2000; Udem, Holzwarth, & Hänsch, 2002), ranging metrology (Coddington, Swann, Nenadovic, & Newbury, 2009; Joohyung Lee, Kim, Lee, Lee, & Kim, 2010), high-resolution atomic clocks (Bloom et al., 2014; Nemitz et al., 2016), and astronomy (C.-H. Li et al., 2008; Steinmetz et al., 2008). In comparison with other types of lasers, fiber lasers are compact, reliable, highly efficient, robust, and always deliver high-quality laser beams (Fermann & Hartl, 2013; X. Liu, Cui, Han, Yao, & Sun, 2015; Nishizawa, 2014; C. Zhao et al., 2012). Additionally, they have the advantages of alignment-free, small size, simple structure, high environmental stability, and low cost (Cai, Chen, Chen, & Hou, 2018; Y. Chen, G. Jiang, S. Chen, Z. Guo, X. Yu, C. Zhao, H. Zhang, Q. Bao, S. Wen, & D. Tang, 2015; Sheng et al., 2013; J. Xu, Liu, Wu, Yang, & Wang, 2012).

Fiber lasers have been developed rapidly over the past decade. It is worth noting that fiber lasers have been revolutionized by the advances in fiber optics. Fiber optics provides an appropriate platform and has a significant impact on fiber lasers developments. A fiber laser uses rare-earth doped fiber as a gain medium. Rare-earth ions such as erbium (Er), ytterbium (Yb), holmium (Ho), thulium (Tm) and neodymium (Nd) are doped into the fiber to provide a gain in various wavelength regions. Specifically, erbium-doped fiber laser (EDFL), which is operating at 1.5  $\mu\text{m}$  region, is considered as one of the most widely developed fiber lasers and have received huge interest from the researchers. They have many practical advantages such as compact, efficient heat dissipation, inexpensive and

robust (Chong, 2008). Additionally, EDFL operates at wavelength region around 1550 nm, which falls in a low loss window of standard single-mode fiber (SMF).

The pulsed fiber laser can be realized by either active or passive techniques. The active technique achieved by inserting an optical modulator into the laser cavity (Alvarez-Chavez et al., 2000; Kee, Lee, & Newson, 1998). On the other hand, the passive technique does not require an external device, instead, it is obtained through incorporating a saturable absorber (SA) inside the laser cavity. The passive techniques are, in general, cost-effective and more useful than active approaches which require additional switching electronics (Svelto & Hanna, 2013). Additionally, passive techniques are more compact, simple, and flexible to be implemented compared to the active method (Shi-Xiang, Wen-Xue, Qiang, Hui, & He-Ping, 2008).

To date, several types of passive SAs have been intensively investigated for generating Q-switched and mode-locked fiber lasers, including semiconductor saturable absorber mirrors (SESAMs) (J. Y. Huang et al., 2008; Ursula Keller, 2003; Lecourt, Martel, Guezo, Labbe, & Loualiche, 2006; J. Liu, J. Xu, & P. Wang, 2012; Z.-C. Luo et al., 2015; Nodop et al., 2007; Spühler et al., 2001), carbon nanotubes (CNT), (Ahmed et al., 2015; X. Xu et al., 2014; U. Zhang et al., 2013; Zhou, Wei, Dong, & Liu, 2010), and two-dimensional (2D) nanomaterials such as graphene and transition-metal dichalcogenides (TMDs) (Cao, Wang, Luo, Luo, & Xu, 2011; Jiang Liu, Jia Xu, & Pu Wang, 2012; Popa et al., 2011).

SESAMs, however, demand an expensive and complex fabrication. Additionally, they have limited applications as they are not compatible with optical fiber. The CNTs, on the other hand, have the advantages of fiber compatibility, low cost and ease of fabrication over the traditional SAs (J. Y. Huang et al., 2008; Lecourt et al., 2006; D. Lin et al., 2010; Philippov, Kir'yanov, & Unger, 2004). CNTs also have a wide operating bandwidth, low saturation intensity and sub-picosecond recovery time. CNTs, however,

require a complex band gap control which limits their operation at certain wavelengths (Q. L. Bao et al., 2009). The dominance of CNTs was reduced with the introduction of graphene, as it has a cost-efficient and simple fabrication and manufacturing processes and ultrafast recovery time. Consequently, graphene became the dominant choice for SAs (J. Liu, Wu, Yang, & Wang, 2011; L. Zhang et al., 2012). In comparison to CNT, graphene has a higher damage threshold and a super broad bandwidth. But, graphene, however, has the drawbacks of low modulation depth as well as a small optical absorption range which posed a limitation in many applications (Luo et al., 2010).

Recently, black phosphorous (BP) have also attracted much attention as it has the potential of a promising SA for both Q-switched and mode-locked fiber laser applications (Y. Chen, G. Jiang, S. Chen, Z. Guo, X. Yu, C. Zhao, H. Zhang, Q. Bao, S. Wen, D. Tang, et al., 2015; Jiang, Yin, Zheng, Yu, & Cheng, 2015; L. Kong et al., 2016). BP has a highly useful property of a controllable band gap through the control of its thickness. However, BP oxidation could be accelerated in the air when it illuminated to high power illuminates, which could lead to reducing the damage threshold (Feng et al., 2016). Moreover, BP is a hydrophilic material (J. Li et al., 2016) which is easily damaged when exposed to water and oxygen.

Lately, TMDs have been extensively studied as SAs for both Q-switching and mode-locking operations (Heping Li et al., 2015; Zhengqian Luo et al., 2014; S. Wang et al., 2014; H Zhang et al., 2014; Meng Zhang et al., 2015). For instance, molybdenum disulfide ( $\text{MoS}_2$ ) has attracted much attention as it has a unique absorption property and also a thickness-dependent band gap (Du et al., 2014; Hao Liu et al., 2014). Yet, TMDs have a low impurity and uniformity and low damage threshold which restricts the long-term stability of the pulsed fiber laser.

Nevertheless, there have been increasing concerns about the effect of long-term exposure of the nanomaterials on human health (Nel, Xia, Mädler, & Li, 2006). On the

other hand, many attempts in the last few years have been carried out to find new materials to be used as a high-performance SAs. In that regards, organics material might be a promising candidate as new SA as they are biocompatible (Šafaříková et al., 2018) and have a broad spectral tunability. Additionally, organic material have a large and ultrafast nonlinear response and they can be solution-processed at low cost. Moreover, organic material would be a highly desirable alternative for developing non-hazardous and environmentally-friendly photonic devices which could find increasing applications in biomedical lasers.

In comparison with traditional inorganic materials, organic materials have the advantages of simple and low-cost manufacturing process and easy control of physical properties (Nel et al., 2006). Despite numerous works reported on the use of organic materials in the linear regimes, the applications of organic materials in nonlinear optics, especially in the fiber lasers pulse generation, have yet to be fully explored.

Bis[2-(4,6-difluorophenyl)pyridinato-C<sup>2</sup>,N](picolinato)iridium(III) (FIrpic), bis(8-hydroxyquinoline)zinc (Znq<sub>2</sub>) and Tris(8-hydroxyquinoline)aluminum (Alq<sub>3</sub>) might be a good candidate to be used as an efficient SA. They are widely utilized in the development of organic light-emitting diodes (Adachi, Kwong, et al., 2001; Jaewon Lee et al., 2008). Additionally, they have a straightforward synthesis process, excellent stability and favourable electrochemical properties. Furthermore, they have simple, low-cost and straightforward fabrication process, which would be very interesting for practical applications. They might be a strong potential candidate for developing pulsed fiber laser with a very interesting performance.

This research tries to draw attention to the potentials that organic materials have in fiber laser technology. The proposed organic materials were used in a simple all-compact EDFL cavity and produced a very interesting result. To the best of our knowledge, this

research will be the first to demonstrate the usage of these proposed materials as SAs in fiber laser cavity.

## **1.2 Objectives of Research**

This thesis has the goal of drawing attention to the potential that organic-based SAs have in pulse fiber laser technology. This thesis shows that these materials can be fabricated at a low-cost, easy and straight-forward process. Furthermore, this thesis shows that different types of Q-switched and mode-locked fiber lasers have been demonstrated based on organic materials, which further indicates their potentials. Several objectives have been outlined to guide the research direction toward the goal:

1. To fabricate and characterize newly developed organic materials, Flrpic, Znq<sub>2</sub> and Alq<sub>3</sub>, to be used as a high-performance SAs.
2. To design an optimized fiber laser cavity to generate Q-switch and mode-locked pulses.
3. To develop single, dual-wavelength and tunable wavelength Q-switched fiber laser with high stability and wide tunability by using the fabricated organic SAs.
4. To generate a soliton mode-locked fiber laser with nanosecond, picoseconds and femtoseconds pulse width by using the developed organic SAs.

## **1.3 Contributions**

The contributions of this research work are summarized as follow:

1. A simple and compact Q-switched and mode-locked EDFL is successfully developed using organic materials-based SA. The newly thin-film SAs are prepared by drop-casting technique. A small piece of the prepared thin-film SAs was sandwiched between two ferrules and integrate into the EDFL ring cavity.

2. Demonstration of single, dual and tunable wavelength Q-switched fiber lasers by using the developed SAs.
3. Demonstration of various mode-locked EDFLs using the developed SAs. Soliton mode-locked EDFL lasers with nanosecond, picosecond and femtosecond were developed by using Flrpic, Znq<sub>2</sub> and Alq<sub>3</sub>, respectively. Additionally, dark pulse-mode-locked fiber laser was demonstrated by managing the polarization of the cavity. Furthermore, supercontinuum wavelength with picosecond pulse width was obtained by using 100 m PCF fiber.
4. To the best of our knowledge, this is the first time that these organic materials were used in fiber laser technology.

#### **1.4 Thesis Overview**

Though out this thesis, a comprehensive study of Q-switched and mode-locked pulse generation by using organic materials is presented. This thesis consists of six chapters. This chapter presents a background, motivation and objectives of this study.

Chapter 2 presents the literature review of this research work. Theoretical aspects for pulse propagation in optical fiber are addressed, along with a review on the recently used SAs. The important parameters of the pulsing operation of the fiber laser are also described.

Chapter 3 illustrates the fabrication process and characterization of Flrpic, Znq<sub>2</sub> and Alq<sub>3</sub> as SAs. All the SAs are fabricated in a simple, low cost, straight-forward process. Polyvinyl alcohol (PVA) was used to form the thin-film SAs. FTIR, absorption spectrum, SEM image, linear and non-linear absorption were used to characterize the SAs thin-films.

Chapter 4 reports various passively Q-switched EDFLs using the developed SAs. Firstly, Flrpic was used to produce a Q-switched laser with both single and dual-

wavelength operations. Secondly,  $\text{Znq}_2$  was used to produce single and tunable-wavelength operation. Thirdly,  $\text{Alq}_3$  was used to generate Q-switched pulsing at 1560 nm. Finally,  $\text{Alq}_3$  was used to generate a Q-switched laser at 1067 nm to investigate the potential of organic materials at 1.0  $\mu\text{m}$  region.

Chapter 5 demonstrates several types of mode-locked fiber lasers. Flrpic and  $\text{Znq}_2$  were used to develop nanosecond and picosecond soliton mode-locking operation, respectively.  $\text{Znq}_2$  was also used to produce dark pulses and supercontinuum mode-locking operations. A mode-locked with a femtosecond pulse width was demonstrated by using  $\text{Alq}_3$ .  $\text{Alq}_3$  was also used to produce mode-locking operation at 1.0  $\mu\text{m}$  region.

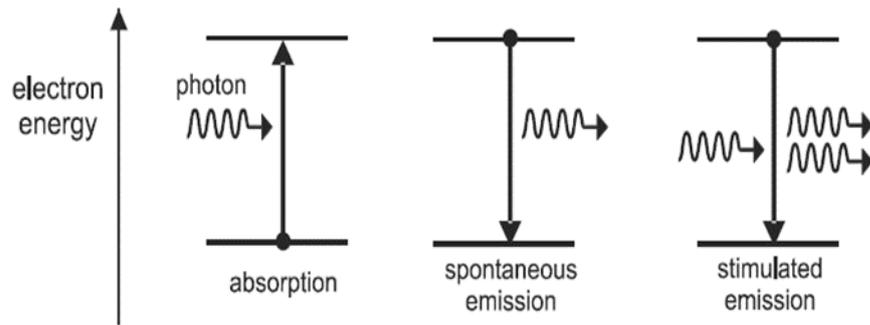
Finally, chapter 6 concludes the thesis and presents a summary of the research findings. The final chapter also provided a recommendation for future works.

## CHAPTER 2: LITERATURE REVIEW

### 2.1 Overview of Fiber Lasers

When the lasers were firstly developed in the 1960s, it was said that the laser was a solution in search of a problem. It was not clear how it would be used. The laser in the coming decades become an enabling technology with applications as diverse as optical communications, range finding, laser reflectometry, remote sensing, laser surgery, reading-writing data on CDs and DVDs, bar-code scanners and precision cutting device manufacturing (Quimby, 2006). It is not an exaggeration to state that the laser had made the modern technological world the way it is today.

The laser consists of three basic elements: a pumping power to supply energy, a gain medium to amplify the light and a feedback mechanism. The feedback will form an optical cavity, in which the light will be circulated back and forth through the gain medium. The amplification is achieved by stimulated emission. Albert Einstein has proposed this process in 1917. To understand stimulated emission, we need to consider the ways that light interacts with an atom, see Figure 2.1 below. There are three processes involved; absorption, spontaneous emission and stimulated emission. In absorption, an atom is raised to a higher energy level (excited state) from the lowest energy level (ground state) by absorbing an incident photon. That atom will fall back to the ground state and emits a photon, this is called spontaneous emission. The stimulated emission occurs when an atom that is already in the excited state is stimulated to drop to ground state and emit a photon (which is identical to the photon that was originally absorbed by the atom in the first place). Amplification is the process of duplicating the photons. As the number of photons increases, the intensity of light increases.



**Figure 2.1: Amplification process in the laser's gain medium (Quimby, 2006)**

A fiber laser is a type of laser in which the gain medium is an optical fiber doped with rare-earth elements such as erbium, ytterbium, and thulium. The fiber lasers technology was born in 1961 when Snitzer demonstrated a laser oscillation in glass (Snitzer, 1961a) and then in fiber laser (Snitzer, 1961b). In 1964, fiber laser oscillation in a gain fiber was demonstrated by Koester and Snitzer (Koester & Snitzer, 1964). In the 1980s and 1990s, fiber lasers have received a tremendous research interest due to the appearance of reliable and reasonably powerful diode-pumped laser technology. This was further motivated by the application of the erbium-doped fiber amplifier (EDFA) in optical telecommunication. The telecommunication age was and still contributing to the development of fiber laser technology. On the other hand, one can observe from reviewing the millstone of the fiber laser technology development, that fiber laser is a result of joint success in the field of optical fiber technology and solid-state lasers. Fiber lasers have adopted solutions from both fields. Today, fiber lasers complement gas and bulk solid-state high-power lasers (Ter-Mikirtychev, 2014).

## 2.2 Light Amplification

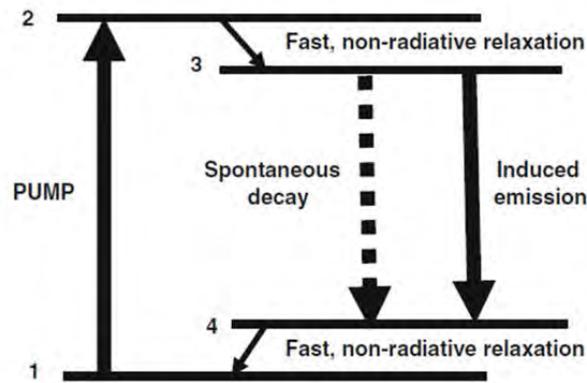
To understand the principle of fiber laser, it is important to understand the basics of optical amplification. At thermal equilibrium, the atomic system is naturally relaxed, as the lower energy level is more populated than the higher energy level. High energy level might

increase its population at higher temperatures, but it never becomes higher than the lower level. In order to achieve an optical gain through stimulated emission, equilibrium status should be avoided and the population at high energy level should be higher than the lower level, this is known as population inversion. The population inversion can be realized by pumping method. The laser system can be classified into three-level and four-level systems depending on the active medium.

Figure 2.2 illustrates the four-energy-level system. As the pump energy is launched into the gain medium, atoms will be excited from level 1 (ground level) to level 2. Then the atoms will decay spontaneously to level 4 after fast relaxation to energy level 3 (non-radiative relaxation and takes only sub-nanosecond to picosecond). Then the atoms will have non-radiative relaxation from level 4 and returning to level 1. The amplification occurs during the slow spontaneous decay from level 3 to level 4 (with durations from milliseconds to nanoseconds). Atoms will be accumulated at level 3, while level 4 will remain unpopulated, due to the fast relaxation from Level 4 to level 3.

Amplification can be made in fiber laser by achieving population inversion in the gain medium. The amplification of lasing increases with the length of the gain medium until the saturation level is reached. The amplification in fiber lasers can be very high and it is only limited by the available light intensity or the laser oscillations inside the fiber laser cavity (in case the optical feedback becomes equal to the amplification). The optical feedback is build-up by either a ring or Fabry Perot resonator. The high gain of the gain medium enables the fiber laser to operate with a low optical feedback power, thus simplifies the developments of fiber lasers.

The gain medium, as mentioned above, are rare earth elements such as ytterbium, neodymium, erbium or thulium and are ionized in silica in order to form a trivalent state. In the next subsection, we will focus on erbium-doped fiber (EDF) as it is widely used as a gain medium in fiber laser that generates laser in 1550 nm region, which it is interest for communication applications.

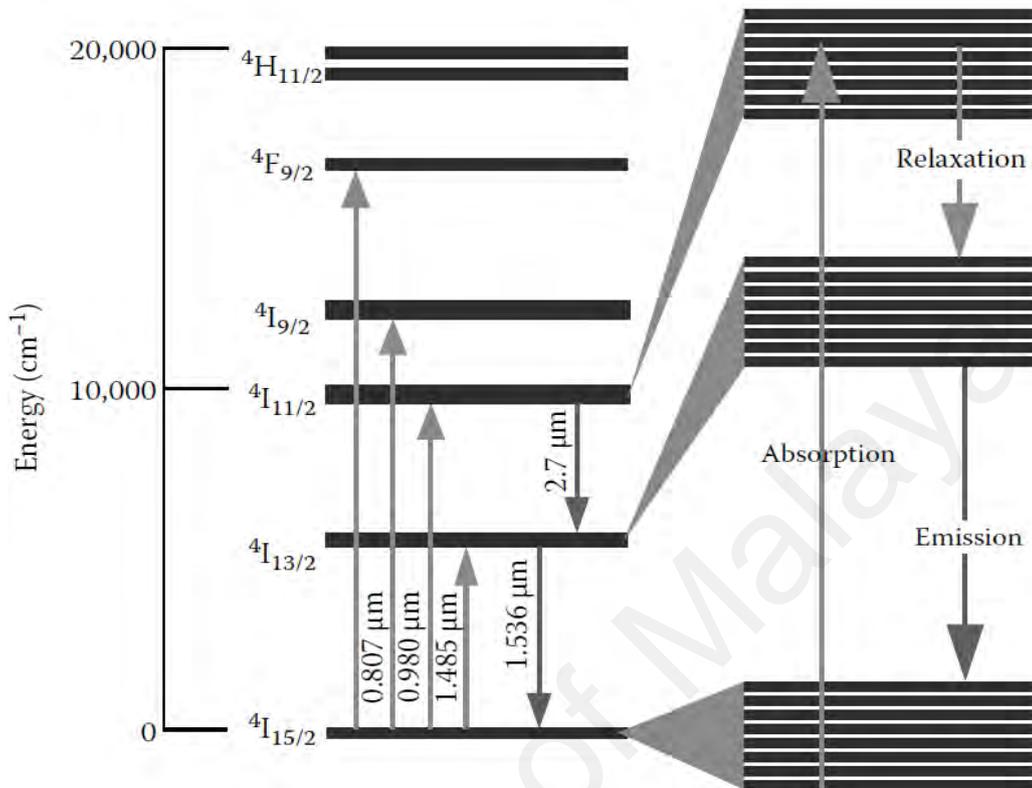


**Figure 2.2: Four-energy-level scheme of laser operation**

### 2.3 Spectroscopic Properties of Erbium-Doped Fibers

EDFs have attracted much attention in the 1980s and they are probably the most studied in comparison with other rare earth materials, due to their importance in optical telecommunication. An enormous number of successful commercial development and research papers are triggered by the demand for optical amplifiers and fiber lasers that operating at 1.5  $\mu\text{m}$  and for telecommunication applications (Ter-Mikirtychev, 2014). Figure 2.3 illustrates the energy level of the erbium ion ( $\text{Er}^{3+}$ ). The transition in EDF from  $^4\text{I}_{13/2}$  to  $^4\text{I}_{15/2}$  is considered as the main transition since it provides a gain in the C-band region, as  $^4\text{I}_{13/2}$  is the only metastable state at room temperature (Ngo, 2016). The EDF has the maximum emission and absorption peak at a wavelength around 1530 nm, therefore, the operating wavelength must be chosen in the band that the absorption cross-section is much smaller than the emission cross-section, which is around 1.55- $\mu\text{m}$ . The EDF can be pumped with a laser diode at a wavelength of 810, 980, and 1480 nm. The pumping operation at 980 and 1480 nm are more preferred as the pumping at 810 nm is inefficient. This is attributed to excited-state absorption (ESA), which will cause an undesirable waste of pumping photons. Pumping at 980 nm is one of the most commonly used for EDFA as it has a high gain efficiency and high signal-to-noise ratio (SNR) (Digonnet, 2001). Additionally, it gives better quantum conversion efficiencies and better noise figures (NFs) for power amplifiers. On the

other hand, EDF is also widely used for ultrashort pulse generation as the fiber dispersion at 1.5  $\mu\text{m}$  is irregular and the EDF has a broad spectrum range.



**Figure 2.3: Energy level diagram of Erbium (Ngo, 2016)**

## 2.4 Pulsed Fiber Lasers

Fiber lasers will typically produce continuous wave (CW) operation, in which the light intensity is constant in time. CW operation is useful for application that requires precise frequency control, such as optical telecommunications and optical spectroscopy (Quimby, 2006). However, there are many practical applications in which pulsed lasers are more desirable such as laser ranging, laser cutting and laser surgery.

Fiber lasers can generate optical pulses by the techniques of Q-switching or mode-locking. Pulsed fiber lasers can be generated by active and passive techniques (Ursula Keller, 2003). The active technique utilizes a modulator to produce the pulses, while a passive technique is obtained through inserting a saturable absorber (SA) device inside a

laser cavity. The first SA was demonstrated in 1966 to generate pulses in Nd: glass laser (Stetser & DeMaria, 1966). Due to the simplicity of passive Q-switched and mode-locking techniques, they have been widely explored in the last few years (Grelu & Akhmediev, 2012). Passive Q-switching laser operation depends on the SA to modulate the Quality factor Q inside the laser cavity and produce pulses accordingly. While the passive mode-locking technique, on the other hand, uses SA in a laser cavity to act as a mode-locker and produce extremely short pulses, i.e. fixed-phase relationship is induced between the longitudinal modes of the laser's resonant cavity. In the remaining of section 2.4, we will address the fundamentals behind pulses generation.

### 2.4.1 Q-switching

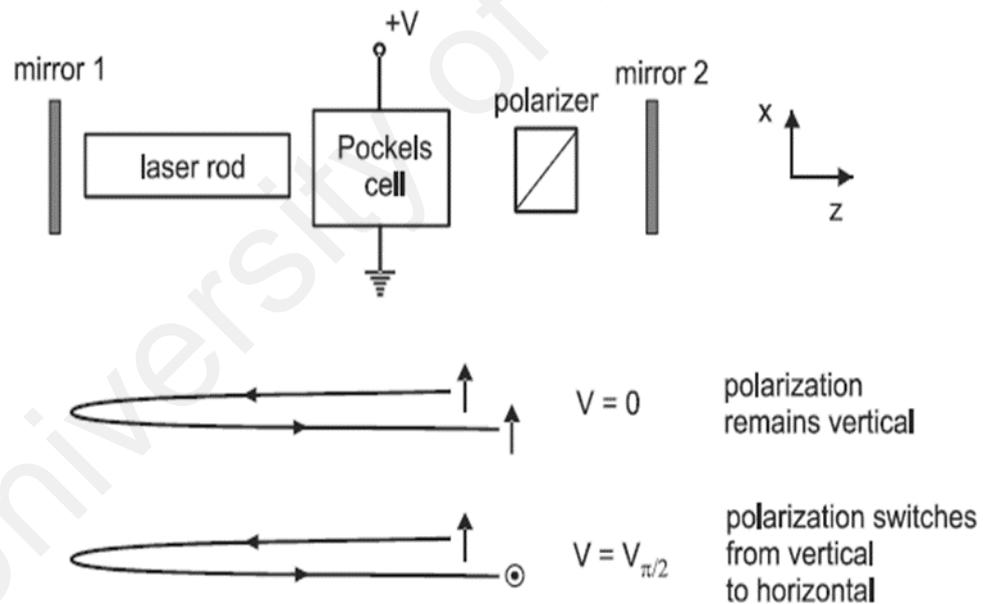
The Q-switching is a well-known technique for generating pulses based on modulating the Q-factor of the laser cavity. The Q represents the quality of the laser resonator in terms of cavity losses in such a way that high Q indicates low intra-cavity loss and vice versa. The Q-switching phrase refers to the switching of the laser inter cavity loss from low to a high value to produce a short pulse width. The Q-switching produces pulse duration typically from microseconds to nanoseconds (N. N. Razak, Latiff, Zakaria, & Harun, 2017). There are many practical methods for producing Q-switched laser, each uses a different technique to modulate the Q of the cavity.

The use of a rotating mirror is the simplest method for producing Q-switching operation. One mirror in the laser is fixed, while the other mirror rotates at high speed on the vertical axis, see Figure 2.4. When the two mirrors are in parallel (with some tolerance of  $\Delta\theta$ ), then the Q of the cavity will be high. The cavity Q will switch from high to low in a time of  $\Delta\theta/\omega$ , where  $\omega$  is the angular rotating speed of the mirror. This method was used in early solid-state lasers as it simple to be implemented, and recently in fiber lasers.



**Figure 2.4: Pulsed laser based on rotating mirror**

Another way for producing Q-switching operation is by inserting an electro-optic shutter inside the cavity, see Figure 2.5. The shutter is made by placing a polarizer (which is oriented to pass only one polarization of light) and Pockels cell both in the path of the beam inside the cavity. When a high voltage is applied to a Pockels cell, the polarization of the light will be rotated, and the light will be blocked by the polarizer.

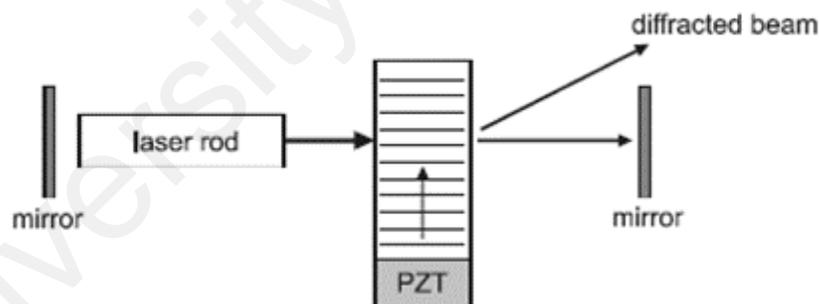


**Figure 2.5: Pulsed laser based on the electro-optic shutter (Quimby, 2006)**

When no voltage is applied, the light will efficiently be transmitted through the polarizer and Pockels cell. When the voltage is applied and removed rapidly, the Q factor of the cavity will be switched accordingly and, hence producing pulses. The switching

time in this technique can be about 20 ns in which a voltage of few kV is required by the Pockels cell. This technique is in common use but requires safety measures due to the high voltages involved.

The acousto-optic shutter can also be used to modulate loss inside a laser cavity. In this method, a transparent crystal is inserted the path of the light in the cavity. Then piezoelectric transducer (PZT) is used to generate acoustic waves with high intensity in the transparent crystal. Correspondingly, these crystals will have periodic variation in the refractive index, which will form a volume-phase grating. This will diffract the light and decreases the Q factor of the cavity and prevent lasing. If the acoustic waves are not applied to crystals, the Q factor is increased and the lasing is enabled, see Figure 2.6. These types of Q-switched lasers have the advantages very low optical insertion loss, relatively simple circuitry and ease of use for kHz's Q-switched repetition rate. However, they have slow switching times and low hold of ratio.



**Figure 2.6: Pulsed laser by using Acousto-optic shutter, (Quimby, 2006)**

In the active Q-switching, the duration and time of the change in Q are under active control, i.e. the timing and repetition rate are determined by the user. Alternatively, the laser cavity can also be Q-switched by itself. This method is termed as passive Q-switching. A passive Q-switching operation can be developed by using an SA inside the laser cavity. An SA can be used to modulate the intra-cavity loss and produce pulses. The

process begins as Q-factor is kept at a low level and prevent oscillation (i.e. high losses). That will lead the gain to build up inside the cavity through accumulating the spontaneous emission. When the Q-factor is suddenly switched to a high level, the lasing will start to build up and a pulse is formed. When the gain is depleted completely and pulse peak power is reached, the laser oscillation stops. The Q factor will be low again and the process starts from the beginning, see Figure 2.7. Passive Q-switching laser operation is normally more efficient in term of implementation cost and operation as it requires less control of cavity parameter. Passive Q-switching is generally convenient, simple and does not require and external driving circuitry.



**Figure 2.7: Passive Q-switched pulse formation**

## 2.4.2 Mode-Locking

In mode-locking operation, the oscillated longitudinal modes are in synchronism, i.e. the phase of the different modes are locked and added constructively. Correspondingly, the laser will produce a train of pulses (Siegman, 1986). The frequency separation between modes, group velocity ( $\Delta v_g$ ) and the gain bandwidth are all responsible for determining the numbers of modes that can lase. The process for producing an ultrashort pulse in a mode-locked laser is much more complicated than Q-switched as it involves non-linearities, dispersion, cross-phase modulation (XPM) and self-phase modulation (SPM). The repetition rate ( $f_{rep}$ ) (for

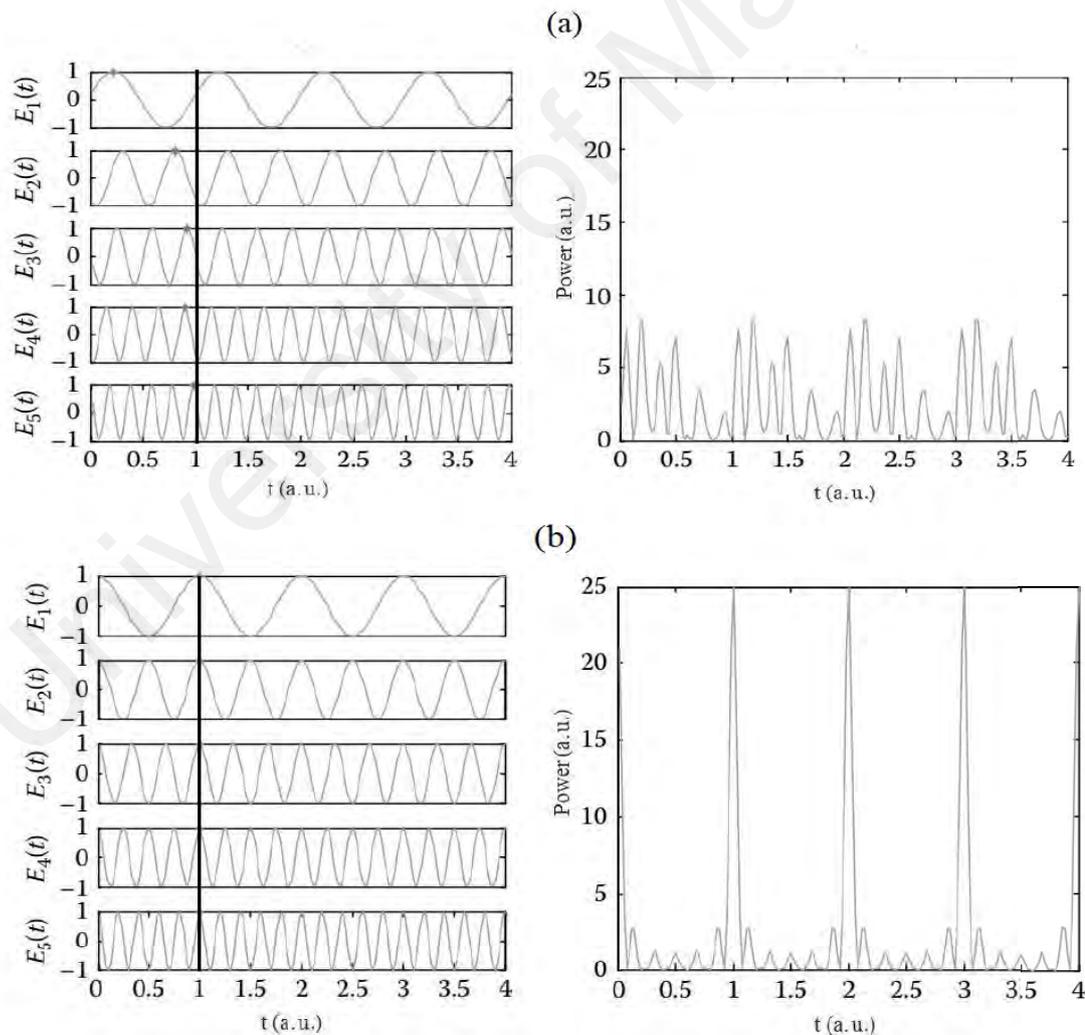
ring cavity) for pulse mode-locked fiber laser is determined by the cavity length, and it is given by the equation.

$$f_{rep} = c / nL \quad (2.1)$$

where  $c$  is the speed of light,  $n$  refractive index and  $L$  is the length of the cavity. The repetition rate is inversely proportional to the round-trip time,  $T_R$  then

$$T_R = nL / c \quad (2.2)$$

Figure 2.8 shows the difference between normal operation, CW operation, and mode-locking operation. Figure 2.8 (a) shows mode at random phases, while in Figure 2.8 (b) the modes are locked and mode-locked pulses are generated.

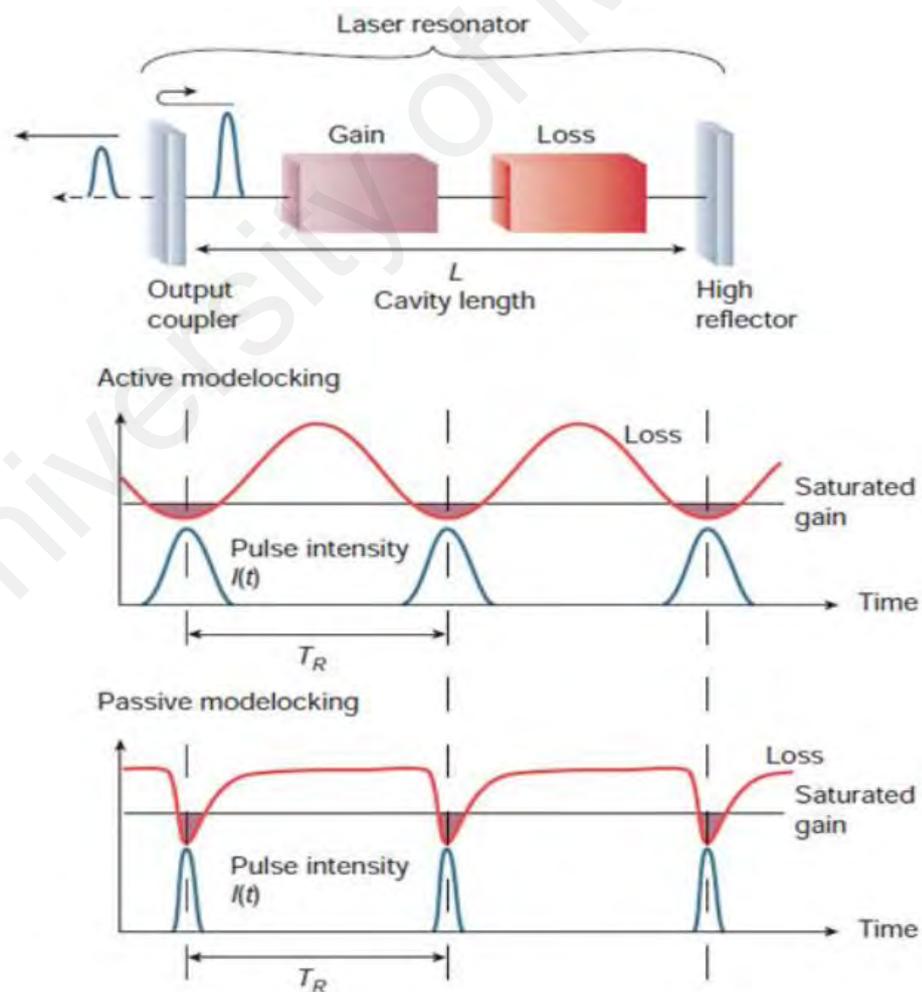


**Figure 2.8: Electric field amplitudes versus time for (a) CW, (b) mode-locking (Ngo, 2016)**

The mode-locked laser produces the pulse width in the range from nanoseconds to femtoseconds, which is depending on the SAs and cavity characteristics. There are two ways to achieve mode-locking: active and passive methods. Active mode-locking operation is like active Q-switching in terms of an external electronic device is used to modulate the signal, see Figure 2.9. Generally, there are two techniques to produce the active mode-locking pulses, that is amplitude modulation (AM) and frequency modulation (FM). AM technique is about using lose cavity to modulate the signal periodically, the pulses are then built up where there is minimum loss inside the laser cavity (Gupta, Onodera, Abedin, & Hyodo, 2002). The FM technique, on the other hand, is achieved through frequency chirping. The pulse is shortened and built up after several round trips due to the chirped light is swept out of the laser cavity (Nakazawa, Kubota, Sahara, & Tamura, 1998). However, active mode-locking has the disadvantages of low stability and low availability of fast external modulators. The passive mode-locking, on the other hand, can generate much shorter pulse width by utilizing SAs (Ursula Keller, 2003). Passive mode-locking is more effective and has more practical importance than active technique. It was demonstrated in 1968 by De Maria et al., in which Nd:glass was used as SA (W. Schmidt & Schäfer, 1968). But the fiber laser did not measure a regular pulse train and it remained a problem for passive techniques for more than two decades. The problem was only solved by utilizing semiconductor saturable absorber mirrors (SESAMs) in 1992 (U Keller et al., 1992).

The SA act as a mode-locker by putting some loss to the intra-cavity. The loss is relatively low for high intensities and high for low intensities pulses. I.e. a short pulse will induce a loss modulation as the high intensity (peak of the pulse) will saturate the SA, while low intensity (leading and trailing edges) would be absorbed by the SA. On the other hand, the gain will increase the intensity of the pulse, while other low-intensity will have their losses more than their gain, and they will die out (Duling III & Duling, 1995;

Ursula Keller, 2004), see Figure 2.9. The SA can produce very short pulses as the loss modulation of SA can be very fast due to the fast recovery time of the SA. Currently, the passive mode-locked laser based on SAs can produce ultrafast pulses in the range between picoseconds to femtoseconds depending on the SA parameters (M. Ahmed et al., 2014; Jung et al., 2014). Soliton mode-locking is one of the techniques that is frequently used to generate sub-picosecond and femtosecond pulse width in a fiber laser. Solitons are of great interest, as they maintain their velocity and shape through time. The soliton operation results from an interplay between the dispersion and nonlinear effects. The pulses obtained typically have a high pulse quality and hardly any chirp with a nice hyperbolic secant ( $\text{sech}^2$ ) shape. In this thesis, the focus will be on developing passive soliton mode-locking fiber lasers.



**Figure 2.9: Schematic laser cavity setup for active and passive mode-locking (Ursula Keller, 2003)**

## 2.5 Saturable Absorbers (SAs)

SAs are nonlinear optical devices which have an intensity-dependent transmission. The SAs are widely utilized as a passive modulator in both Q-switched and mode-locked lasers. The parameters of choosing an SA are damage threshold, wavelength range, saturation intensity, recovery time and modulation depth (H Zhang et al., 2014). The damage threshold is defined as the maximum input light intensity that SA can absorb before it been damaged. The wavelength range is the region of operation that the SA must be synchronized with the gain of the corresponding lasing medium. The saturation intensity is known as the required light intensity to saturate the absorption of the SA. While the speed limit on the switching device (which affect the duration of the pulses) is known as recovery time. The modulation depth is the value for the maximum possible change of absorption the SA can have. Other parameters such as cost of fabrication and environmental stability are also important in choosing a SA.

These mentioned parameters are governed by the atomic arrangement and chemical composition of the SA material. These parameters can be described in the equation of saturable absorption and as shown below (Bao et al., 2011) :

$$a(I) = \frac{a_0}{1 + \frac{I}{I_s}} + a_{ns} \quad (2.3)$$

where  $a_0$  is modulation depth,  $a_{ns}$  is nonsaturable absorption,  $I$  is the light intensity  $I_s$  is the saturation intensity. While the saturable absorption can be expressed as,

$$a_s = N\beta \quad (2.4)$$

where the  $N$  and  $\beta$  represent the concentration of the material and absorption ground cross-section respectively. The saturable absorption indicates that a certain number of photons are absorbed per unit distance from the SA device. In case that the saturable absorption coefficient was large, then photons will be absorbed over a short distance into

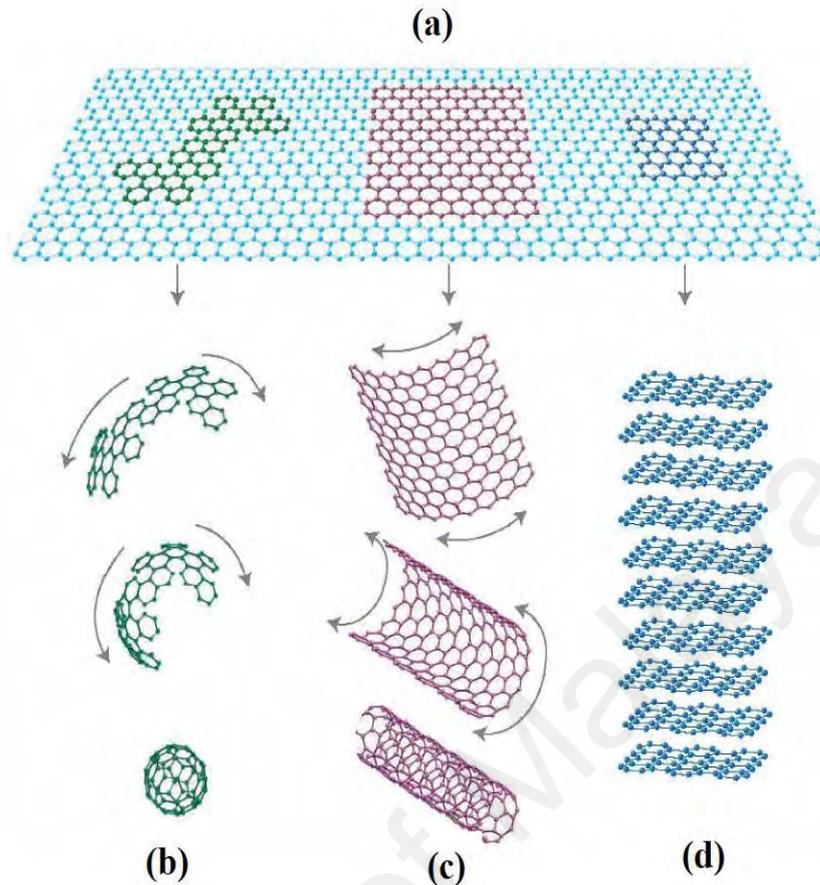
the SA. The non-saturable absorption, on the other hand, is the loss that cannot be saturated by the SA because of the nonsaturable defect in absorption and scattering loss.

## **2.6 Saturable Absorber Materials**

SAs can be divided into two categories: artificial and real SA. The nonlinear polarization rotation effect can be used inside a laser cavity to mimic the behaviour of a real SA by inducing an intensity-dependent transmission. This technique is simple, but the stability of the generated pulses is difficult to be maintained since the operation is strongly dependent on the oscillating light polarization. Real SA uses materials that show an intrinsic nonlinear decrease in absorption as the light intensity increase. To date, several types of SAs have been intensively investigated for developing Q-switched and mode-locked fiber lasers. In this part, we restrict the focus to real SAs.

### **2.6.1 Carbon Nanotubes**

Carbon Nanotubes (CNT) was found by Iijima in 1991 during the arc discharge method (Ando & Iijima, 1993). CNTs can be produced from graphene through rolling the graphene on the cylindrical nanostructure. The atomic structure of graphitic materials of 0D Fullerene, 1D CNT, 2D graphene and 3D graphite is shown in Figure 2.10 (Geim & Novoselov, 2010). Several methods have been proposed to utilize CNT as SA in fiber lasers, for example, optical deposition, free space arrangement and sandwiching the SA between two fiber ferrules (Z Sun, Hasan, & Ferrari, 2012). CNTs can be single-walled nanotubes (SWCNTs) or multi-walled nanotubes (MWCNTs) (that is according to the number of walls that form the CNT). SWCNTs have been considered as a promising SA device due to their intrinsic saturable wide absorption wavelength bandwidth, ultrafast recovery time and absorption properties (Harun et al., 2012). However, SWCNTs suffer from a low damage threshold (M. H. M. Ahmed et al., 2014).



**Figure 2.10: (a) 2D graphene (b) 0D Fullerene, (c) 1D CNT and (d) 3D Graphite (Geim & Novoselov, 2010)**

On the other hand, MWCNTs have attracted much attention due to their advantages such as the fabrication cost of MWCNT material is about 50%–20% of that of SWCNT (L. Zhang et al., 2011). Additionally, the damage threshold of MWCNTs is higher than that of SWCNTs (Ramadurai et al., 2008) as the outer walls can protect the inner walls from damage. However, the MWCNTs has a high saturation intensity which it is not a very attractive feature for mode-locking operation as this would require high irradiation intensity to reach absorption saturation (Lim et al., 2006). Therefore, there are only a few demonstrations for MWCNTs as a SA. Generally, CNTs often demand band gap engineering or charity control which limits its operation at certain wavelengths (Q. Bao et al., 2009). Table 2.1 review the works that have been demonstrated by using CNT as SA.

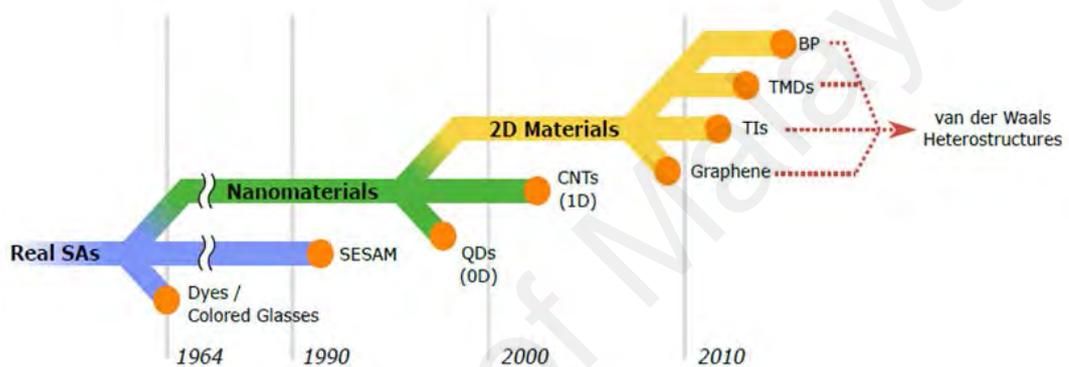
**Table 2.1: CNT as a saturable absorber in fiber laser cavities**

Laser Type	SA	Wave-length (nm)	Pulse width	Repetition rate	Maximum Output power (mW)	SNR (dB)	Reference
Q-switched laser	SWCNT	1533.6	16.9 to 8 $\mu$ s	9.52 to 33.33 kHz	9.5	50	(Ahmed et al., 2015)
		1073	6.45 to 3.11 $\mu$ s	17.1 to 23.6 kHz	14.65		(Al-Masoodi et al., 2014)
	MWCNT	1534.5	11.7 to 8.3 $\mu$ s	38.11 to 48.22 kHz	4.8	27	(Azooz et al., 2015)
		1533.6	9.5 to 4.2 $\mu$ s	6.12 to 33.62 kHz	12.4	40	(Ahmed et al., 2015)
		1055–1063	17 to 8.87 $\mu$ s	10.41 to 29 kHz	1	36	(AHH Al-Masoodi, Ahmed, Arof, & Harun, 2018)
Mode-locked laser	SWCNT	1533.6	1.8 ps	15.3 MHz	4.3	-	(M. Ahmed et al., 2014)
		1564.2	0.77 ps	17.7 MHz	0.91	37.4	(Markom, Sen-Winson, Paul, & Harun, 2017)
	MWCN	1550	1.28 ps	4.54 MHz	-	-	(Cheng, Lin, & Lin, 2013)

### 2.6.2 2D Nanomaterials Saturable Absorber

2D nanomaterials have different optical, thermal, electronic and mechanical properties from its 3D form (Novoselov et al., 2004). 2D material can be used in the field of optoelectronics particularly in photovoltaics, photo-detection, and photoluminescence. Additionally, the 2D materials can be used as SA to generate Q-switched and mode-locked pulse lasers (Huang & Luo, 2014; Zhang et al., 2014). The usage of 2D nanomaterials has

started since the 1960s, as 2D nanomaterials provide greater benefits due to their extraordinary properties (Savage, 2012). The 2D nanomaterials provide enhanced biological functionality, chemical and physical due to their high surface-to-volume ratios, uniform shapes, and surface charge. Over the years, several materials have been extracted from 2D nanomaterials families such as graphene, topological insulators (TIs), TMDs and black phosphorus (BP) (Kambe et al., 2014; Xia, Wang, & Jia, 2014), see Figure 2.11 below. These materials will be reviewed in detail in the following paragraphs.



**Figure 2.11: The evolution of real saturable absorbers (Robert Woodward & Kelleher, 2015)**

### 2.6.2.1 Graphene

Geim and Novoselov have discovered single-layer graphene in 2004 and found that the graphene does not exist in a free-state (Geim & Novoselov, 2010). Single-layer graphene and few-layer graphene can be produced from graphite by using scotch-tape method (Geim & Novoselov, 2010) and also by chemical vapour deposition (CVD) method (Obraztsov, Obraztsova, Tyurnina, & Zolotukhin, 2007). Graphene has become the interest of researchers as it has valuable and unique electronic properties (Geim & Novoselov, 2010). Single-layer graphene has an optical transmittance of about 97.7% which enables the materials to be used in the broadband application (Bonaccorso, Sun, Hasan, & Ferrari, 2010). Graphene has been addressed in several fields, as its nonlinear

optical properties have enabled the material to be utilized in photonic applications (Hasan et al., 2009). Graphene has been widely explored as SA in Q-switched and mode-locked fiber lasers, as it has an ultrafast saturable absorption. The first usage of graphene as SA in mode-locked fiber laser was in 2009 (Zhipei Sun et al., 2010). It should be mentioned that graphene has the drawback of zero band gaps which limited its capability in several applications. Several works had demonstrated graphene as SA for mode-locked and Q-switched fiber laser, as illustrated in Table 2.2.

**Table 2.2: Graphene as an SA for both Q-switch and mode-lock laser**

Laser Type	Wavelength (nm)	Pulse width	Repetition Rate	SNR (dB)	Output power (mW)	References
Q-switched laser	1558.28	9.58 to 6	47.94 to 67.8 kHz	62	-	(Zuikafly, Khalifa, Ahmad, Shafie, & Harun, 2018)
	~1550	2.8 to 0.2 $\mu$ s	31.7 to 236.3 kHz	-	7.8	(Wei, Zhou, Fan, & Liu, 2012)
	1027.03	3.2 to 1.3 $\mu$ s	28.9- 110 kHz	-	15.6	(L. Zhang et al., 2012)
Mode-locked laser	1069.8	0.58 ps	0.9 MHz	-	0.37	(Zhao, Tang, Wu, Bao, & Loh, 2010)
	1565	0.756 ps	1.79 MHz	65	2	(Q. Bao et al., 2009)
	1561.6	1.3 ps	6.99 MHz	-	10.7	(Song, Jang, Han, & Bae, 2010)
	1568.1	58.8 ps	7.29 MHz	-	1.68	(G. Liu, Feng, Zhang, Jiang, & Zhang, 2015)

### 2.6.2.2 Topological Insulator

TIs have attracted much of the research focus due to the efficient saturable absorption, large modulation depth and high nonlinear susceptibility. The most two important compounds of TI are Bismuth (III) Telluride ( $\text{Bi}_2\text{Te}_3$ ) and Bismuth (III) Selenide ( $\text{Bi}_2\text{Se}_3$ ). The  $\text{Bi}_2\text{Te}_3$  and  $\text{Bi}_2\text{Se}_3$  have complex layer structures, and they react with the elements at 500-900° C. These materials can be used in different fields, such as optoelectronics, television cameras, and switching devices as they have the properties of thermoelectric. However, the constructing process of TI materials are complicated, and thus they have limited applications. Table 2.3 review the utilization of TI in fiber laser technology.

**Table 2.3: TI as an SA for both Q-switch and mode-lock laser**

Laser Type	Wavelength (nm)	Pulse width	Repetition Rate	SNR (dB)	Output power (mW)	References
Q-switched laser	1070	252.6 to 6.6 $\mu\text{s}$	24.4 to 55 kHz	-	18.6	(A. H. H. Al-Masoodi et al., 2018)
	1560	62.72 to 5.1 $\mu\text{s}$	20.99 to 89.29 kHz	71.4	5.6	(Ismail et al., 2018)
	974	39.8 to 4.9 $\mu\text{s}$	40.1 to 6.2	50	6.1	(L. Sun et al., 2014)
	1493.6 - 1508.9	6.1 to 7.64 $\mu\text{s}$	36.6 to 26.1	58	0.21	(H Ahmad, Soltanian, et al., 2015)
Mode-locked laser	1935	0.795 ps	27.9 MHz	-	-	(Jung et al., 2014)
	1557	1.08	8.635	60	-	(Mao et al., 2015)
	~1031	46 ps	44.6 MHz	58	33.7	(Dou et al., 2014)

### 2.6.2.3 Transition Metal Dichalcogenides

Semiconducting TMDs such as MoS<sub>2</sub>, MoSe<sub>2</sub> and WS<sub>2</sub> are having renewed interest for photonic and optoelectronic applications, expanding upon studies made in the 1960s. (Frindt, 1965; Wilson & Yoffe, 1969), with the use of modern processing techniques allowing new insight. The properties of TMDs based SA depends on the number of layers in the material (Q. H. Wang, Kalantar-Zadeh, Kis, Coleman, & Strano, 2012). For example, MoS<sub>2</sub> has an indirect band gap around 1.29 eV band gap, which increases because of stronger quantum confinement as the number of layers is reduced, resulting in a direct band gap of 1.80 eV (Mak, Lee, Hone, Shan, & Heinz, 2010). MoS<sub>2</sub> is widely used in microelectronics devices due to its good absorption coefficient (Wen, Xu, Liu, Lai, & Tang, 2017). However, TMDs suffer from a low optical damage threshold (Horiguchi, Honda, Kato, Nakashima, & Niidome, 2008; Latiff et al., 2017). Table 2.4 reviews the demonstration of TMDs in fiber laser technology.

**Table 2.4: TMD as a SA for pulsed lasers**

Laser Type	Wavelength (nm)	Pulse width	Repetition Rate	SNR (dB)	Output power (mW)	Reference
Q-switched laser	1551.4	10.7 to 5 $\mu$ s	14.25 to 38.43 kHz	37	5.2	(M. H. M. Ahmed, A. H. H. Al-Masoodi, A. A. Latiff, H. Arof, & S. W. Harun, 2017)
	1086& 1093	21.3 to 10 $\mu$ s	4.8 to 17.2 kHz	-	60	(Tey et al., 2017)
	1559.8	14.28 to 9.6 $\mu$ s	70 to 104.1 kHz	-	12.82	(N. Razak, Yasin, Zakaria, Abdul Latiff, & Harun, 2017)
Mode-locked laser	1598.94	0.83 ps	17.1 MHz	41	1.26	(Ahmed, Latiff, Arof, Ahmad, & Harun, 2016)

**Table 2.4, continued**

<b>Laser Type</b>	<b>Wavelength (nm)</b>	<b>Pulse width</b>	<b>Repetition Rate</b>	<b>SNR (dB)</b>	<b>Output power (mW)</b>	<b>Reference</b>
Mode-locked laser	1073.86	28.3 ps	18.8 MHz	30	1.82	(A. H. H. Al-Masoodi, Ahmed, et al., 2017)
	1559	2.46 ps	1.8 MHz	62	-	(G. Wang, 2017)

#### 2.6.2.4 Black Phosphorous

Black Phosphorous (BP) has recently joined the family of 2D materials (Churchill & Jarillo-Herrero, 2014). BP is widely used in optoelectronic and electronic applications as it has the properties of excellent conductivity and direct band gap (Han et al., 2017). BP has a direct band gap and can be tuned from  $\sim 0.3$  to  $\sim 2$  eV which correspond to the wavelength between  $\sim 4$  to  $\sim 0.6$   $\mu\text{m}$  (Tran, Soklaski, Liang, & Yang, 2014). This feature makes it an interesting SA for fiber laser application. The research on BP has made steady progress in last few decades, including the study of crystal structures (Jamieson, 1963), superconducting properties (Wittig & Matthias, 1968), optical properties (Warschauer, 1963), and optical phonon (Ikezawa, Kondo, & Shirotnani, 1983). However, these works did not attract much attention from the semiconductor research community. BP was only rediscovered from the perspective of 2D nanomaterials only when successful studies of graphene and few-layer TMDs were made. However, it should be mentioned that BP suffers from low damage threshold (Latiff et al., 2017), additionally, BP is a hydrophilic material (J. Li et al., 2016) which is easily damaged when exposed to water or oxygen. The table below review the work achieved by using BP as SA.

**Table 2.5: BP as a SA for passive pulsed lasers**

<b>Laser Type</b>	<b>Wavelength (nm)</b>	<b>Pulse width</b>	<b>Repetition Rate</b>	<b>SNR (dBm)</b>	<b>Output power (mW)</b>	<b>Reference</b>
Q-switched laser	1550.9	22.34 to 5.35 $\mu$ s	7.46 to 28.57 kHz	-	0.18	(Fauziah, Rosol, Latiff, & Harun, 2017)
	1552.9	20.5 to 7.04 $\mu$ s	9.1 to 44.33 kHz	50	5.7	(M. H. M. Ahmed et al., 2017)
	1562	40 to 9.84 $\mu$ s	9.6 to 44.72 kHz	67	3.54	(Huanhuan Liu et al., 2019)
	1069.4	7.9 To 10.8 $\mu$ s	8.2 to 32.9 kHz	30	10	(Ahmed Al-Masoodi, Ahmed, Abdul Latiff, Arof, & Harun, 2016)
Mode-locked laser	1560.7	0.57 ps	6.88 MHz	59	0.3	(Ahmed, Latiff, Arof, & Harun, 2016)
	1033.76	3.27 ps	10 MHz	39	4.8	(A. H. H. Al-Masoodi, Yasin, et al., 2017)
	1085.58	7.54 ps	13.5 MHz	45	10	(Hisyam, Rusdi, Latiff, & Harun, 2017)
	1571.45	0.946 ps	5.96 MHz	70	-	(Y. Chen, G. Jiang, S. Chen, Z. Guo, X. Yu, C. Zhao, H. Zhang, Q. Bao, S. Wen, D. Tang, et al., 2015)

## CHAPTER 3: PREPARATION AND CHARACTERIZATION OF ORGANIC SATURABLE ABSORBERS

### 3.1 Introduction

Organic materials have received considerable attention in the literature recently due to their many advantages. For instance, their excellent mechanical and chemical properties allow them to be processed by highly scalable industrial approaches. The ability of these materials of creating an interface with living tissues and cells has also received significant attention during the last few years. One of the main reasons that made organic materials successful is their ability to form close contact with the biological material (Šafaříková et al., 2018). The advance development of these materials has led to the demonstration of plastic bioelectronic devices that can be utilized as an efficient substitute for conventional devices based on inorganic materials and precious metals. They have been used in the application of cell cultures for modulating or sensing the electrical activity of cells (Martin, 2015; Owens & Malliaras, 2011; Rivnay, Owens, & Malliaras, 2014). Additionally, organic materials can also be processed so that they can be implanted into the human body for applications in drug delivery, neural recording and sensing (Kaltenbrunner et al., 2013; Khodagholy et al., 2014; Someya, Bao, & Malliaras, 2016). These materials can be used to develop ultralight, weight ultra-flexible and stretchable sensor arrays.

Organic materials might have the potential to be used as a bio-compatible, environmentally-friendly and high performance saturable absorbers (SAs). This is attributed to their ability to provide a large and ultrafast nonlinear response and broad spectral tunability (Boulet, Mohammadpour, & Shankar, 2015; Clark & Lanzani, 2010; Williams, 1983). They also offer mechanical flexibility, light-weight and low production cost (Someya et al., 2004) and thus they might increase the potential for applications in

biomedical laser processing and sensing (Khazaeinezhad et al., 2017). These materials, in comparison to inorganic materials, require simple fabrication and offer versatile molecular design (Clark & Lanzani, 2010; C. W. Lee, Kim, & Lee, 2014). Due to their customized properties for optoelectronics, organic materials have been utilized in various applications including organic solar cells (OSCs) (J. Y. Kim et al., 2007; Peumans, Uchida, & Forrest, 2011; Tang, 1986; Xue, Uchida, Rand, & Forrest, 2004), organic light-emitting diodes (OLEDs) (Adachi, Baldo, Thompson, & Forrest, 2001; Holmes et al., 2003; O. Y. Kim & Lee, 2012; Yook & Lee, 2012), organic bistable memory devices (Briseno et al., 2006; Muccini, 2006) and organic thin-film transistor (C. Kim, Facchetti, & Marks, 2007; J.-M. Kim et al., 2012).

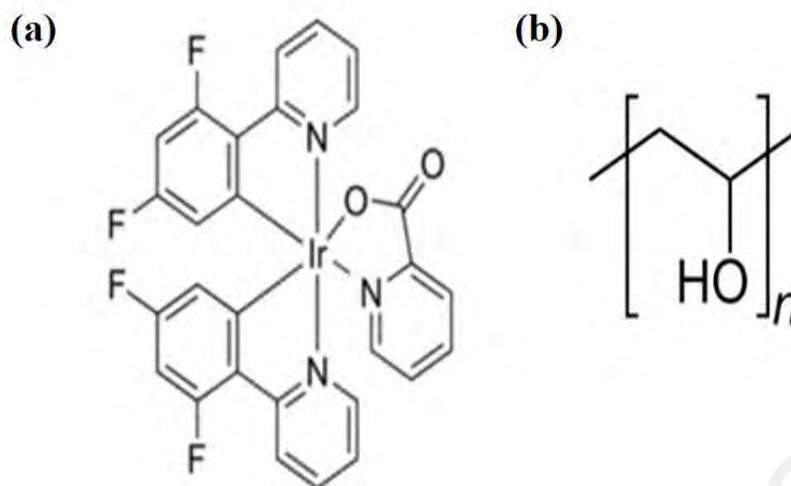
Up to date, many works have been devoted to the demonstration of pulsed fiber laser by using a passive SA. Therefore, finding a new material to be used as a high-performance SA is of great interest, since the current SAs still suffer from many drawbacks as discussed in the previous chapters. Besides, there have been increasing concerns about the usage of the nano-materials as SAs as they might be dangerous on human health (Khazaeinezhad et al., 2017). Therefore, a non-hazardous biocompatible alternative SA would be highly favourable, especially for developing environmentally-friendly photonic devices which could find increasing applications in in-situ or in-vivo sensing and biomedical laser processing. Additionally, most of the developed SAs still have the issues of wavelength dependency, high manufacturing cost, difficulty in processing and difficulty in controlling the physical properties. A new SA that can overcome the above drawbacks will have a great research impact and could find many practical applications.

In this research work, three organic materials; Bis[2-(4,6-difluorophenyl)pyridinato-C<sup>2</sup>,N](picolinato)iridium(III) (FIrpic), bis(8-hydroxyquinoline)zinc (Znq<sub>2</sub>) and Tris(8-hydroxyquinoline)aluminum (Alq<sub>3</sub>) were proposed and demonstrated as SAs for both Q-switched and mode-locked pulse generations. This chapter discusses the fabrication and

characterization of these SAs. These materials, FIrpic,  $Znq_2$  and  $Alq_3$ , have previously attracted much attention as biological labels and probes as they have a higher resistance to photobleaching and intense and long-lived emission (Lo, 2007). Compared with the conventional inorganic materials, they have comparable high optical damage threshold (higher than 350 mW) and high output pulses energy. Furthermore, they have a low-cost and straightforward fabrication process, which could make them very useful and attractive in pulsed fiber laser technology.

### **3.2 Fabrication and Characterization of FIrpic as SA**

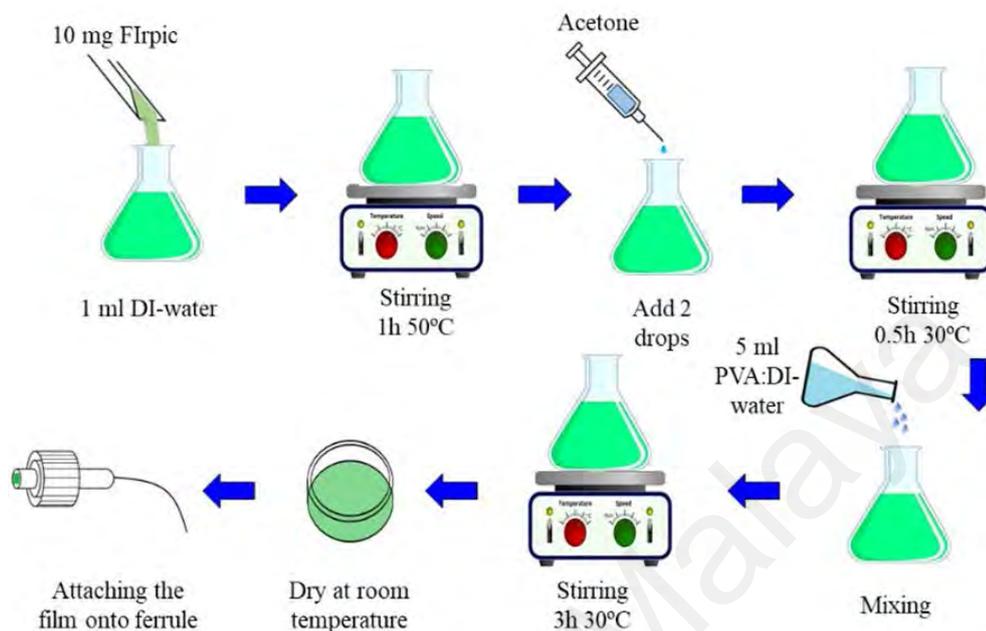
FIrpic is a blue phosphorescent bis-cyclometallated iridium complex. It can be produced by a straightforward synthesis process and has excellent stability and desirable electrochemical properties. FIrpic also has a suitable energy level and high emission efficiency (E. Baranoff & B. F. E. Curchod, 2015). It remains stable on the substrate during deposition regardless of the surface morphology. FIrpic also remains stable without any compositional or surface morphological damage after being exposed to an atmospheric environment or X-ray irradiation (Park et al., 2012). For these qualities, FIrpic is expected to become an ideal material for use as an organic SA. In this work, FIrpic was embedded into polyvinyl alcohol (PVA) to form a thin-film so that it can be easily integrated into a laser cavity to function as SA. PVA is reported to be an excellent host for producing a high-performance SA (Mao et al., 2015). Additionally, PVA has been intensively used in many applications due to its favourable physical properties such as biocompatibility, hydrophilicity and good chemical resistance (X. Chen, 2002; K. Wu, Zhang, Wang, Li, & Chen, 2015). Figures 3.1(a) and (b) show the molecule structure for FIrpic and PVA, respectively.



**Figure 3.1: Molecular structures of (a) FIrpc and (b) PVA**

FIrpc material used in this study was purchased from Sigma Aldrich. The FIrpc-PVA thin-film was prepared by a solvent casting technique and the preparation process is summarized in Figure 3.2. At first, 1 ml of deionized (DI) water was added to 10 mg of FIrpc material. The solution was stirred at 50° C for one hour then 2 drops of acetone were added to the solution to help in solving the FIrpc so that it could be distributed equally inside the solution. On the other side, PVA solution was prepared by adding of 1 g of PVA into 100 ml of DI water following by ultra-sonication for one hour at room temperature. Next, 5 ml of PVA solution was mixed with the prepared FIrpc solution. The mixture was then stirred for about three hours. After that, the mixture was poured to a borosilicate glass petri dish and dried at room temperature for 3 days. A thin-film with a diameter of 60 mm was obtained. The thickness of the produced films is about 50  $\mu\text{m}$  and the mass fraction of FIrpc is about 16.7 wt%. The mass fraction of FIrpc is calculated by  $\frac{w_{FIrpc}}{(w_{PVA}+w_{FIrpc})} \times 100$ , where  $w_{PVA}$  and  $w_{FIrpc}$  are the weights of PVA and FIrpc, respectively (Hamdalla & Hanafy, 2016). Finally, a small piece of the thin-film was attached to the fiber ferrule for use as SA device. The pristine PVA thin-film was also prepared in this work from the PVA solution for comparison purpose. It was obtained by

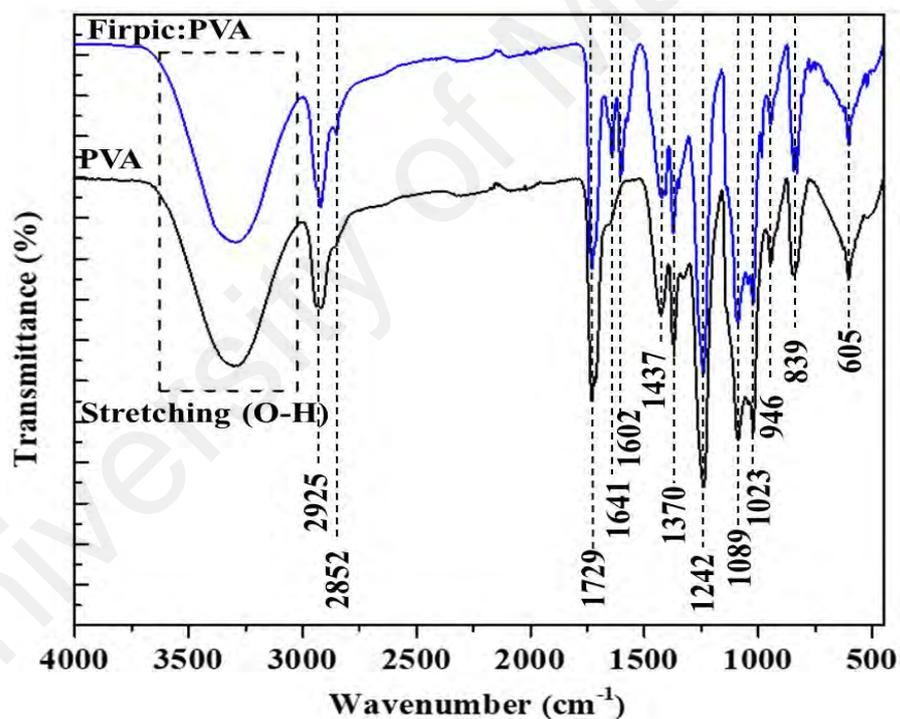
pouring the prepared PVA solution onto a borosilicate glass petri dish and dried it at room temperature for about 3 days.



**Figure 3.2: Fabrication process of FIrpc-PVA thin-film**

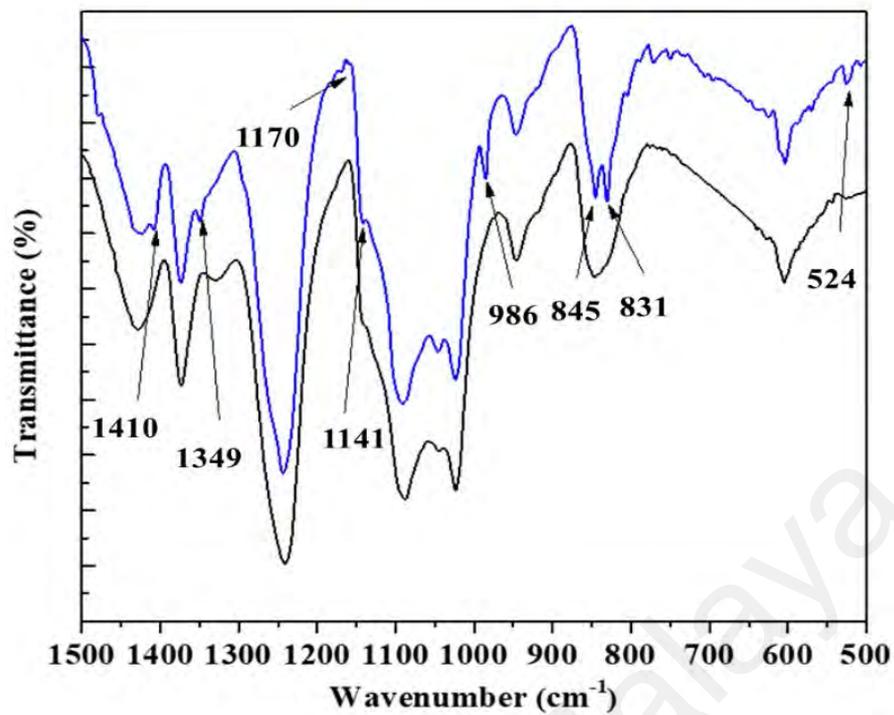
The chemical groups in the PVA and FIrpc-PVA thin-films were investigated using Perkin Elmer Spectrum 400 Fourier Transform Infrared Spectroscopy (FTIR) Spectrometer. FTIR spectra were obtained for both PVA and FIrpc-PVA thin-films based on transmittance mode in the range of wavenumber from  $4000$  to  $450\text{ cm}^{-1}$  with a resolution of  $2\text{ cm}^{-1}$ . Figure 3.3 (a) compares the plotted spectrum of the FIrpc-PVA with the pristine PVA film. Most of the characteristic bands correspond to PVA could be observed in a pristine PVA film. In the spectrum of PVA, there is a broad and strong absorption at  $3000\text{--}3600\text{ cm}^{-1}$ , peaking at  $3298\text{ cm}^{-1}$ , which is due to the symmetrical stretching vibration of O–H from the intermolecular and intramolecular hydrogen bonds. The PVA stretching vibration peaks of  $\text{CH}_2$ , CH, C=O, C–O–C, C–O and C–C were observed at  $2925$ ,  $2852$ ,  $1729$ ,  $1089$ ,  $1023$  and  $839\text{ cm}^{-1}$ , respectively, while the  $\text{CH}_2$  bending, (CH+OH) bending, CH wagging and  $\text{CH}_2$  rocking and (OH) wagging of PVA appear at  $1437$ ,  $1370$ ,  $1242$ ,  $946$  and  $605\text{ cm}^{-1}$ , respectively. Two peaks significantly

appear at 1641 and 1602  $\text{cm}^{-1}$  in the Firpic-PVA thin-film which belong to the stretching vibration of C=C and C=N in the Firpic molecules, respectively (Hammer, Baker, Lenardi, & Gissler, 1997; Y. C. Kong, Yang, Li, & Chen, 2015; Lei et al., 2017). Figure 3.3 (b) shows the vibration peaks of small intensities at the fingerprint region of the FTIR spectrum ( $1500\text{-}500\text{ cm}^{-1}$ ) after adding Firpic into PVA. The appearance of these peaks is due to the small fraction of the organic material. These peaks refer to C-F, C-N and C-O stretching vibration bonds at the range of  $1400\text{-}1000\text{ cm}^{-1}$ , while the peaks at the range between  $1000\text{-}500\text{ cm}^{-1}$  correspond to the bending vibration of C-H and O-H bonds (DeCoste & Peterson, 2013; Mehta et al., 2016; Yap, Kida, Aoyama, Mori, & Sasaki, 1998).



(a)

**Figure 3.3: (a) FTIR spectra (b) FTIR fingerprint region of PVA and Firpic-PVA thin-films**

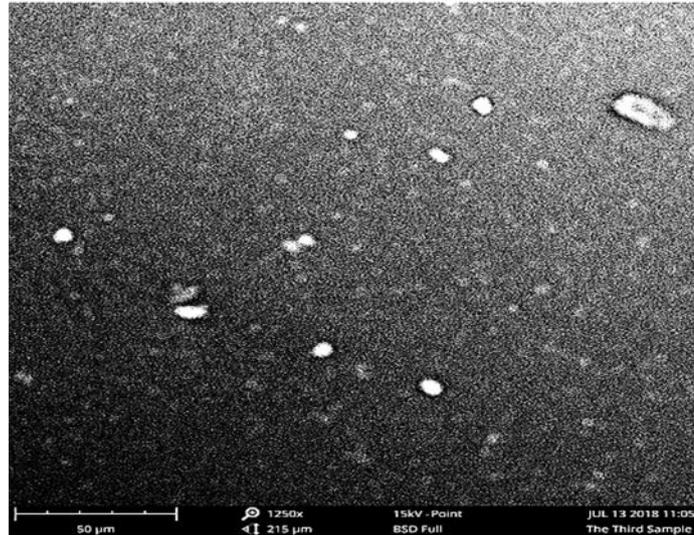


(b)

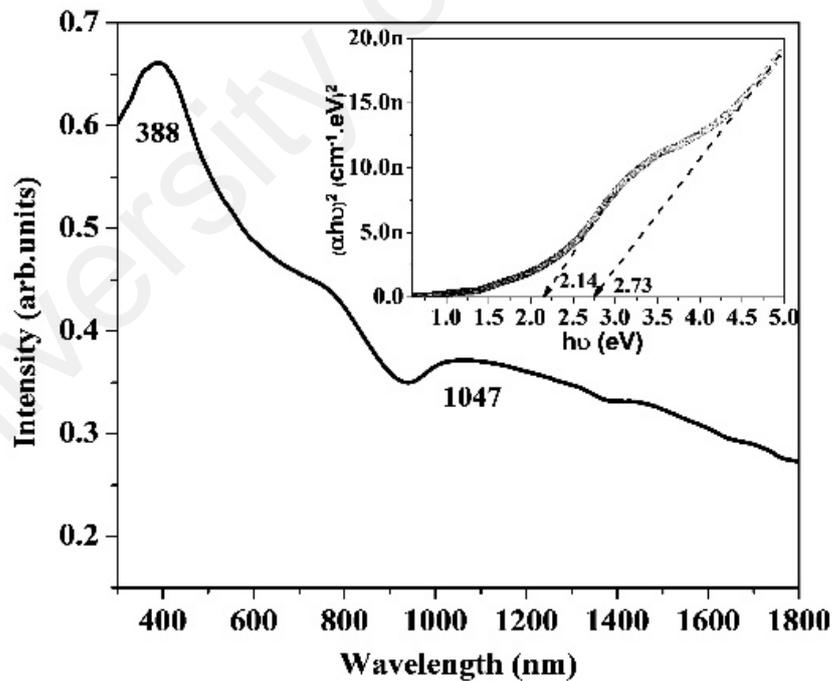
**Figure 3.3, continued**

SEM image of FIrpic-PVA thin-film, which is presented in Figure 3.4 (a), shows a homogenous surface with the appearance of some micron-sized particles in the range of 2-13  $\mu\text{m}$ . These particles could belong to FIrpic agglomeration powder and the homogenous surface reveals the uniform distribution of FIrpic molecules along with the polymer. Figure 3.4 (b) shows the optical absorbance spectrum of FIrpic-PVA thin-film. Two asymmetric and broaden peaks centred at 388 and 1047 nm, respectively, can be clearly detected. These peaks could belong to the absorption band of FIrpic and PVA doped with FIrpic molecules, respectively. Baranoff and Curchod reported the absorption band of FIrpic in  $\text{CH}_2\text{Cl}_2$  solution at 256 nm (E. Baranoff & B. F. Curchod, 2015). The redshift of the FIrpic absorption band is due to the formation of the micro-particles during fabrication and it is embedded in PVA polymer. The PVA absorption band comes from the doped material. The optical band gap,  $E_g$ , of the composited thin-film can be calculated from the following equation  $(\alpha h\nu)^n = B(h\nu - E_g)$ , where  $h\nu$  is the photon

energy,  $B$  is a constant relative to the material and  $n$  equals to 2 for direct transition and  $\alpha$  is the absorption coefficient which can be calculated by Beer-Lambert's law as  $\alpha(\nu) = 2.303 \times Abs(\lambda)/d$ , where  $d$  is the thin-film thickness.



(a)



(b)

**Figure 3.4: (a) SEM image of FIRpic-PVA thin-film, (b) optical absorption spectrum of FIRpic-PVA thin-film. (An inset Figure is the calculated optical band gap obtained by extrapolation to  $\alpha = 0$ )**

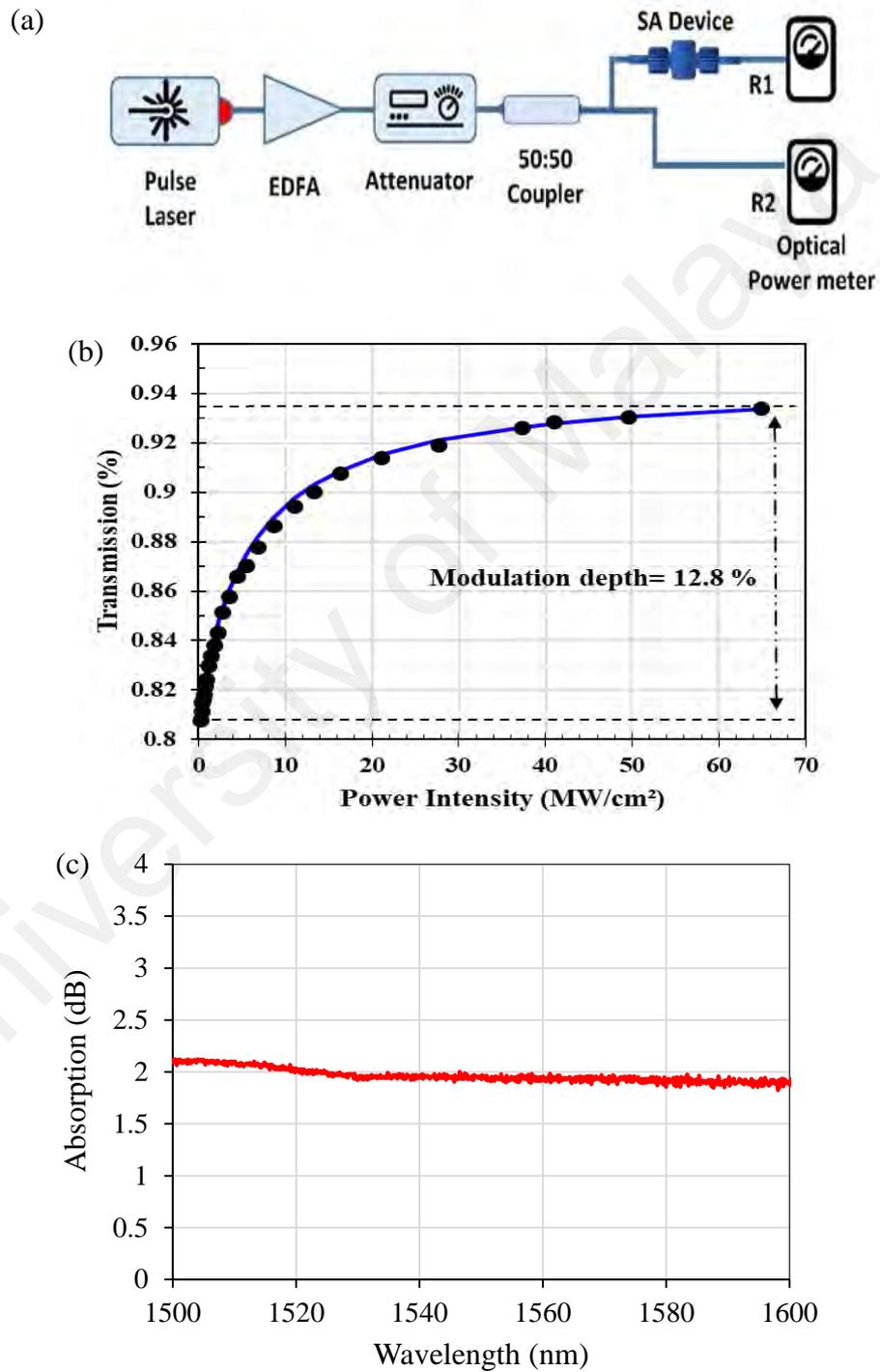
By linear extrapolation at the horizontal axis of  $(\alpha h\nu)^2$  against  $h\nu$ , we can obtain the optical band gap which is equal to  $h\nu$ . Two band gaps of FIRpic:PVA thin-film can be observed at about 2.14 and 2.73 eV, as can be seen in the inset of Figure 3.4 (b). These band gaps correspond to the PVA doped by FIRpic and micro-particles of FIRpic. Doping PVA by material modifies the band gap of PVA (6.28 eV) (Abdullah, Aziz, Omer, & Salih, 2015). So, 16.7 wt% of FIRpic significantly decreases the band gap of the PVA. It should be mentioned that the energy band gap of FIRpic is 2.65 eV (Adamovich et al., 2003; Ren et al., 2004).

To investigate the saturable absorption of the FIRpic-PVA thin-film, the nonlinear transmission property was characterized using a standard 2-arm transmission measurement scheme. Figure 3.5 (a) shows the measurement set-up, which consists of a home-made mode-locked laser as a pulsed source. The laser used has a pulse width, repetition rate and centre wavelength of 2.83 ps, ~1 MHz and 1572 nm, respectively. An erbium-doped fiber amplifier (EDFA) was used to amplify the mode-locked pulse train and a variable optical attenuator was utilized to vary the output power of the source. The output of attenuator was split equally into the test part (R1) (in which Alq<sub>3</sub>: PVA SA was incorporated) and reference part (R2) by a 3-dB coupler. The output powers of R1 and R2 were measured by an optical power meter. By varying the attenuation, the transmission of the SA was determined by computing the difference between the average output powers of R1 and R2. The experimental data for transmission was fitted according to a simple two-level SA model (Zhen et al., 2015)

$$T(I) = 1 - \alpha_s * \exp(-I/I_{sat}) - a_{ns} \quad (3.1)$$

where  $T(I)$  is the transmission,  $\alpha_s$  is the modulation depth,  $I_{sat}$  is the saturation intensity,  $I$  is the input intensity, and  $a_{ns}$  is the non-saturable absorption. Figure 3.5 (b) shows the plotted nonlinear transmission characteristic of the FIRpic-PVA thin-film. As shown in the figure, it has a modulation depth of ~12.8% with a saturation intensity of 5.5 MW/cm<sup>2</sup>.

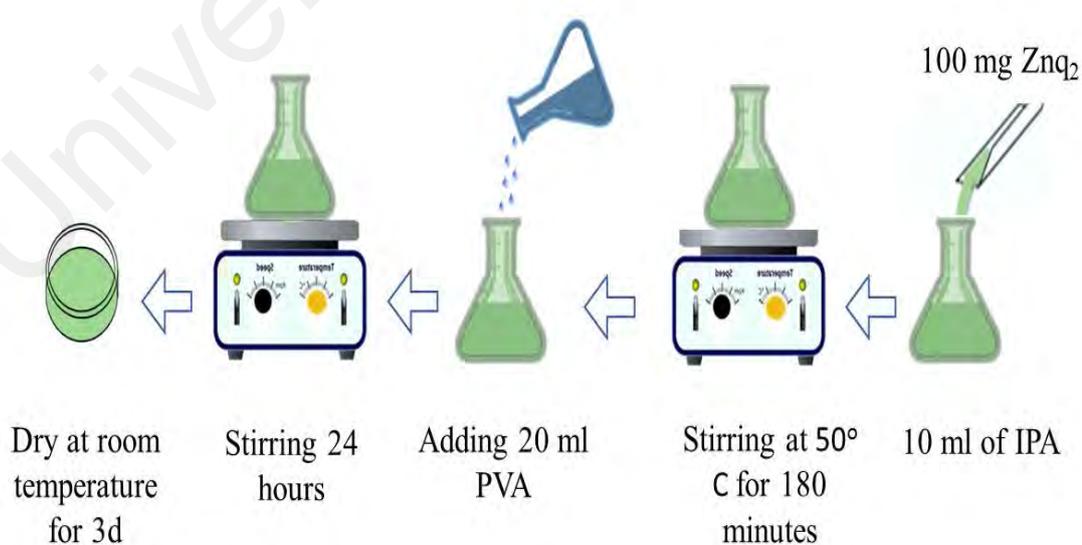
The linear optical absorption of FIRpic PVA is also measured by transmitting a broadband light from the white light source through the thin-film, then the output intensity was measured by an optical spectrum analyzer (OSA). The result is shown in Figure 3.5 (c). The SA shows a flat absorption of about 2 dB in the range of 1500–1600 nm.



**Figure 3.5: (a) Non-linear transmission measurement (b) Non-linear transmission profile and (c) linear absorption of FIRpic: PVA**

### 3.3 Fabrication and Characterization of Znq<sub>2</sub> as SA

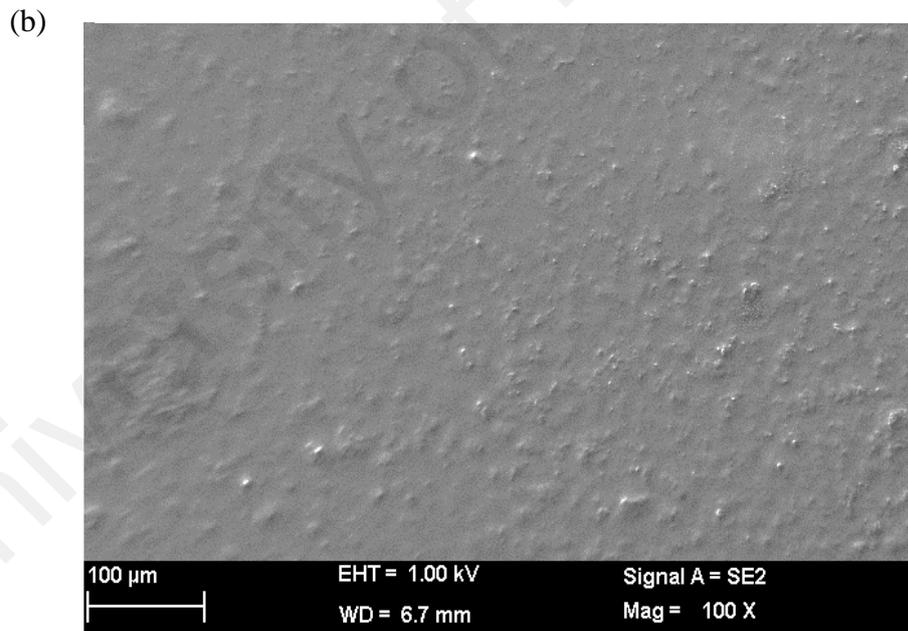
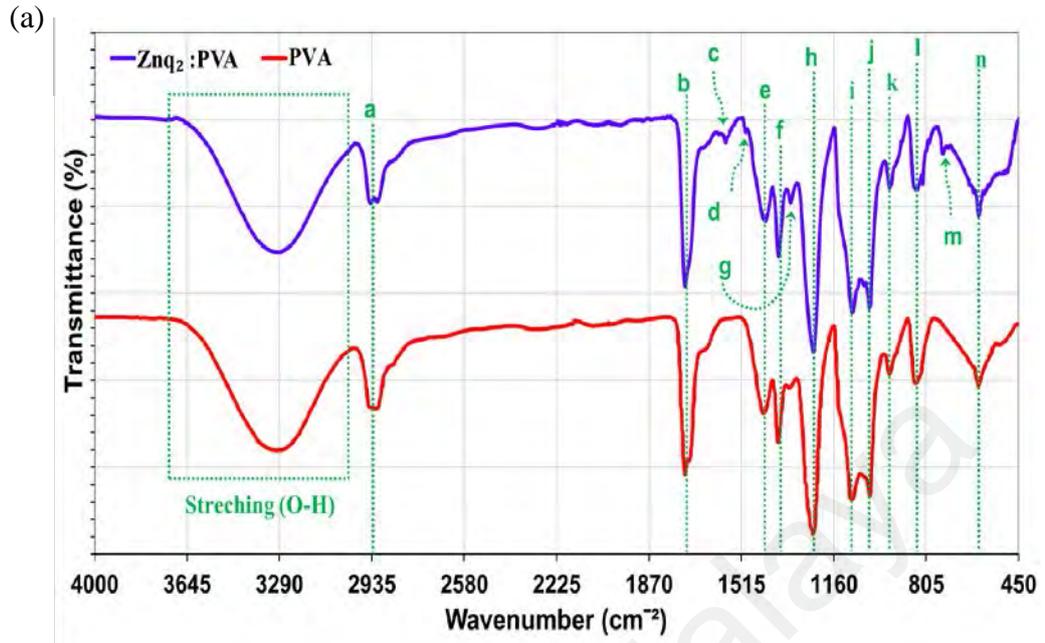
Znq<sub>2</sub> has excellent electron transport and high electroluminescence at room temperature (Yin et al., 2000). It also has high quantum yields, low operating voltage and good injection efficiency. Znq<sub>2</sub> could produce an efficient fluorescence with high stability and this made it one of the most famous and most important light emitter and electron transport material candidates for OLED. In this study, the optical property of the Znq<sub>2</sub> film is further investigated for SA application. The SA was fabricated by the drop-casting technique in which Znq<sub>2</sub> was embedded into PVA to form a thin-film. Znq<sub>2</sub> powder was purchased from Sigma Aldrich, it has a weight of 353.69 g/mol and purity of 99%. First, 100 mg of Znq<sub>2</sub> powder was dissolved into 10 ml of (Isopropyl alcohol) IPA, then the solution was stirred at 50° C for 180 minutes. On the other hand, 1 g of PVA powder was dissolved into 120 ml of distilled water, followed by stirring for 30 minutes. Then, 20 ml of PVA solution was added to Znq<sub>2</sub> solution. The mixture was then stirred for 24 hours at room temperature. The mixture was then poured into a petri dish to form a thin-film. The produced thin-film had a thickness of ~50 μm. The fabrication process is summarized in Figure 3.6.



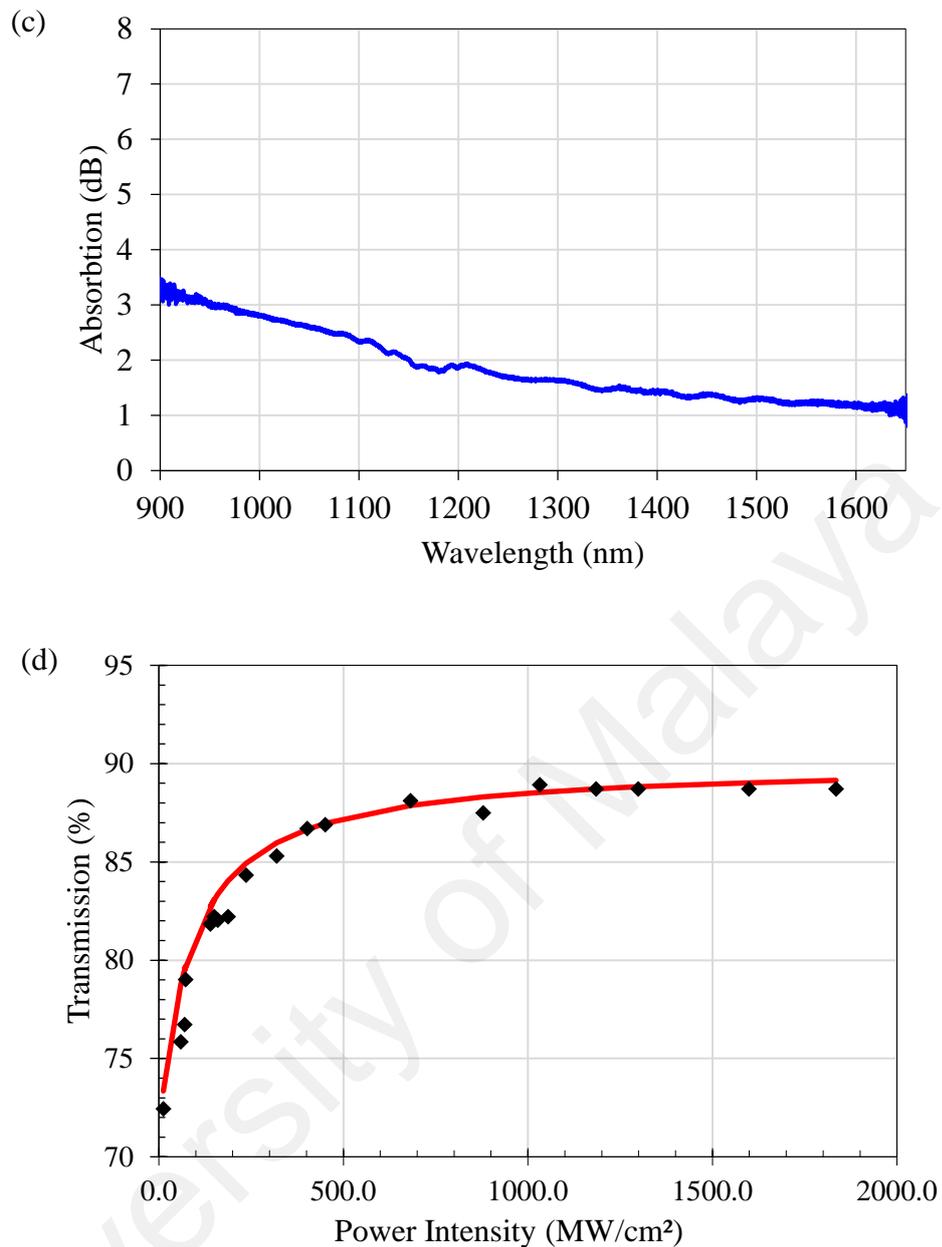
**Figure 3.6: Fabrication process of Znq<sub>2</sub>:PVA SA**

An FTIR spectrometer was then used to analyse the chemical groups in the SA thin-film. Figure 3.7 (a) shows the FTIR transmission spectra obtained in the range between 4000 to 500  $\text{cm}^{-1}$  with a resolution of 2  $\text{cm}^{-1}$  for both PVA and Znq<sub>2</sub>: PVA thin-films. The Znq<sub>2</sub>: PVA curve shows similar FTIR spectrum as PVA except for two peaks at 1577 and 1327  $\text{cm}^{-1}$ , which are assigned to the quinoline group of Znq<sub>2</sub>, additionally, the pyridyl and phenyl groups in Znq<sub>2</sub> are observed 1499  $\text{cm}^{-1}$ . While, the peak at 743  $\text{cm}^{-1}$  is attributed to the in-plane ring deformation (Bharathi Devi, Arulmozhichelvan, & Murugakoothan, 2017; Pan, Liang, Mao, Zhu, & Chen, 2007; Shao, Wang, Wang, & Chen, 2011). Figure 3.7 (b) shows the SEM image of Znq<sub>2</sub>: PVA. It shows a homogeneous distribution of the Znq<sub>2</sub> within the PVA. The linear absorption of the SA was investigated in the wavelength range between 900 nm to 1650 nm.

The SA had an absorption of  $\sim 1.2$  dB at the region of 1550 nm, see Figure 3.7 (c). The linear absorption was measured by comparing the optical spectrum of the light before and after it penetrates the SA thin-film. To check the non-linear transmission of the SA, a mode-locked fiber with a pulse width, repetition rate, and central wavelength of 1.2 ps, 2 MHz and 1562 nm, respectively, was used as pulse source as described in the previous section. An EDFA and variable optical attenuator were used to amplify and to vary the output power of the pulsed laser, respectively. The output of attenuator was split equally into two points, R1 and R2. R1 represents the output power as it went through the SA while R2 is the reference point (i.e. the output power). The transmission of the SA was determined by computing the difference between R1 and R2. Znq<sub>2</sub> has a modulation depth and saturation intensity of 17.6% and 103.6  $\text{MW}/\text{cm}^2$ , see Figure 3.7 (d)



**Figure 3.7: (a) FTIR of Znq<sub>2</sub>: PVA and PVA thin-films (a=2925, b=1729, c=1577, d=1499, e=1428, f=1374, g=1327, h=1242, i=1089, j=1023, k=946, l=845, m=743 and n=604 cm<sup>-1</sup>) (b) SEM image, (c) linear absorption spectrum of Znq<sub>2</sub>: PVA thin-film and (d) modulation depth of Znq<sub>2</sub>: PVA thin-film**

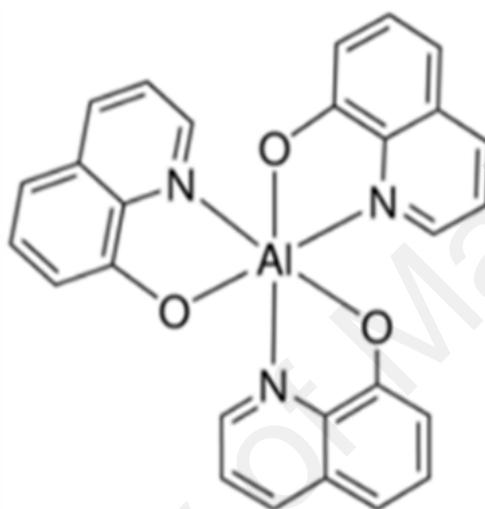


**Figure 3.7, continued**

### 3.4 Fabrication and Characterization of Alq<sub>3</sub> as SA

Alq<sub>3</sub> is considered the most rigorously studied small-molecule organic semiconductor (Xie et al., 2016); thus it became the choice for organic SA in this research work. Alq<sub>3</sub> which has superior thermal stability, can be easily synthesized and purified (Hairong Li, Zhang, Wang, & Zheng, 2003). Alq<sub>3</sub> also has high photoconductivity and long operational lifetimes (Fukuda, Wei, Ichikawa, & Taniguchi, 2007). Furthermore, Alq<sub>3</sub> has the

advantages of highly stable thin-film formation and excellent heat resistance (Yawalkar, Dhoble, Thejo Kalyani, Atram, & Kokode, 2013). Alq<sub>3</sub> powder used was purchased from Sigma Aldrich (product no. 697737-1G) (Sigm-Aaldrich, 2019) and it has a purity of 99.995% and molecular weight of 459.43 g/mol. Figure 3.8 shows the molecular structure of the Alq<sub>3</sub>, which was embedded into the PVA polymer by a drop-casting technique at room temperature.



**Figure 3.8: Molecular structure of Alq<sub>3</sub>**

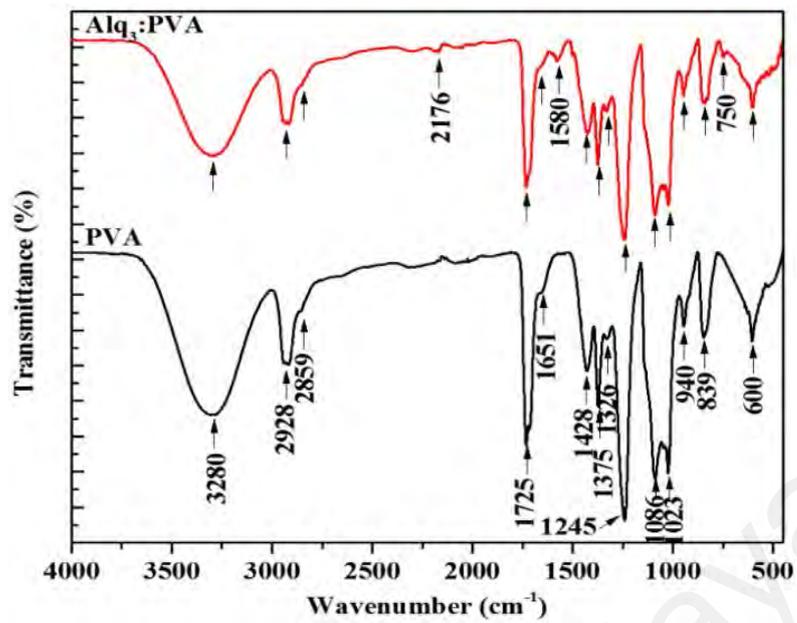
The fabrication process of the Alq<sub>3</sub>: PVA thin-film is illustrated in Figure 3.9. Firstly, 1 ml of distilled water was added to 10 mg of purchased Alq<sub>3</sub> powder. Subsequently, the solution was stirred at 50° C for one hour and then 2 drops of acetone were added to the solution for equal distribution of Alq<sub>3</sub> along with the solution. On the other side, 1 g of PVA was dissolved in 100 ml distilled water followed by ultra-sonication for one hour at room temperature. Next, 5 ml of PVA solution was mixed with the Alq<sub>3</sub> solution. The mixture solution was then stirred for about three hours at room temperature. After that, the mixture was poured into a borosilicate glass petri dish with a diameter of 60 mm to form a thin-film, which can be easily separated after drying for three days at room temperature. The thickness of the thin-film was measured using Mitutoyo micrometer

screw gauge and it was about 50  $\mu\text{m}$ . Finally, the fabricated film was cut into a tiny piece and attached to a fiber ferrule.

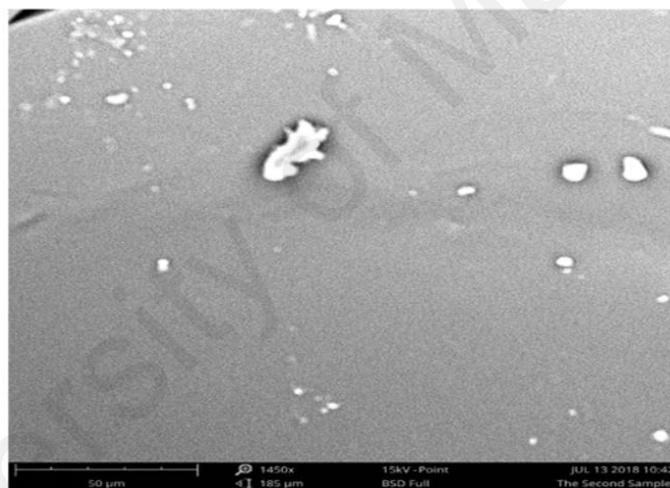


**Figure 3.9: The fabrication process of Alq<sub>3</sub>: PVA thin-film**

The fabricated film was characterized to analyze the chemical groups in the materials by using Perkin Elmer Spectrum 400 FTIR Spectrometer. In the experiment both pure PVA and Alq<sub>3</sub>: PVA thin-films were analyzed based on transmittance mode with a resolution of 2  $\text{cm}^{-1}$ . Figure 3.10 (a) shows the FTIR transmission spectra for both PVA and Alq<sub>3</sub>: PVA thin-films, which were obtained in the range of wavenumber from 4000 to 500  $\text{cm}^{-1}$ . As shown in the figure, various FTIR absorption peaks were obtained for PVA, which are related to the hydroxyl and acetate groups. On the other hand, the Alq<sub>3</sub> embedded into PVA thin-film shows similar FTIR spectrum as PVA thin-film except for the appearance of three peaks at 2176, 1580 and 750  $\text{cm}^{-1}$ . 2176, 1580 and 750  $\text{cm}^{-1}$  peaks correspond to Al-O-H, C=N and Al-N modes for Alq<sub>3</sub> chemical bonding (Correia, Santos, Garcia, Peres, & Wang, 2015; Laukamp, Salama, & González-Álvarez, 2016; Quintero et al., 2013), respectively.



(a)



(b)

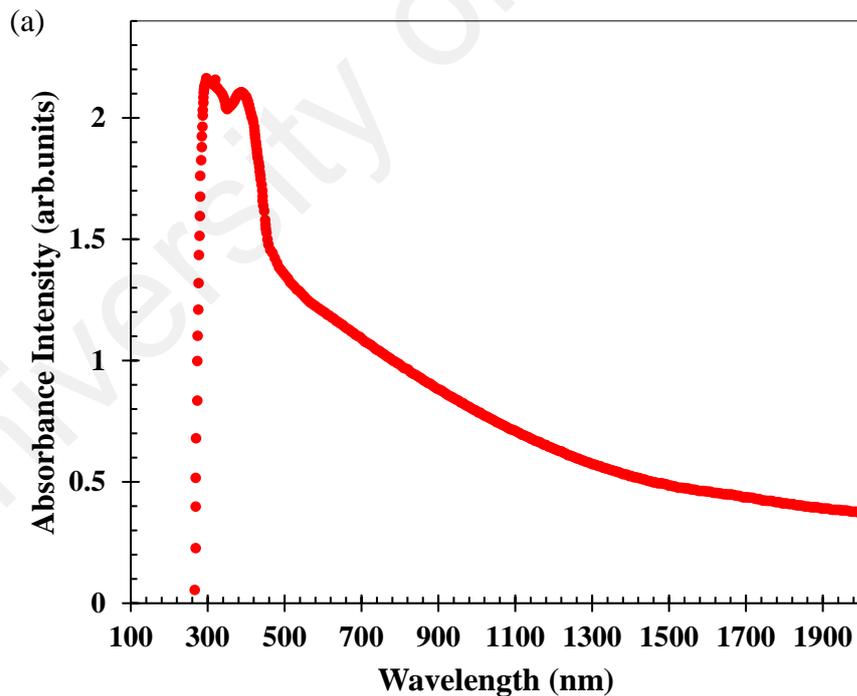
**Figure 3.10: (a) FTIR spectra of PVA and Alq<sub>3</sub>: PVA thin-films and (b) SEM of Alq<sub>3</sub>: PVA thin-film**

The SEM image of Alq<sub>3</sub>:PVA thin-film was also obtained as shown in Figure 3.10 (b). It indicates a soft surface with a uniform distribution of Alq<sub>3</sub> in the PVA polymer. The dots shown on the surface is contamination, which could be formed from the aggregated particles of the large molecules of Alq<sub>3</sub> which are difficult to be dispersed among the PVA molecules. The absorbance characteristic of the prepared Alq<sub>3</sub>: PVA polymer thin-film was also measured using a spectrophotometer (Perkin Elmer, Lambda

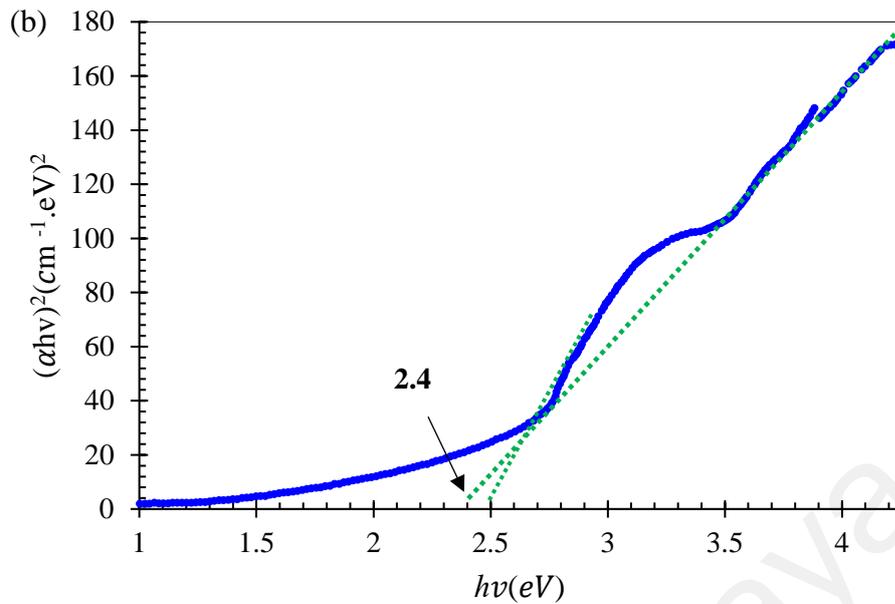
750), which operates in a wavelength range between 250 and 2000 nm. The optical absorbance spectrum of the Alq<sub>3</sub>:PVA thin-film is depicted in Figure 3.11 (a). The appearance of two peaks is seen at around 300 and 385 nm which are assigned to the  $\pi \rightarrow \pi^*$  transition of PVA and Alq<sub>3</sub> materials, respectively due to unsaturated bonds (Aziz, Rasheed, Saeed, & Abdullah, 2017; Cuba & Muralidharan, 2015; Elashmawi, Hakeem, & Selim, 2009). The absorption coefficient ( $\alpha$ ) can be obtained by Beer-Lambert's law as  $\alpha(\nu) = 2.303 \times Abs(\lambda)/d$ , where  $d$  is the thin-film thickness and it is around 50  $\mu\text{m}$ . Alq<sub>3</sub> is an organic semiconductor and thus the optical band gap,  $E_g$ , can be estimated from the absorption coefficient in equation  $(\alpha h\nu)^n = B(h\nu - E_g)$  by extrapolating  $h\nu$  to  $\alpha = 0$ , where  $B$  is a constant relative to the material,  $h\nu$  is the photon energy, and  $n = 2$  for direct transition. The observed band gaps of the Alq<sub>3</sub>: PVA thin-film are about 2.5 and 2.4 eV, as can be seen from the Tauc plot in Figure 3.11 (b), which belong to Alq<sub>3</sub> and modified PVA thin-film (Abdullah et al., 2015; Divayana et al., 2007), respectively. The optical band gap of Alq<sub>3</sub> can be recognized due to the large particles of Alq<sub>3</sub> on the Alq<sub>3</sub>: PVA thin-film, as can be observed in the SEM image. Moreover, doping PVA by 10 mg of Alq<sub>3</sub> modified the optical band gap of PVA from 6.28 to 2.4 eV due to the uniform distribution of the Alq<sub>3</sub> molecules along with the PVA polymer (Abdullah et al., 2015).

It should be noted that even though the band gap of Alq<sub>3</sub> is about 2.5 and 2.4 eV (which means that absorption peaks of Alq<sub>3</sub> are located near 500 nm), the saturable absorption was observed near 1000 nm which is sub-band gap absorption. The sub-band gap absorption is attributed to some defects in the material. The evidence of these defects and their physical origin in Alq<sub>3</sub> is addressed in ref. (Greco, Mirea, Ghica, Cölle, & Schwoerer, 2005). These defects create new states in the band gap. These states absorb sub-band gap photons and affect the absorption spectra, causing artefacts and features that interpreted as lower band gap than the actual non-defected value. These defects lead to decreasing the optical energy gap and shifting the absorption edge towards the higher

wavelength of the incident (S. Wang et al., 2014). A similar case has been reported in ref. (K. Wu et al., 2015). The authors used WS<sub>2</sub> as SA in which it has a direct band gap of ~2.0 eV (~630 nm) and the indirect band gap is ~1.4 eV (~886 nm) (Chhowalla et al., 2013), but they still produced a mode-lock operation in 1.5 μm region. The authors attributed the sub-band gap absorption to some defects as well as absorption of edge modes and two-photon absorption (TPA). MoS<sub>2</sub> also has a direct band gap of ~1.8 eV (688 nm) and the indirect band gap is 0.86 - 1.29 eV (1443 -962 nm) (S. Wang et al., 2014), but the sub-band gap absorption was observed in which the saturable absorption property was observed in a wide band beyond this limitation. Further investigation is still required to understand the sub-band gap absorption in Alq<sub>3</sub>, as this is beyond the scope of this thesis. That investigation would help to address the absorption mechanism in Alq<sub>3</sub> in 1 μm and 1.5 μm region in further details.

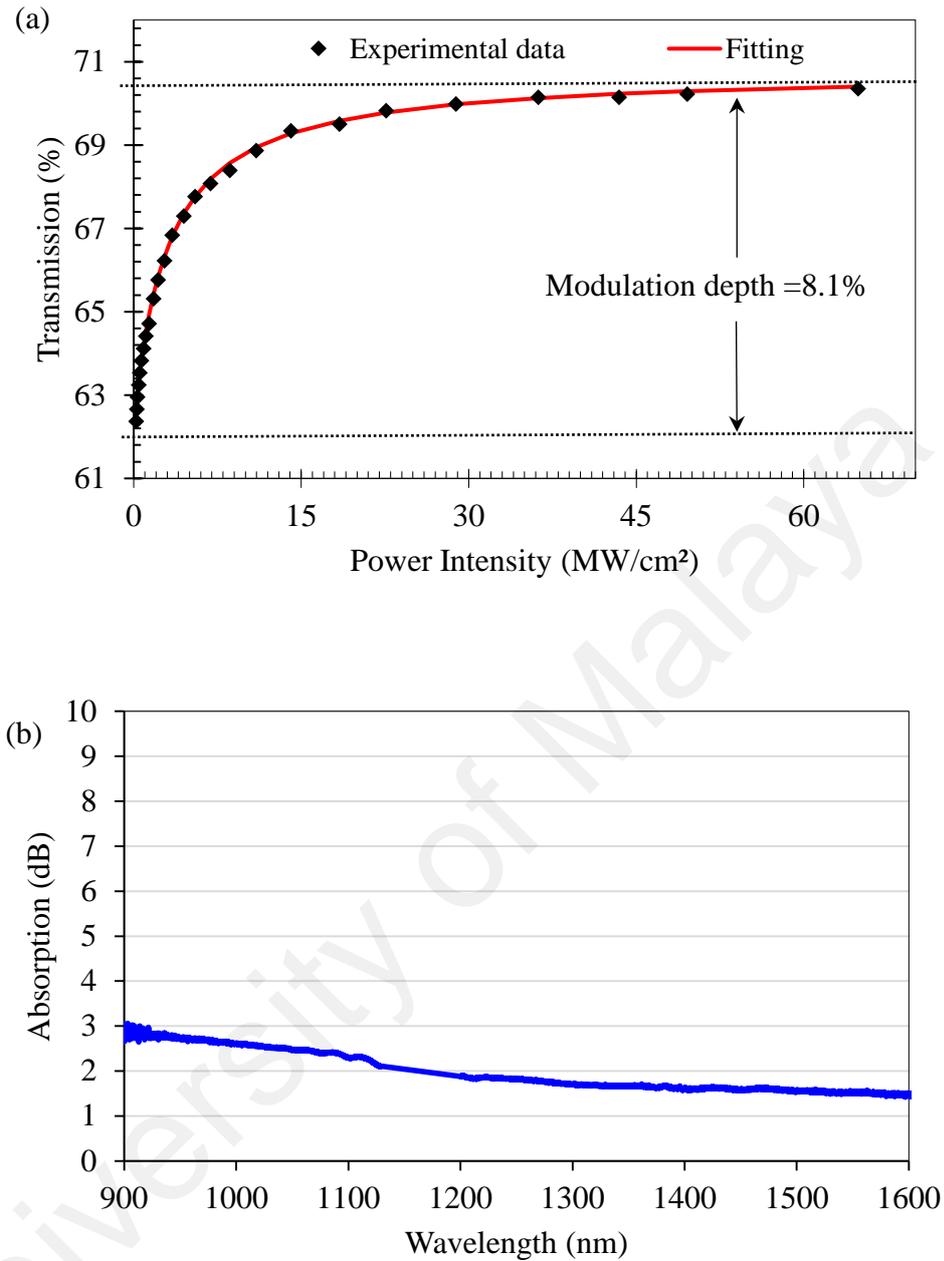


**Figure 3.11: (a) The measured absorption spectrum for the Alq<sub>3</sub>:PVA thin-film and (b) the Tauc's plot**



**Figure 3.11, continued**

The proposed Alq<sub>3</sub>: PVA thin-film has a broadband absorption region covering the visible to 2000 nm as shown in Figure 3.11 (a). This shows the material can be used as SA to generate Q-switched and mode-locked pulses with broad spectral tunability. Besides the linear absorption, the modulation depth of the Alq<sub>3</sub> SA is also important to determine the recovery and response time of the material whether it is fast enough to support both Q-switching and mode-locking pulses generation. For instance, the recovery time should be between the pulse duration (of the Q-switching operation) and the upper-state lifetime of the gain medium. This SA could also be used to produce a mode-locking operation by balancing the dispersion and nonlinearity characteristics of the laser cavity. The non-linear transmission of the Alq<sub>3</sub>: PVA was characterized by using the setup shown in Figure 3.5 (a). Figure 3.12 (a) shows the nonlinear transmission characterization of Alq<sub>3</sub>: PVA film in which a saturation intensity and modulation depth of 3 MW/cm<sup>2</sup> and ~8.1% were obtained, respectively. Figure 3.12 (b) presents that Alq<sub>3</sub>:PVA has a flat absorption of about 2 dB in the range from 900–1600 nm.



**Figure 3.12: (a) Nonlinear transmission and (b) linear absorption of the fabricated Alq<sub>3</sub>: PVA film**

### 3.5 Summary

Three SA thin-films were successfully developed based on three different organic materials; FIrpic, Znq<sub>2</sub> and Alq<sub>3</sub>. Each of these materials was embedded into PVA to form a thin-film by solvent casting technique. The fabricated films were successfully characterized in terms of FTIR spectra, linear absorption and nonlinear transmission. The

characteristics of these films were summarized in Table 3.1. It is obtained that the modulation depth of the three organic materials was above 6%, which should be sufficient to make the developed SAs function as Q-switchers or mode-lockers.

**Table 3.1: Comparison of the modulation depth, saturation intensity and non-Saturable absorbance between Flrpic, Znq<sub>2</sub> and Alq<sub>3</sub>**

SA material	Modulation depth (%)	Saturation intensity (MW/cm <sup>2</sup> )	Non-Saturable absorbance
Flrpic	12.8	5.5	93.5
Znq <sub>2</sub>	17.6	103.6	88.7
Alq <sub>3</sub>	8.1	3	70

## CHAPTER 4: Q-SWITCHED PULSE GENERATION BY USING ORGANIC MATERIALS

### 4.1 Introduction

The search of new techniques for generation of a short pulse laser is always at the centre of research, as pulsed lasers have many applications in medicine, remote sensing, long-range optical communication and fiber sensor. Passive Q-switching is the typical technique and one of the most effective ways of producing short and high-energy laser pulses. Since they are advantageous in terms of simplicity, compactness and design flexibility (Petropoulos, Offerhaus, Richardson, Dhanjal, & Zheludev, 1999). Q-switched lasers can be produced by modulating the Q-parameter (quality factor) such that a series of short and intense pulses are generated (Kurkov, 2011; Morkel, Jedrzejewski, Taylor, & Payne, 1992; Svelto & Hanna, 2013). Here Q is defined as the ratio of the energy stored in the cavity to the losses per oscillation cycle. The pulse width of the Q-switched laser is usually in a range of  $\mu\text{s}$  to few ns while the repetition rate is normally in the kHz region. Q-switched pulses train can be generated from either active or passive methods. Active methods are normally obtained by using electro-optic modulator, which is driven by an external electrical generator to generate pulses (Alvarez-Chavez et al., 2000; Kee et al., 1998). Passive Q-switching, on the other hand, can be realized by incorporating a saturable absorber (SA) device in the laser cavity. Although active Q-switching can address long term stability and controllable repetition rate, it has the disadvantages of low damage threshold and low peak power. On the contrary, passive techniques are more useful due to their advantages of low cost, compactness, simplicity and flexibility as compared to the active Q-switching, which requires additional electronic components (Svelto & Hanna, 2013).

Semiconductor saturable absorber mirror (SESAM) was among the earliest types and the most popular SA used to passively generate Q-switching pulses in a fiber laser cavity

(S. X. Xu, Li, Hao, Zhai, & Zeng, 2008). However, they are costly and have a relatively small operation wavelength range. SESAMs dominance was suppressed with the introduction of carbon nanotubes (CNTs) (Y. Gao et al., 2013). CNT based SA offered significant advantages in terms of a simpler fabrication and easier packaging. Furthermore, the operating wavelength range in CNT is much wider than SESAM (A. Schmidt et al., 2008). However, CNT has restricted practical applications due to the dependence of response spectral range on the nanotube sizes. Graphene, on the other hand, (J. Zhao et al., 2012) and topological insulators such as bismuth telluride ( $\text{Bi}_2\text{Te}_3$ ) (Zakaria et al., 2018), bismuth selenide ( $\text{Bi}_2\text{Se}_3$ ) (H Ahmad, Salim, Azzuhri, Soltanian, & Harun, 2015) and antimony telluride ( $\text{Sb}_2\text{Te}_3$ ) (J Sotor, Sobon, Grodecki, & Abramski, 2014), have been successfully employed in various fiber lasers setup to produce Q-switching pulses. However, graphene has the drawbacks of wavelength-dependent, low modulation depth (between 0.5% and 1.8% per layer (Sobon, 2015)) as well as a small optical absorption range which pose a limitation in many applications, while TIs have complex fabrication processes that allow the flow of electrons on their surface (M. H. M. Ahmed et al., 2017). Black Phosphorous (BP) (L. Kong et al., 2016) and Transition Metal Dichalcogenides (TMD) materials such as Tungsten disulfide ( $\text{WS}_2$ ) (J. Lin, Hu, Chen, Gu, & Xu, 2015), Tungsten diselenide ( $\text{WSe}_2$ ) (B. Chen et al., 2015), Molybdenum diselenide ( $\text{MoSe}_2$ ) (RI Woodward et al., 2015), and Molybdenum Sulfide ( $\text{MoS}_2$ ) (S. Wang et al., 2014) have also gained many interests. They have good potential for pulse laser applications due to their thickness-dependent band gap and unique absorption properties (B. Chen et al., 2015; M Zhang et al., 2015). However, the process of fabricating these materials as SAs are slightly complicated.

To date, there are many new approaches in research to develop new high-performance SAs for generating a Q-switched laser operation (Hisyam, Rusdi, Latiff, & Harun, 2016). Finding a new material to be used as high-performance SAs for Q-switching pulse

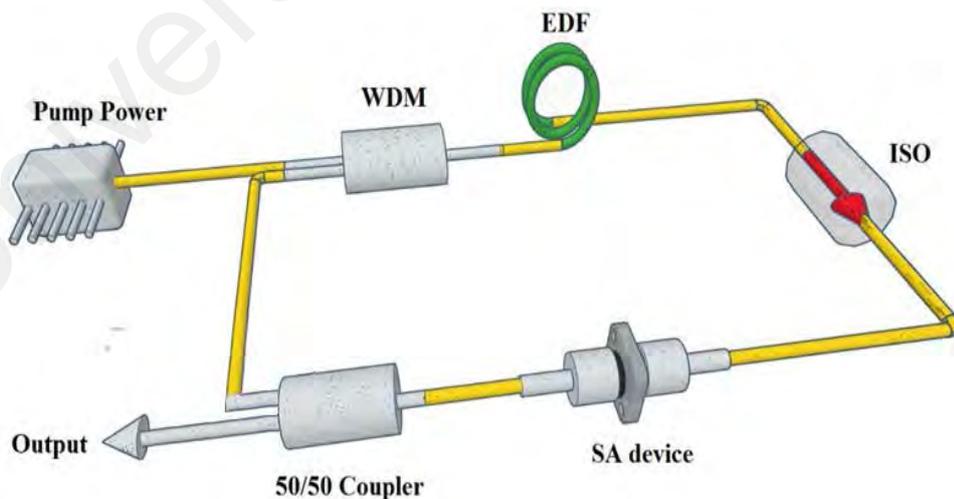
generation is of great interest. In that regards, organics materials are a promising candidate for developing new SAs, as they may have broad spectral tunability, large and ultrafast nonlinear response and they can be solution-processed at low cost. Additionally, organic materials would be highly desirable as a bio-compatible alternative for developing non-hazardous and environmentally-friendly photonic devices which could find increasing applications in biomedical laser processing and sensing. In comparison to traditional inorganic materials, organic materials have the advantages of versatile molecular design, simple fabrication, low-cost processing and easy control of physical properties of materials. Hence, organic materials have been utilized in different areas, which include solar cells (Peumans et al., 2011), bi-stable memory devices (Briseno et al., 2006), light-emitting diodes (OLEDs) (Holmes et al., 2003) and organic thin-film transistor (Choi, Jin, Park, Kim, & Gal, 2012).

In this chapter, various Q-switched Erbium-doped fiber lasers (EDFLs) are demonstrated using the newly developed organic material-based SA devices. Here, three organic materials; Bis[2-(4,6-difluorophenyl)pyridinato-C<sup>2</sup>,N](picolinato)iridium(III) (FIrpic), bis(8-hydroxyquinoline)zinc (Znq<sub>2</sub>) and Tris(8-hydroxyquinoline)aluminum (Alq<sub>3</sub>) are used for the generation of stable Q-switched laser pulses. A small piece of the developed SA thin-film was sandwiched between two ferrule connectors and incorporated into the laser setup. The Q-switching pulses were successfully generated as the pump power was increased above the threshold. The pulse width reduced, and the repetition rate increased as the pump power was further increased, which confirmed the Q-switching operation.

## 4.2 Q-switched Pulse Generation with FIrpc Based SA

### 4.2.1 Experimental Setup

The proposed setup of the fiber laser cavity is shown in Figure 4.1. A 980 nm laser diode (LD) was utilized to pump the laser cavity and it was integrated into the laser cavity via the 980 port of 980/1550 nm wavelength division multiplexer (WDM). The output port of the WDM was connected to an erbium-doped fiber (EDF) which works as a gain medium. The cavity length was about 8 m that is 5.5 m single-mode fiber (SMF-28), 2 m EDF and 0.5 m WDM. The EDF has an erbium ions absorption of 23 dB/m at 980 nm with numerical aperture, core and a cladding diameter of 0.16, 4  $\mu\text{m}$  and 125  $\mu\text{m}$ , respectively. The other end of EDF was connected to a polarization-independent isolator (ISO) to maintain unidirectional laser propagation before being connected to the SA device while the other end of the SA device was connected to a 50/50-output coupler (OC). The FIrpc thin-film SA was cut into  $1 \times 1 \text{ mm}^2$  and sandwiched between two fiber ferrules to form a SA device. An index matching gel was pre-applied with a very small quantity to hold the film between the fiber ferrules as well as to prevent spurious reflection.

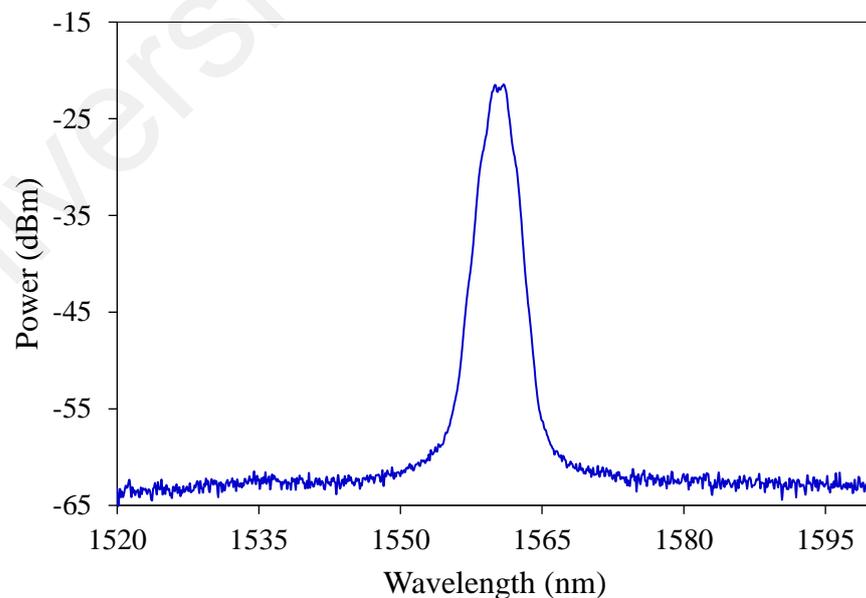


**Figure 4.1: Experimental setup of Q-switched EDFL with FIrpc thin-film SA in a ring cavity**

One of the 50% output coupler port was connected to the 1550 nm port of the WDM, thus completing the fiber laser cavity. The other 50% output of the OC was utilized for testing purposes. The output spectrum, temporal characteristic in the time domain, output power and radio frequency (RF) spectrum were measured by using an optical spectrum analyzer, 500 MHz oscilloscope through 7 GHz photodetector, optical power meter and RF spectrum analyzer, respectively.

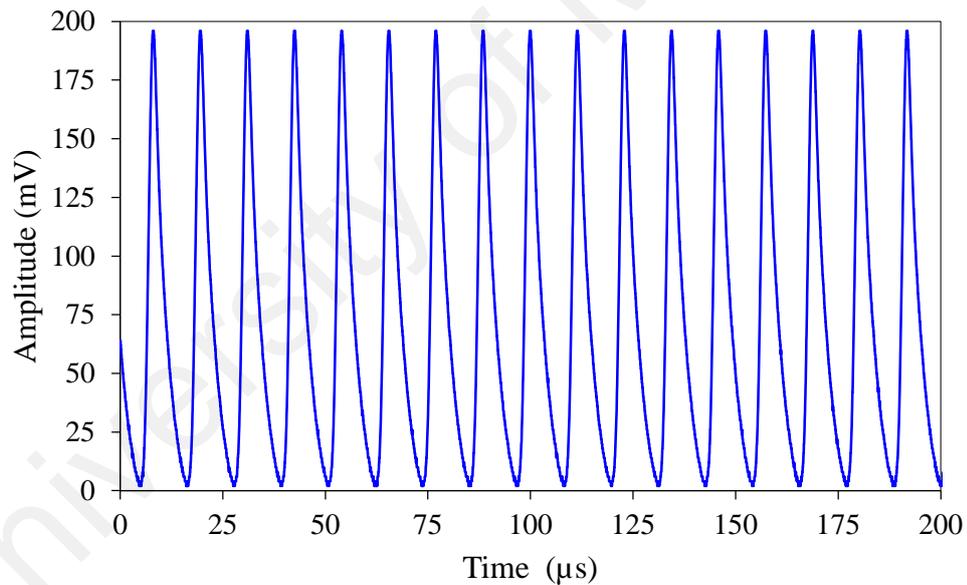
#### 4.2.2 Q-switching Performance

At the initial phase of the experiment, the laser operated in continuous wave (CW) regime as the pump power was increased above the threshold of 20 mW. A self-starting and stable passively Q-switched EDFL oscillation using FIpric was observed when the pump power exceeded the threshold at 30 mW. Figure 4.2 depicts the output spectrum of the Q-switching pulse in EDFL at the threshold pump power, which centers at a wavelength of 1560.4 nm with 2.2 nm 3-dB bandwidth. The Q-switching pulse train operation remained stable as the pump power was raised to 208 mW.



**Figure 4.2: Optical spectrum of FIpric based Q-switched EDFL**

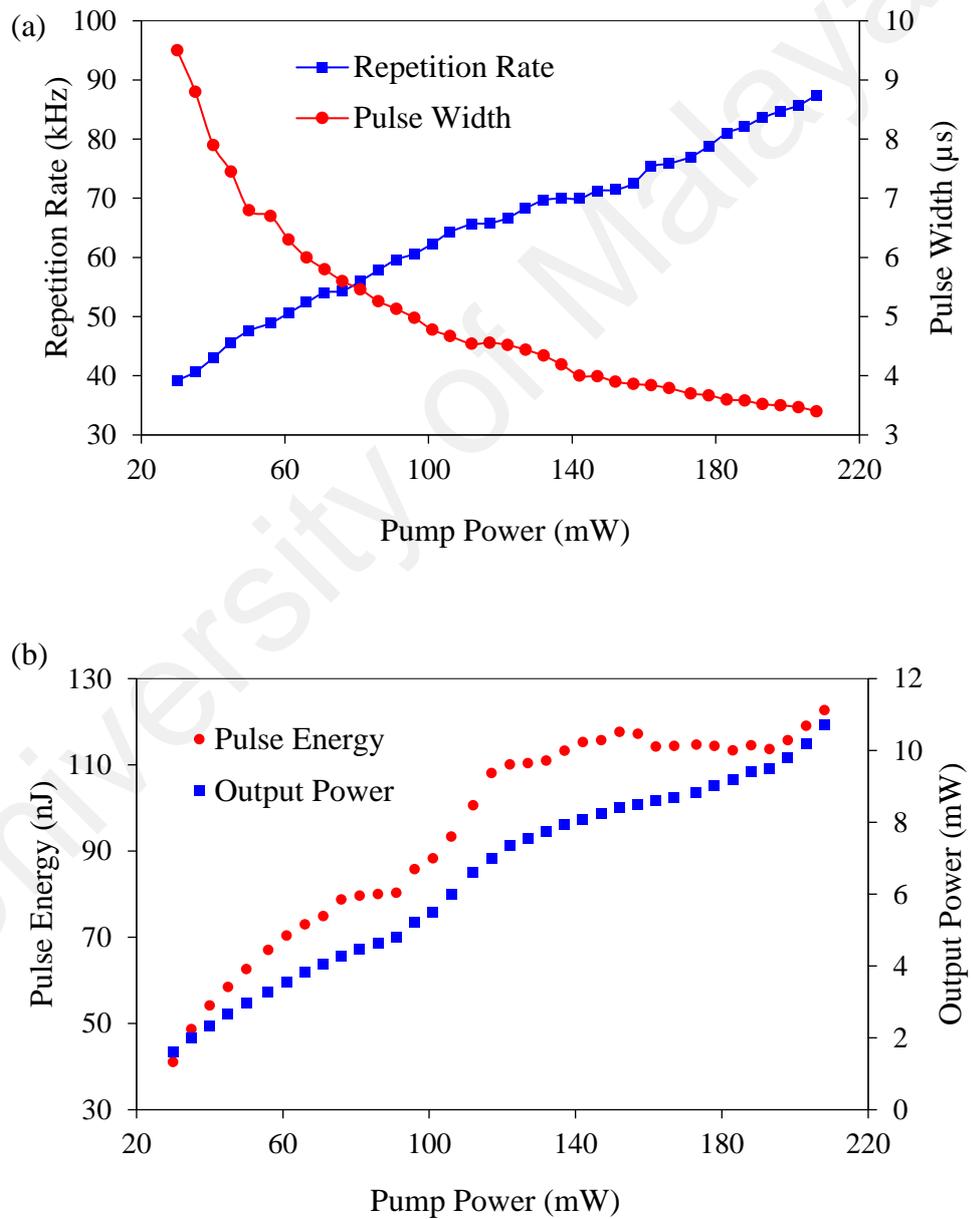
To prove that the FIrpic based SA triggered the Q-switched operation, the cavity was launched with no SA inside. In this case, no Q-switched operation was observed even when the incident pump power was tuned over a wide range. Figure 4.3 shows the oscilloscope pulse train at a pump power of 208 mW. The pulse train maintained a uniform intensity distribution without observable fluctuation, nor amplitude modulation in a laboratory condition for 2 hours. This confirms the stability of the regime and its capability to operate with various repetition rates and pulse widths. By increasing the pump power up to the maximum available input of 270 mW, the pulses became unstable as FIrpic SA was over-saturated without thermal damage. The thermal damage threshold of the SA is higher than 270 mW.



**Figure 4.3: Typical pulse train of the Q-switched EDFL at LD power of 208 mW**

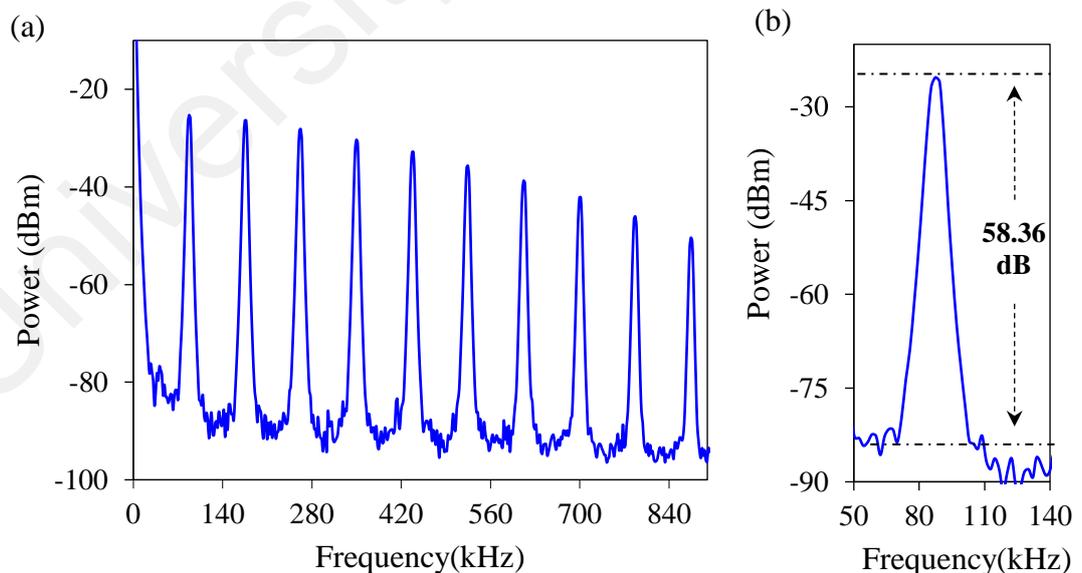
Figure 4.4 (a) shows a monotonic increment of repetition rate from 39.22 to 87.4 kHz as the pump power was increased from 30 to 208 mW. The pulse-width decreased from 9.5 to 3.4 μs at the same pump power range, which agrees to the typical characteristic of a Q-switched laser. The shortest pulse width of 3.4 μs is obtained at 208 mW pump power. This long pulse duration is attributed to the long cavity length (Dong, Hao, Hu, & Liaw,

2011; M. Wu, Chen, Zhang, & Wen, 2014). We expect that a short pulse duration and enhanced overall Q-switching operation would be achieved by shortening the cavity length, enhancing the cavity birefringence and reducing the cavity losses (Degnan, 1995; Herda, Kivistö, & Okhotnikov, 2008). Raising the incident pump power resulted in more gain feed to saturate the SA. Consequently, the threshold energy stored in the gain medium was reached earlier, leading to an increase in the repetition rate and a decrease in the pulse width.



**Figure 4.4: (a) Repetition rate and pulse width versus input pump power and (b) pulse energy and output power versus input pump power**

As the pump power was increased from 30 to 208 mW, the pulse energy increased from 41 to 122.6 nJ and the output power linearly increased from 1.61 to 10.72 mW with an efficiency of 4.89%, as shown in Figure 4.4 (b). To check the stability of the Q-switching operation, we measured the RF of the output spectrum with a span of 900 kHz. Figure 4.5 (a) and (b) shows the fundamental RF peaks at 87.4 kHz with a span of 900 kHz and 90 kHz, respectively. The signal-to-noise ratio (SNR) is 58.36 dB at the fundamental frequency, which depicts excellent Q-switching stability (H. Ahmad, Zulkifli, Thambiratnam, & Harun, 2013). In comparison with other works that addressed a similar cavity setup with SAs like CNT, Bi<sub>2</sub>Se<sub>3</sub>, BP, MoSe<sub>2</sub> and TiO<sub>2</sub> in ref (Harith Ahmad et al., 2017; Harith Ahmad, Siti Aisyah Reduan, et al., 2016; M. H. M. Ahmed, A. H. H. Al-Masoodi, A. A. Latiff, H. Arof, & S. W. J. I. J. o. P. Harun, 2017; HH Liu, Chow, Yamashita, Set, & Technology, 2013; L. Sun et al., 2014), respectively, Firpic showed a very high SNR, a very high output power, high pulse energy and wide input pump power range (i.e. from 30 mW to 208 mW).



**Figure 4.5: (a) RF spectrum of Q-switched EDFL at a pump power of 208 mW with a span of 900 kHz and (b) with a span of 90 kHz**

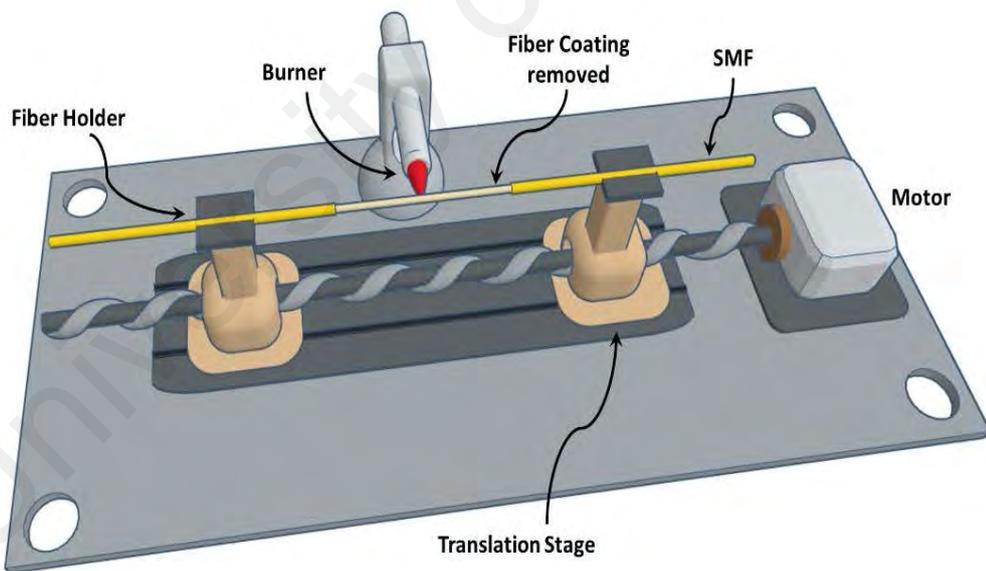
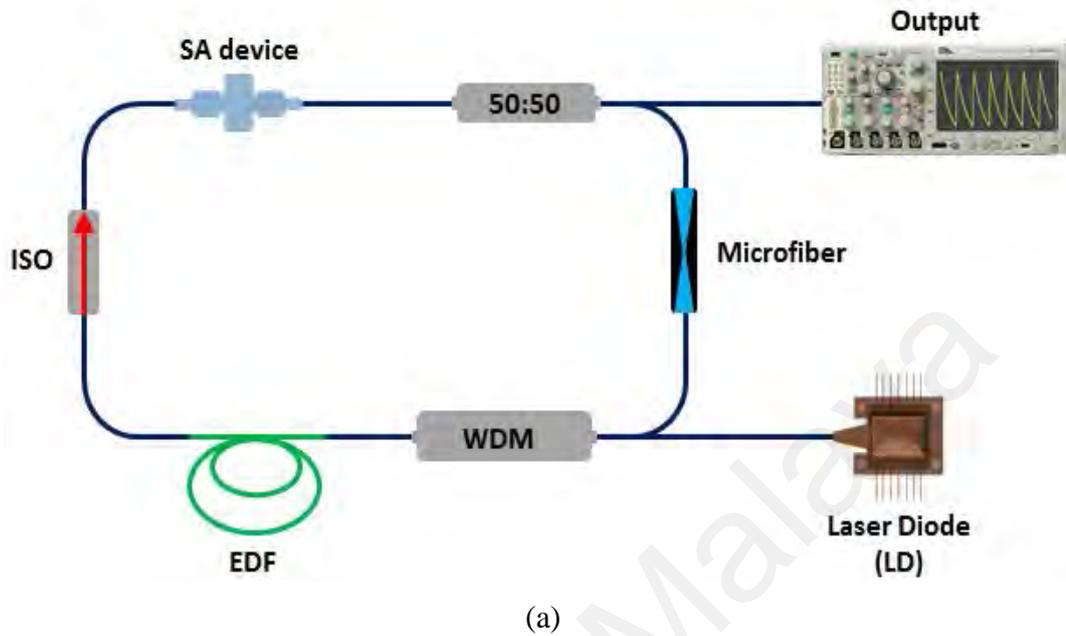
### 4.3 Dual-Wavelength Q-switched EDFL with Microfiber Filter and FIRpic SA

#### 4.3.1 Experimental Arrangement

The proposed setup of the dual-wavelength Q-switched EDFL cavity is shown in Figure 4.6 (a). A 2 m long EDF was used as a gain medium, it has an erbium ions absorption of 23 dB/m at 980 nm  $\text{ps}^2/\text{km}$ . A laser diode (LD) operating at 980 nm was used to pump the EDF via the 980/1550 WDM. The EDF was also connected to the isolator (ISO) device, which was used to prevent light reflection and thus allow a unidirectional operation of the laser. The cavity length was about 8 m that is 5.5 m single-mode fiber (SMF-28), 2 m EDF and 0.5 m WDM. The SA device was made by inserting 1  $\text{mm}^2$  piece of FIRpic thin-film between two fiber ferrules. A non-adiabatic microfiber was used in the cavity to produce dual-wavelength output. Finally, 3-dB output coupler was used to extract 50% of the light from the cavity. The output spectrum, pulse train, and RF spectrum were measured by using OSA, 500 MHz oscilloscope with 7 GHz photodetector, and RF spectrum analyzer, respectively.

A flame brushing technique fabricated the microfiber, see Figure 4.6 (b). A coating-removed SMF was held horizontally by two motorized fiber holders. The fiber holders have a sliding section in which they can be pulled apart. During the tapering process, an oxy-butane torch flame was used to heat the fiber to its softening temperature, and at the same time, the ends of the SMF were pulled apart to reduce the diameter. The torch had 10 mm long flame, and it was perpendicular and had a constant distance (a pinpoint tip) to the clamped fiber. To ensure an optimal flame performance, we carefully controlled the oxygen gas supply pressure. The temperature and flame level are vital parameters to manage to produce the non-adiabatic microfiber (Harith Ahmad, Rahman, Sakeh, Razak, & Zulkifli, 2016; B. Wang, Zhang, Pang, Wang, & Yang, 2011; Zheng et al., 2013). The insertion loss and the inter-modal interference of the fiber were both monitored during the fabrication process by launching amplified spontaneous emission (ASE) in one end

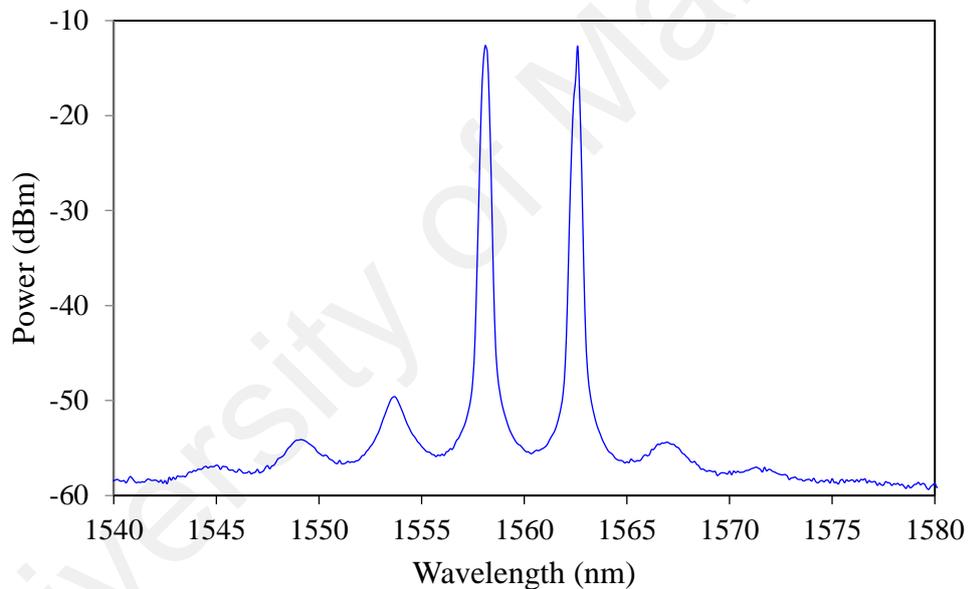
and observing the output by OSA on the other end of the SMF. The produced microfiber had a diameter of  $8\ \mu\text{m}$  and a tapering length of 11 mm.



**Figure 4.6 : (a) Cavity setup for dual-wavelength Q-switched EDFL and (b) experimental setup for the fabrication process of microfiber via the flame brushing technique.**

### 4.3.2 Dual-Wavelength Q-switched EDFL Performance

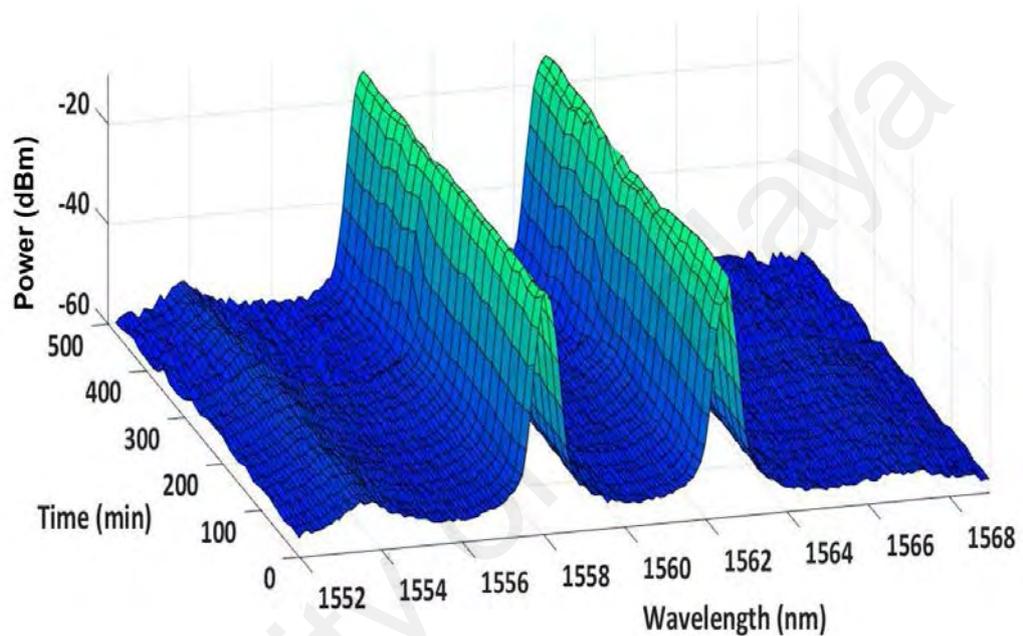
In the proposed laser, the FIRpic based SA was used as a Q-switcher to transform the CW operation into a pulse train, while the microfiber was used to generate the dual-wavelength output. The laser cavity starts to generate a CW laser at a threshold pump power of 25 mW. A stable dual-wavelength Q-switch (DWQS) pulse train was obtained as the pump power was further increased within 35 mW to 213 mW. Figure 4.7 shows the dual-wavelength output spectrum at 213 mW, the peaks were located at 1558.1 nm and 1562.6 nm giving a spacing of about 4.5 nm with almost the same intensity of 12.61 dBm and 12.69 dBm and 3-dB bandwidth of 0.3 nm and 0.1 nm, respectively.



**Figure 4.7: Optical spectrum of the DWQS laser**

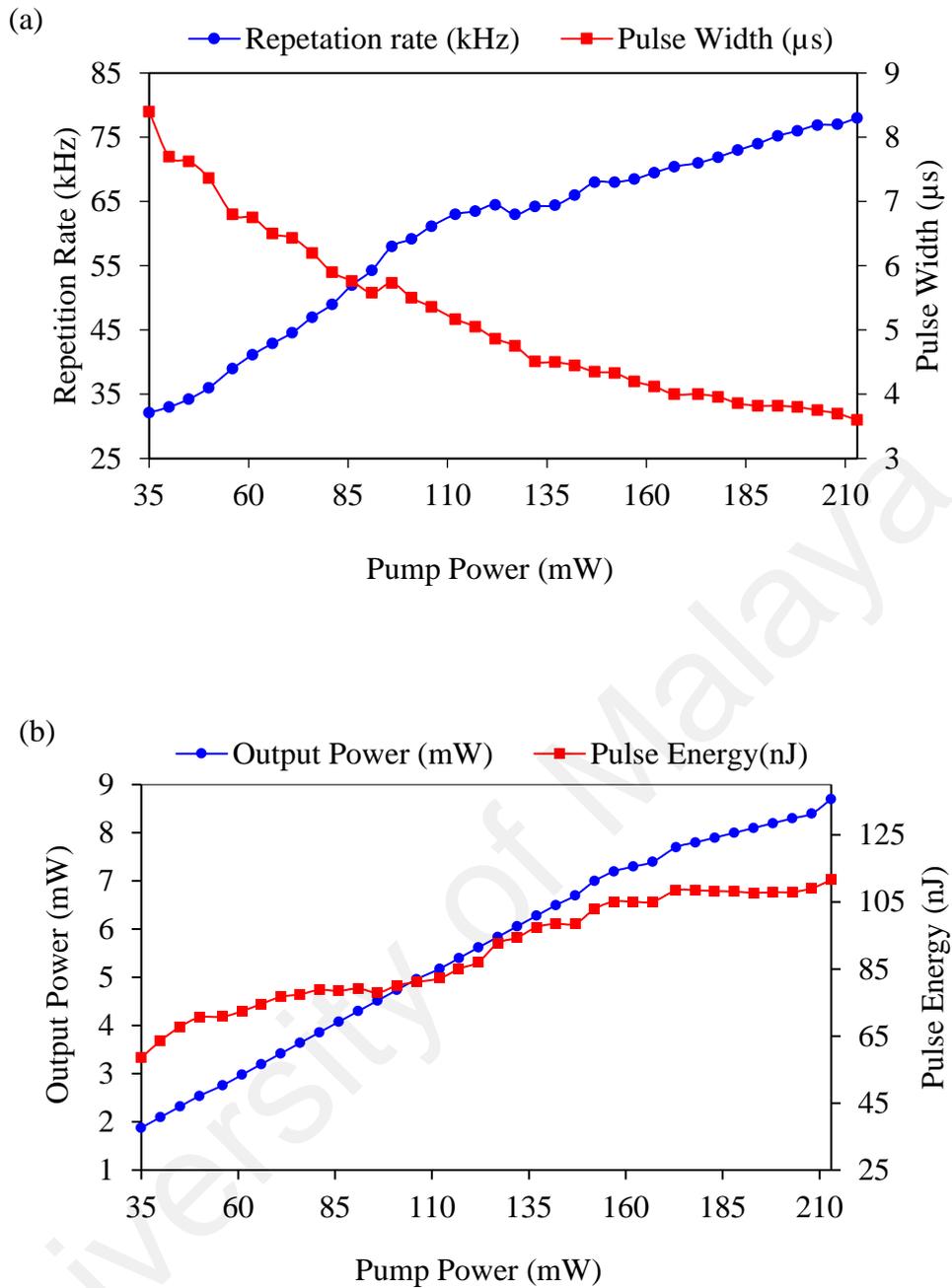
The produced dual-wavelength output was stable throughout the experiment. To check the stability of the proposed laser, the spectrum was sampled every ~25 minutes for the total duration of 500 minutes. The time-based output spectrum is given in Figure 4.8. From this figure, the DWQS is highly stable and provided excellent performance, in which the peaks maintained the same wavelengths and intensities with only minimal fluctuations. Changing the pump power did not affect the obtained spectrum. To make

sure that both peaks have the same pulse width, repetition rate and SNR, we used a tunable bandpass filter (TBF) at the output of the DWQS laser. During the experiment, the TBF was tuned to 1558.1 nm and 1562.6 nm and it was found that there was a negligible difference between the two peaks. However, the TBF was then set to 1562.6 when the data was collected.



**Figure 4.8: Output laser spectrum sampled for 500 minutes**

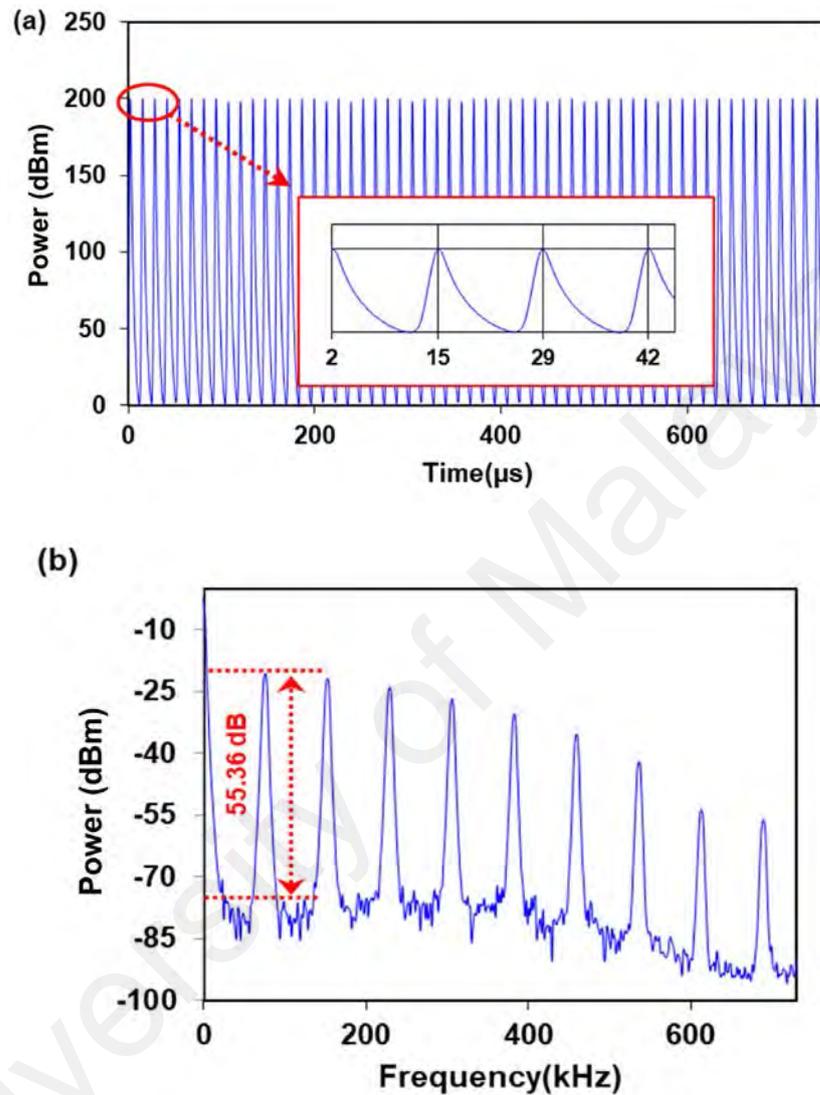
The pulsing was produced as the LD power was increased from 35 mW to 213 mW. The pulse width was decreased from 8.4  $\mu\text{s}$  to 3.6  $\mu\text{s}$  as the input power increased, see Figure 4.9 (a). While, the repetition rate, output power and pulse energy were correlated linearly with the input LD power, they increased from 32.15 kHz to 78 kHz, 1.8 mW to 8.7 mW and 58.48 nJ to 110.13 nJ, respectively, see Figure 4.9 (b). Both the repetition rate and the pulse duration behave as expected, the pulse duration reduced in an almost exponential manner, while the pulse repetition rate increased linearly to the rising LD power.



**Figure 4.9: LD power vs (a) Repetition rate and pulse width and (b) output power and pulse energy**

Figure 4.10 (a) shows the output pulse train at the maximum input LD power, the pulse train was very stable with no modulation or instabilities, which indicates a stable performance. The inset Figure shows the pulse period of about  $\sim 12.8 \mu\text{s}$  which is coincident with the fundamental frequency observed on the RF spectrum. The RF spectrum of DWQS laser with span and resolution bandwidth of 300 kHz and 350 kHz,

respectively, is given in Figure 4.10 (b). The fundamental frequency has an SNR of 55.36 dB, which reveals the excellent stability of the system.



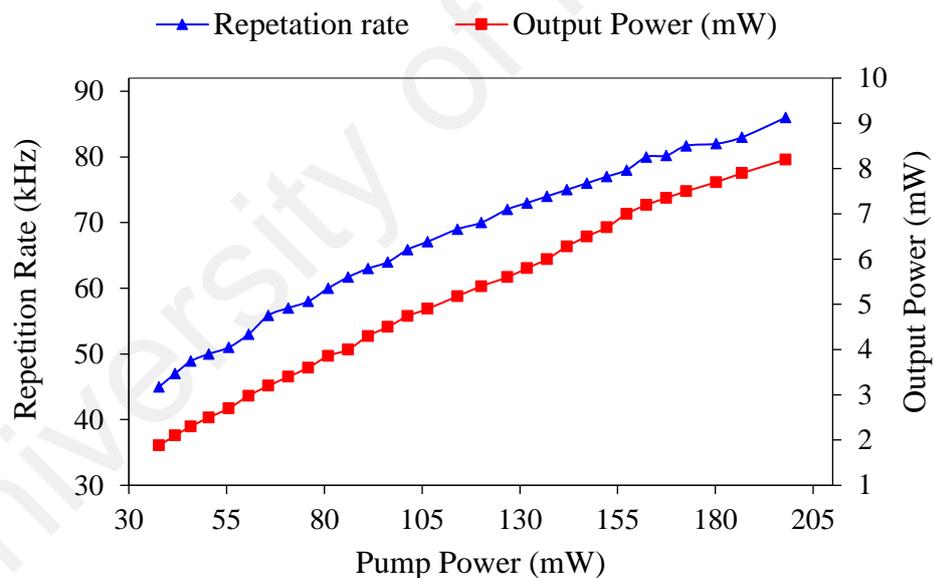
**Figure 4.10: (a) Pulse train at maximum input LD power and (b) RF spectrum with a span of 700 kHz.**

#### 4.4 Q-switched Pulse Generation by Using $\text{Znq}_2$ as SA

##### 4.4.1 Fixed Wavelength Operation

The setup used in this experiment is similar to the previous Q-switched laser based on FIrpic SA as described in section 4.2.1 (Figure 4.1). However, the cavity length of the proposed new laser was slightly shorter, about 6 m comprises of 3.5 m long SMF-28, 2

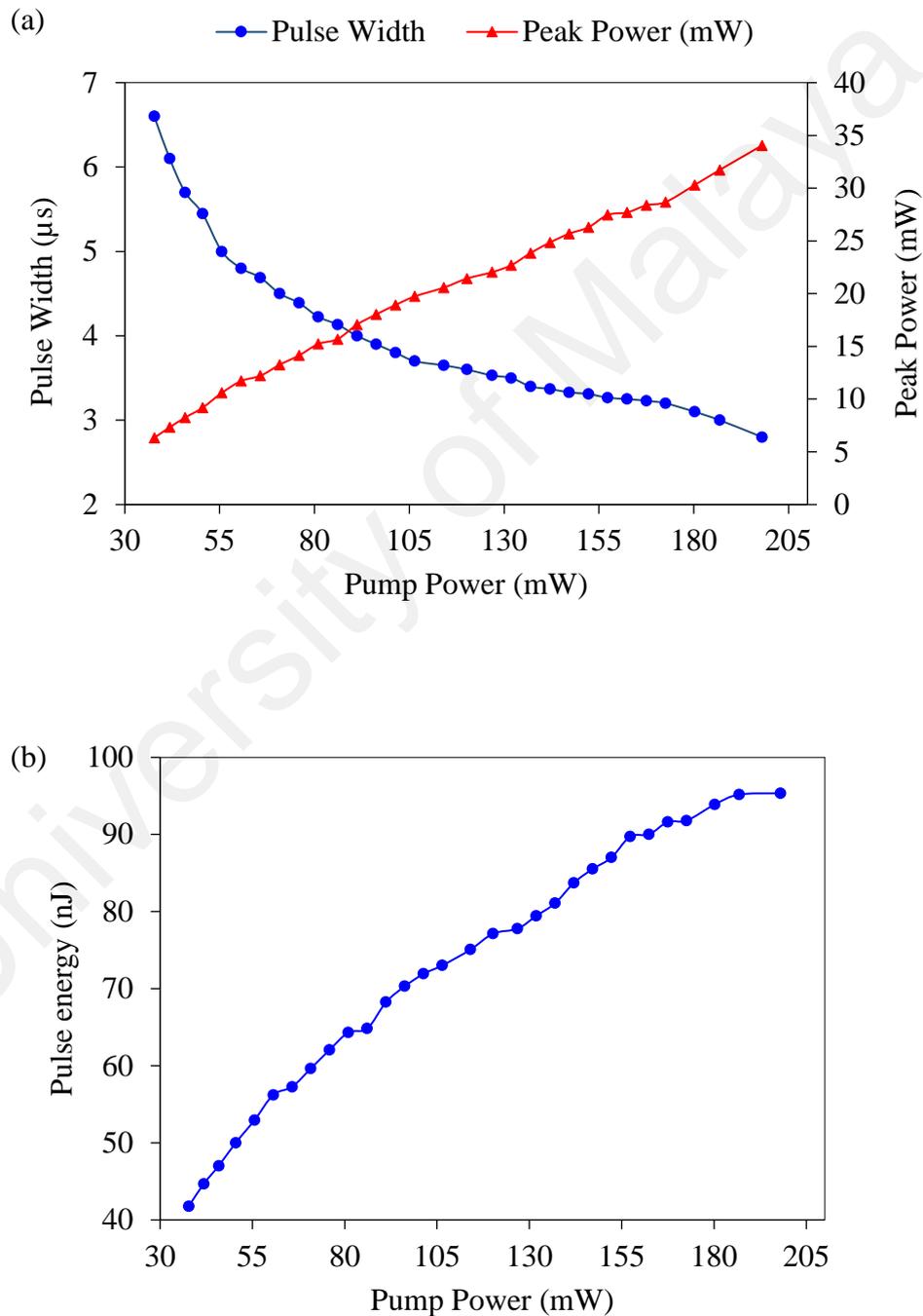
m long EDF and 0.5 m long WDM fiber. The FIRpic SA device was replaced with Znq<sub>2</sub> SA, which was prepared by a similar fashion using the fabricated Znq<sub>2</sub> thin-film. At first, the laser operation was investigated without the incorporation of the SA thin-film in the cavity. As the pump power was gradually increased to reach a threshold of 30 mW, CW laser was generated at a peak wavelength of ~1560 nm. No pulsing operation was observed as the LD power was tuned up to 350 mW. As the SA thin-film was incorporated into the cavity, a stable pulsing operation was observed at LD power was varied from the threshold of 38 mW to 198 mW. At a pump power of 38 mW, the repetition rate was 45 kHz and it increased linearly to 86 kHz as the pump power was increased to 198 mW, see Figure 4.11. The Figure also shows that the average output power increased linearly 1.9 mW to 8.2 mW.



**Figure 4.11: Output power and repetition rate as a function of pump power**

The produced pulse width was decreased from 6.6  $\mu$ s to 2.8  $\mu$ s as the LD power increased, as shown in Figure 4.12 (a). The behaviors of repetition rate and pulse width with the LD power are the typical characteristic of the Q-switching operation. The more power delivered by the LD, the shorter time for the SA to be saturated. This reduces the

rising and falling times of pulses, and hence reduce the pulse period and pulse duration. On the other hand, the peak power increased linearly from 41.8 mW to 96.5 mW. The linear increment of pulse energy and peak power indicates a stable performance of the laser operation. Figure 4.12 (b) shows the pulse energy increasing almost linearly from 41.78 nJ to 95.35 nJ, that indicates a stable performance of the Q-switched laser.



**Figure 4.12: (a) Peak power and pulse width as a function of pump power and (b) pulse energy as a function of pump power**

Figure 4.13 (a) shows the output optical spectrum of the laser, the spectrum is centred at 1560 nm with 3-dB bandwidth of 1.5 nm. Figure 4.13 (b) shows the output pulse train at the maximum LD power. The pulse train is very stable and uniform with no instabilities or modulation. All the pulses had an intensity of 264 mV ( $\pm 2$  mV). The inset Figure shows the pulse train period of  $\sim 11.55 \mu\text{s}$  for a duration of 40  $\mu\text{s}$ .

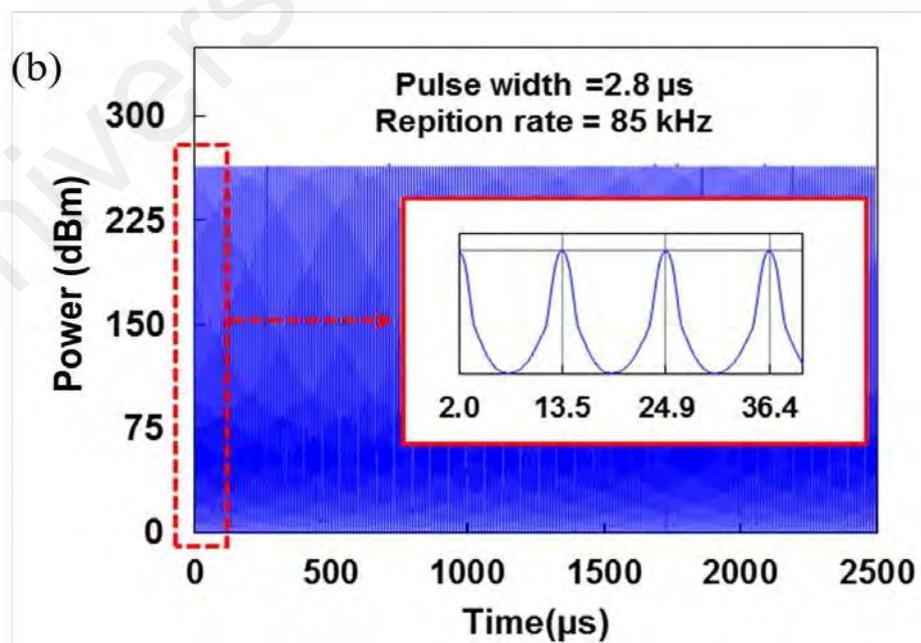
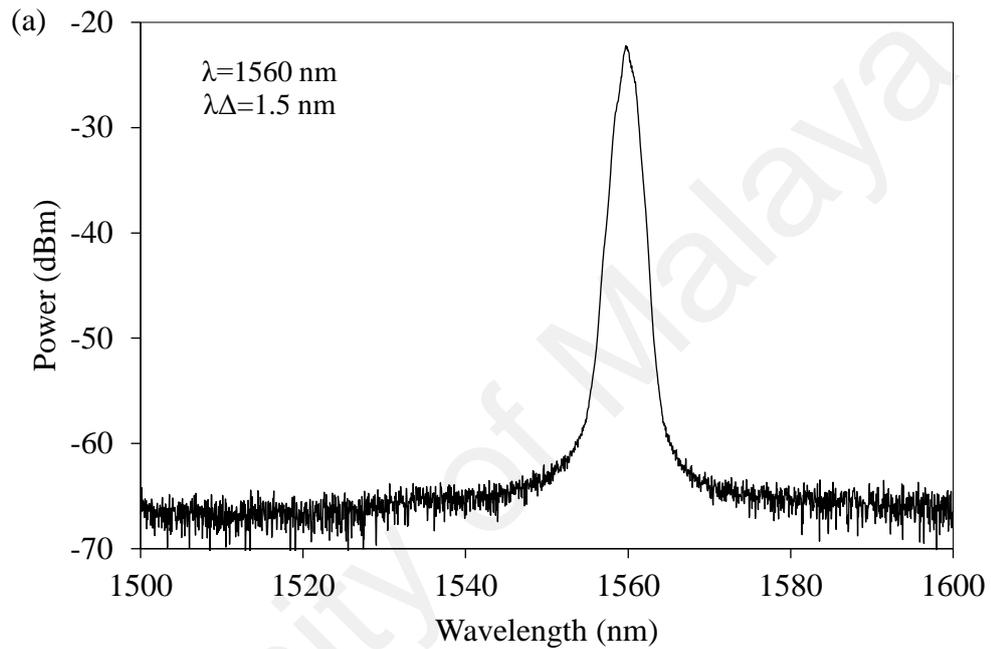
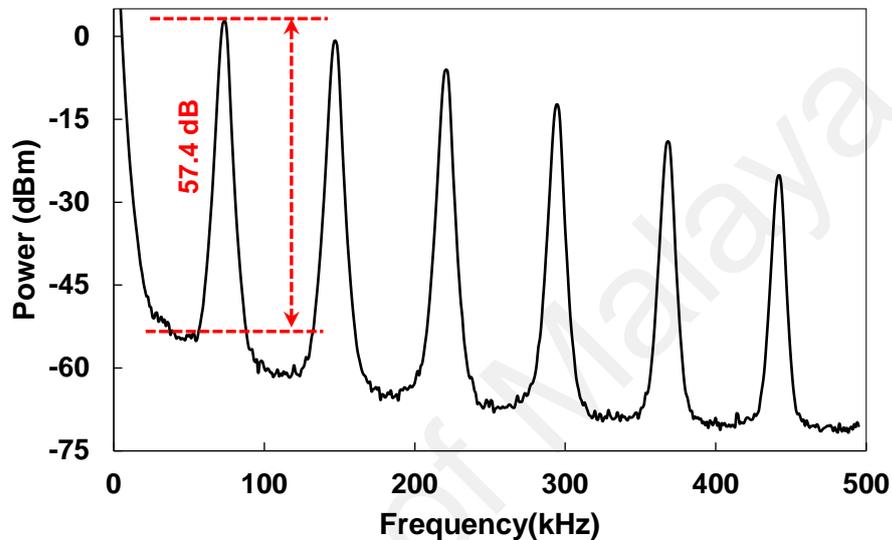


Figure 4.13: (a) Optical spectrum of the Znq<sub>2</sub> based Q-switched EDFL and (b) typical pulse train of the Znq<sub>2</sub> based Q-switched EDFL, Inset shows the pulse period

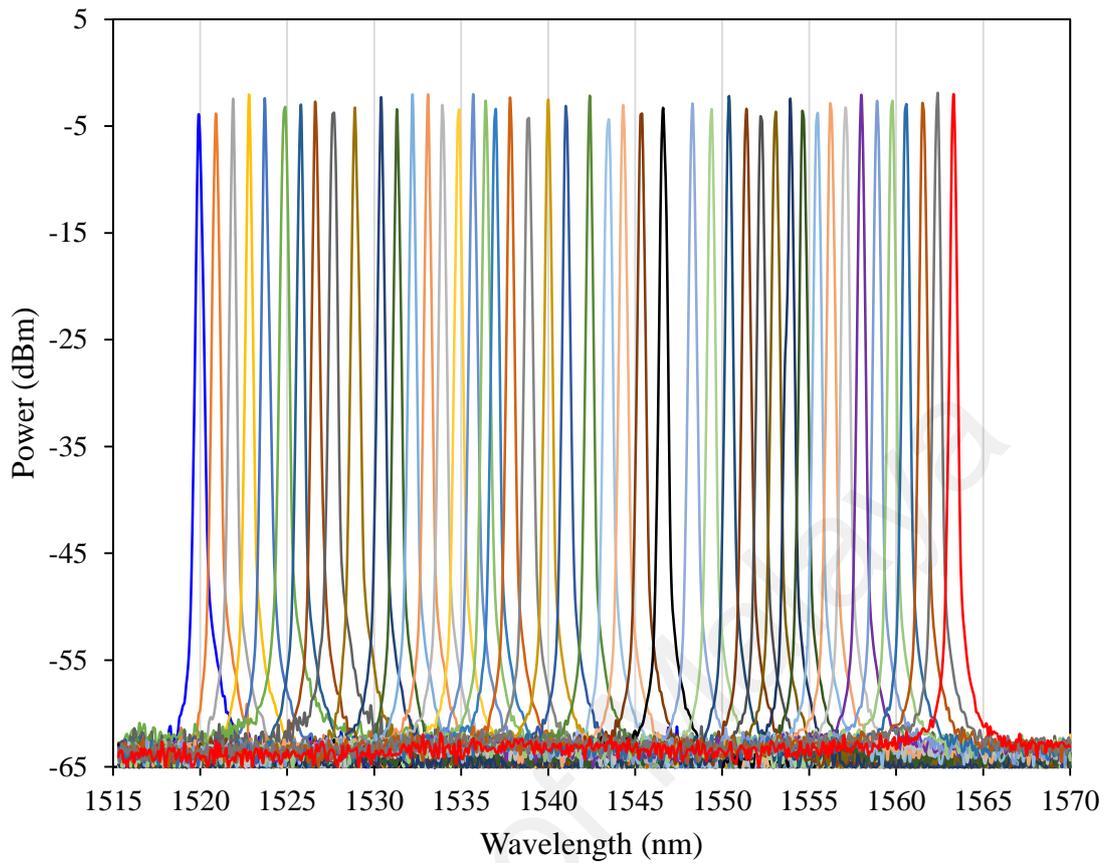
The RF spectrum with a span of 500 kHz is given in Figure 4.14. The figure shows the fundamental frequency is located at 86 kHz with an SNR of 57.4 dB. This high SNR further indicates a stable and good performing Q-switching operation. We believe that the performance of the laser operation can be enhanced given that cavity parameters are enhanced by shortening the cavity length or employing an EDF with a higher gain.



**Figure 4.14: RF spectrum showing the SNR of 57.4 dB**

#### 4.4.2 Tunable Wavelength Operation

In this experiment, we used the same cavity as the one in the previous experiment, except that a TBF was incorporated into the cavity. The TBF was inserted between OC and WDM. Through tuning the TBF at the maximum input pump power, the central wavelength was shifted continuously from 1520.0 nm to 1563.3 nm, giving a range of 43.3 nm and covering almost all C-band region. The TBF allows a minimum increment of 0.1 nm, as it is the smallest granularity provided by the micrometer head of the TBF. Figure 4.15 shows the output spectra of the laser, which was tuned at the step of  $\sim 1$  nm (to ensure clarity) when the LD power was fixed at 206 mW. The peak power of the spectra was maintained at almost the same level of  $-3.23 \text{ dBm} \pm 1.2 \text{ dBm}$  with the same pattern and shape, which suggests that the pulsing operation was very stable.

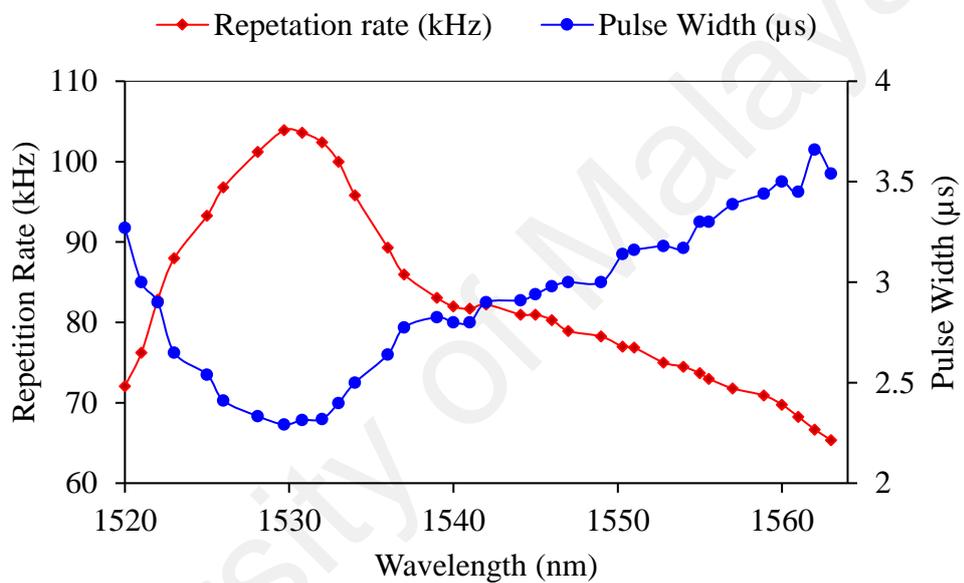


— 1520.00	— 1520.90	— 1521.90	— 1522.80	— 1523.70
— 1524.85	— 1525.80	— 1526.60	— 1527.65	— 1528.50
— 1530.40	— 1531.30	— 1532.20	— 1533.10	— 1533.95
— 1534.85	— 1535.70	— 1536.40	— 1536.60	— 1537.80
— 1538.85	— 1540.00	— 1541.00	— 1542.40	— 1543.45
— 1544.30	— 1545.35	— 1546.40	— 1548.30	— 1549.35
— 1550.40	— 1551.40	— 1552.00	— 1553.00	— 1554.00
— 1554.65	— 1555.25	— 1556.25	— 1557.00	— 1558.00
— 1558.90	— 1559.75	— 1560.55	— 1561.55	— 1562.30
— 1563.30				

**Figure 4.15: Output spectra of the tunable laser at different wavelength operation between 1520.0 nm and 1563.3 nm**

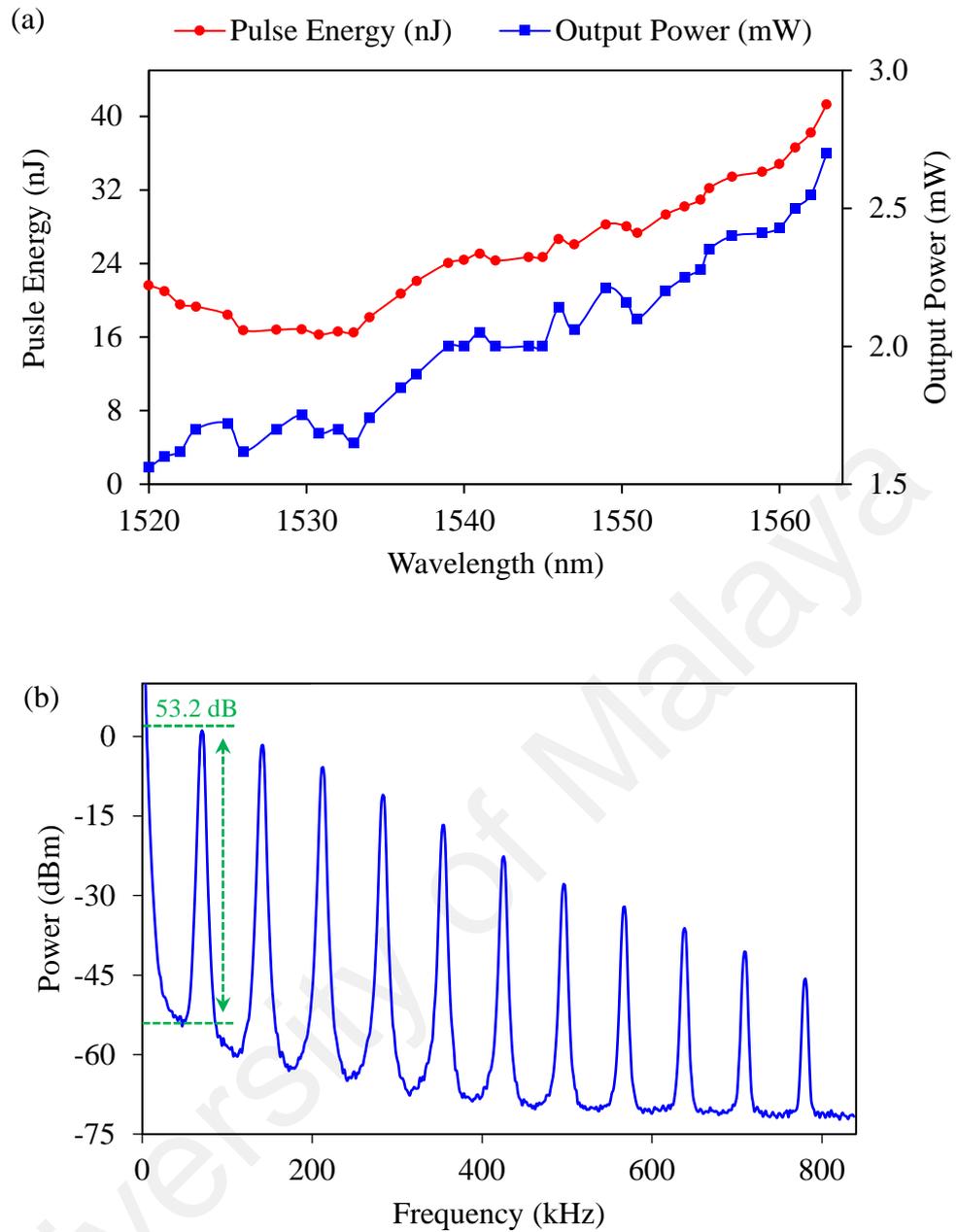
Figure 4.16 illustrates the effect of tunable wavelength operation on the pulse duration and pulse repetition rate. The pulses produced were wavelength dependent. As the TBF was shifted from 1520.0 nm to 1529.65 nm, the pulse duration shortened from 3  $\mu$ s to 2.12  $\mu$ s. Then it was increased gradually to 3  $\mu$ s again as the TBF was tuned to 1563.3 nm. The pulse repetition rate (which behaved inversely to the pulse width) reached its

highest value of 111.6 kHz at 1529.65 nm. This is attributed to the EDF gain is much higher at 1529.65 nm than other wavelengths. A higher gain will result in a shorter time for the SA to be saturated, which will lead to shortening the pulse width and increasing the pulse repetition rate. The effects of wavelength tuning were also investigated on the pulse energy and average output power, see Figure 4.17 (a). The figure shows that the average output power decreased as the operating wavelength shifted to a shorter wavelength even though the LD is kept at the same power of 206 mW.



**Figure 4.16: Pulse repetition rate and pulse width duration vs wavelength**

This is due to the loss of TBF, which is higher at shorter wavelength region. The pulse energy, as it is inversely correlated with the repetition rate, increased from 15 nJ at 1530.4 nm to 37.3 nJ at 1563.3 nm. Figure 4.17 (b) shows the output RF spectrum at a span of 810 kHz at maximum LD power and as the TBF was tuned to 1551.4 nm. Anritsu-MS2683A device recorded the data with a resolution bandwidth (RBW) of 300 Hz. The figure reveals a high SNR value of 53.2 dB.



**Figure 4.17: (a) Output power and pulse energy at different operating wavelengths and (b) RF spectrum with a span of 840 kHz**

It should be noted that recording the long-term operation for a single wavelength will not reflect the performance at other wavelengths. Additionally, the TBF will filter any distortion or fluctuation that output spectrum might have. It should be noted that this experiment took around 4 hours to record all the required data. During the experiment, the laser-produced the same performance in which the output pulse train and SNR were

both very stable. The wider pulse width obtained in this experiment in comparison with the previous work is due to the length and the insertion loss induced to the cavity by the TBF. It is believed that a shorter pulse width can be achieved by using a shorter cavity length of less than 1 meter. By further increasing the LD power up to 350 mW, the output pulse trains showed some fluctuation, then disappeared. This is due to the SA was fully saturated. The pulsing operation was obtained again as the LD power reduced below 206 mW. We concluded that the optical damage threshold value is above 350 mW as if the SA was damaged, no Q-switching operation can be produced again. Additionally, we did not observe any deformation of the SA during and after the experiment.

This experiment demonstrates that Znq<sub>2</sub> could operate as a broadband passive Q-switcher and showed that the Q-switching pulsing was easily tuned to a different wavelength, which is the goal of this work. Broadband absorption of the SA is the key feature to obtain a widely-tunable fiber laser (Y. Huang et al., 2014b). Znq<sub>2</sub> is a promising broadband SA as it showed very comparable performance with other SAs such as SnO<sub>2</sub>, TiO<sub>2</sub>, MoS<sub>2</sub> and MWCNT reported in (H. Ahmad, Hassan, Ahmad, Zulkifli, & Harun, 2016; H. Ahmad et al., 2019; Y. Huang et al., 2014a; Siddiq, Chong, Pramono, Muntini, & Ahmad, 2019) in terms of the wavelength tuning range, SNR, pulse energy, and pulse width. This experiment is suitable for practical applications that require a wide tunable wavelength range with highly stable performance such as environmental sensing, biomedical diagnostics and metrology. The tuning range in this experiment was limited by the TBF and not by SA. Thus, we believe that a wider tuning range can be developed given that a TBF with a wider tuning range is addressed. Additionally, higher pulse energy and higher average output power can be developed by addressing a high-gain fiber (Z. Luo et al., 2014), and enhancing the cavity parameters (Spühler et al., 1999). It should be mentioned that by removing the TBF and adding 52 m SMF a mode-lock operation was observed with pulse-duration and repetition rate of 1.6 ps and 1.8 MHz, respectively.

## 4.5 Q-switched Fiber Laser with Alq<sub>3</sub> SA

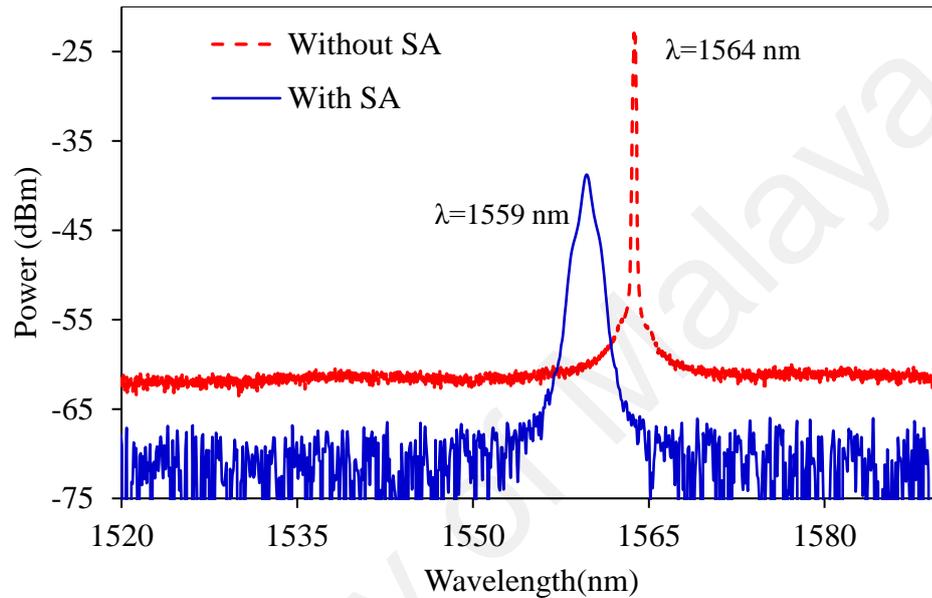
### 4.5.1 1.5 $\mu\text{m}$ Operation

In this subsection, a Q-switched EDFL is demonstrated by using the newly developed Alq<sub>3</sub> PVA thin-film as a SA. The SA device was constructed by sandwiching a piece of  $\sim 1 \times 1 \text{ mm}^2$  of the prepared Alq<sub>3</sub> thin-film in between two optical fiber ferrules. The insertion loss of the SA was measured to be around 0.8 dB. The SA device was incorporated in an EDFL cavity. The cavity length was estimated to be approximately 6 m. At first, the spectrum of the EDFL was measured (before inserting the SA device into the cavity) and CW operation was obtained at a threshold pump power of 15 mW.

As the Alq<sub>3</sub>:PVA SA was incorporated into the cavity, the Q-switching pulses train was observed at the threshold pump power of 20 mW. The pulses train was held stable and robust as the pump power was raised to the maximum input pump power of 122 mW. This low threshold pump power of Q-switched pulse is attributed to a low intra-cavity loss of the Alq<sub>3</sub>:PVA thin-film based SA due to the use of high gain fiber and optimal feedback from the output optical coupler.

Figure 4.18 illustrates the optical spectrum output of the CW and the proposed Q-switched EDFL based on Alq<sub>3</sub>:PVA SA at an input pump power of 20 mW. The laser operation with Alq<sub>3</sub>:PVA SA was obtained at a shorter wavelength regime compared to that of the CW laser without SA. The insertion of the SA increased the cavity loss and thus the lasing wavelength was shifted to a shorter wavelength to acquire more gain to compensate for the loss. As depicted in Figure 4.18, the spectrum is centred at a wavelength of 1564 nm and 1559 nm with 3-dB bandwidth of 0.22 nm and 1 nm for CW and Q-switching pulse, respectively. The spectral broadening of the Q-switched laser was observed because of self-phase modulation inside the cavity. Alq<sub>3</sub> has a band gap of 2.5 eV - 2.7 eV, which corresponds to wavelengths in the visible range. Due to quantum defects in the material, the band gap may reduce and thus the material can function as a Q-

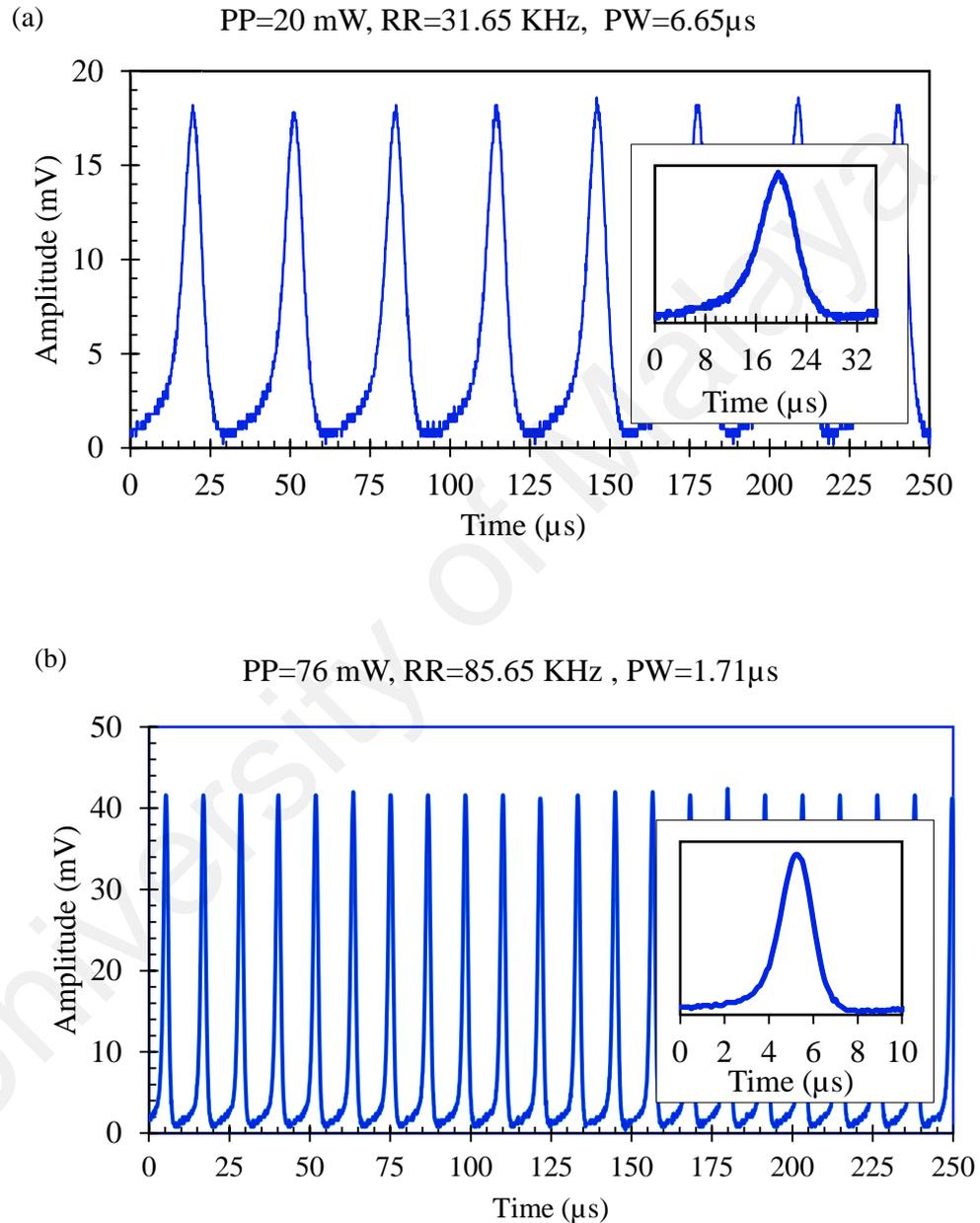
switcher in the C-band wavelength region. This is due to the absorption characteristic, which depends on the crystal form and particle size, as explained in ref. (Sato, Nakashima, Kamikura, & Yamamoto, 2008). Figure 4.19 (a), (b) and (c) show the typical pulse trains at incident pump powers of 20, 76, and 122 mW, respectively. Inset figures show a single pulse profile at each input pump power.



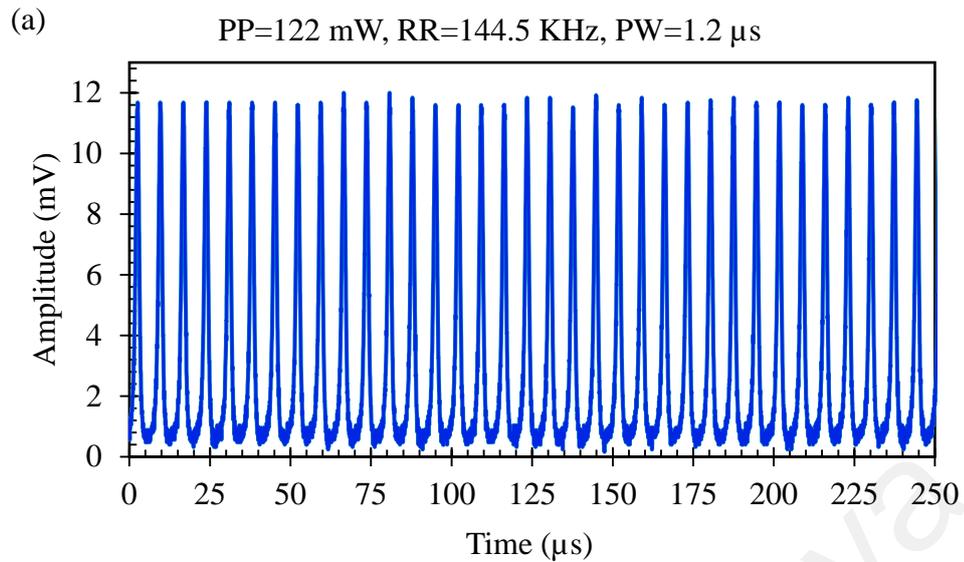
**Figure 4.18: The output spectra of the fiber laser cavity with and without Alq<sub>3</sub>:PVA SA for Q-switched and CW, respectively, at 20 mW pump power**

At pump power threshold of 20 mW, the Q-switched laser operates at a repetition rate and a pulse width of 31.65 kHz and 6.65  $\mu$ s, respectively, as shown in Figure 4.19 (a). By increasing the pump power to 76 mW, the pulse repetition rate was increased to 85.65 kHz, while the pulse width was reduced to 1.71  $\mu$ s (see Figure 4.19 (b)). The maximum repetition rate of 144.5 kHz and the shortest pulse width of 1.2  $\mu$ s were achieved at the maximum pump power of 122 mW as shown in Figure 4.19 (c). It is found that the pulse width shrinks and the repetition rate increases as the pump power increase. The Q-switching pulse trains are stable and have a uniform distribution with no fluctuation, which indicates that the fiber laser can operate in stable Q-switched operation with variable repetition rates. However, as the input pump power is raised above 122 mW, an amplitude

fluctuation was observed, and the pulse train became unstable, and finally, the Q-switching pulses train disappeared. That might be attributed to SA being bleached under high input pump power. Similar behaviour has been reported with different kinds of SAs (H. Ahmad et al., 2017; Q. Wang et al., 2015).

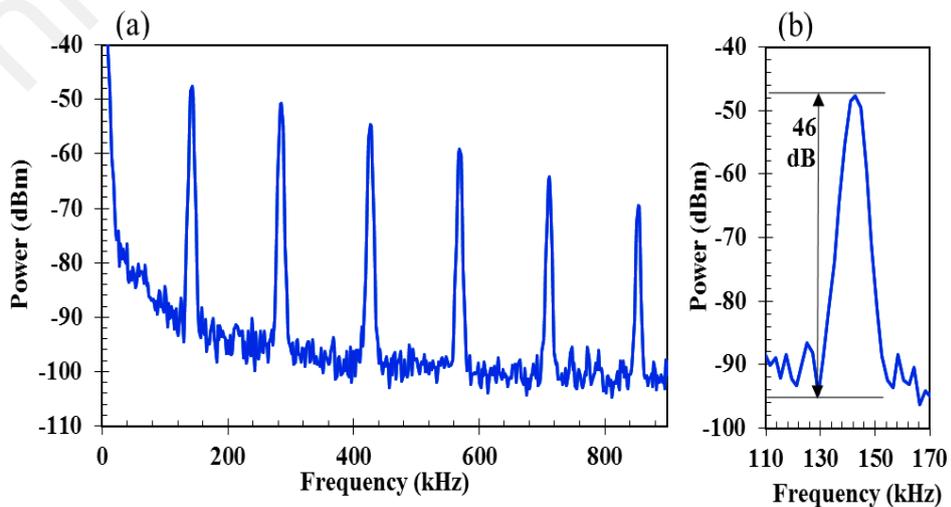


**Figure 4.19: Q-switched output pulse train with inserting of single envelope pulse in EDFL using Alq<sub>3</sub> SA at incident pump powers of (a) 20 mW, (b) 76 mW, and (c) 122 mW. PP: pump power, RR: repetition rate and PW: pulse width**



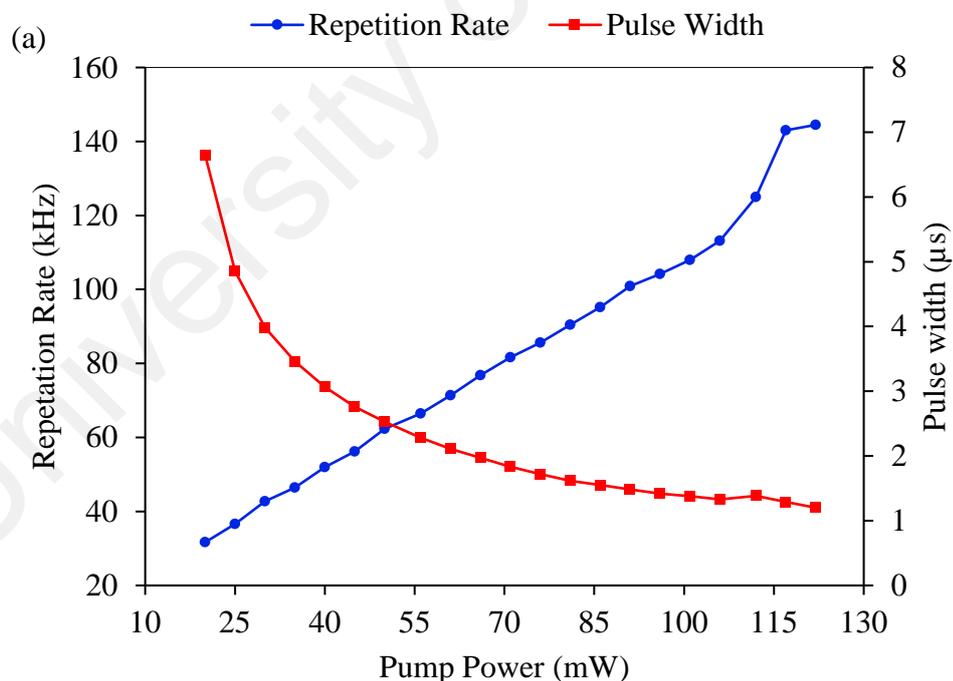
**Figure 4.19, continued**

When the input pump power was decreased to the range between 20 mW to 122 mW, a stable Q-switching pulses train was produced again, proving that the Alq<sub>3</sub> based SA was not damaged. To check the stability of Q-switching pulse operation, we recorded the corresponding RF spectrum at a maximum input pump power of 122 mW as revealed in Figure 4.20 (a). The spectrum depicts the fundamental frequency at 144.5 kHz with SNR of ~46 dB, as shown in Figure 4.20 (b).

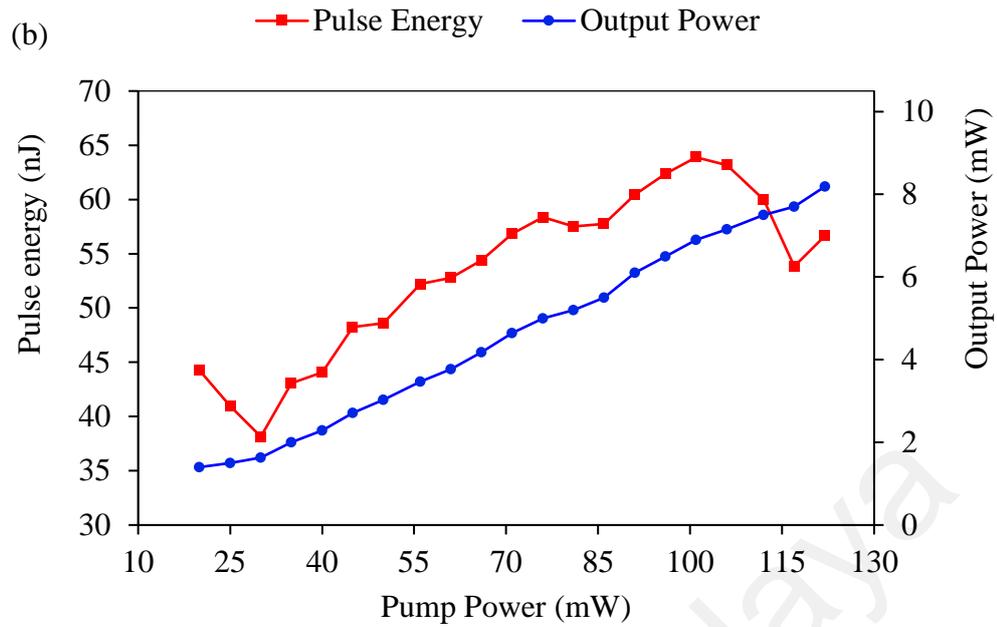


**Figure 4.20: Radio-frequency spectrum at a pump power of 122 mW with a span of (a) 900 kHz (b) 60 kHz**

When the pump power was increased from 20 to 122 mW, the repetition rate increased from 31.65 to 144.5 kHz and the pulse width reduced from 6.6 to 1.2  $\mu\text{s}$  as shown in Figure 4.21 (a). The high repetition rate and short pulse width might be attributed to the recovery time of the SA. When the SA's recovery time is much shorter than the time duration between the Q-switching pulses, the pulse repetition rate will increase with the volumetric input pump power rate (Degnan, 1995). However, further research is required to improve the performance of Alq<sub>3</sub> SA and to determine its recovery time. Figure 4.21 (b) illustrates the output power and pulse energy against the pump power. As the pump power increased, the output power raised from 1.4 to 8.19 mW and the pulse energy also increased from 44.23 nJ to 56.67 nJ. The pulse energy reached its maximum value of 63.89 nJ at a pump power of 101 mW.

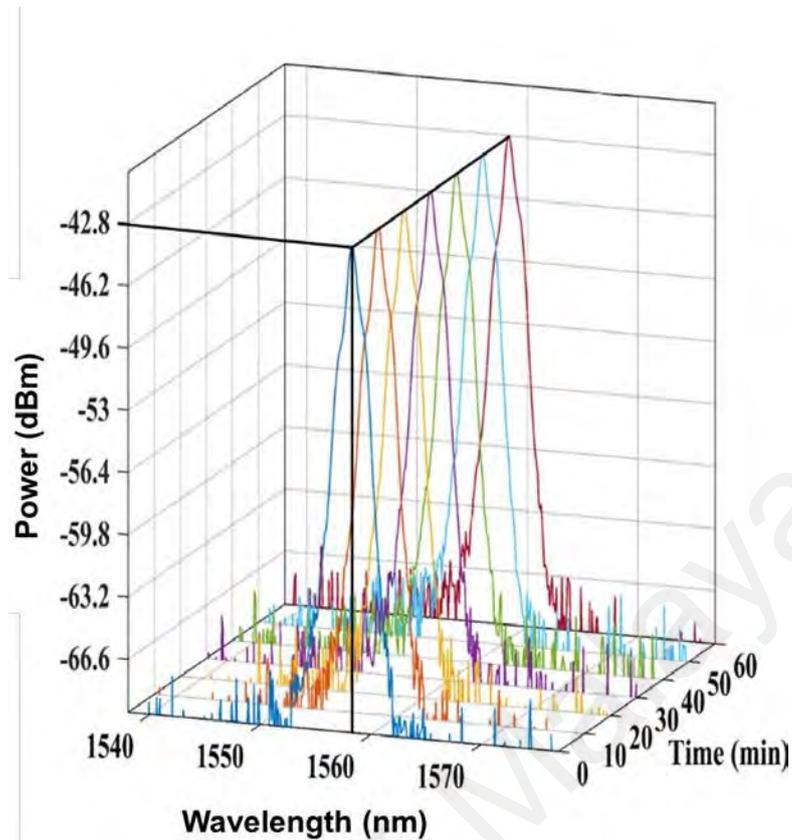


**Figure 4.21: (a) Pulse width and pulse repetition rate versus incident pump power and (b) Output power and pulse energy versus incident pump power**



**Figure 4.21, continued**

Figure 4.22 depicts the output spectrum of the Q-switched operation at 122 mW pump power. The spectrum was sampled at an interval of 10 min for a total duration of 1 hour to examine the consistency of the output spectra. As shown in the figure, all spectra have an almost similar pattern, shape and spectral intensity (all the samples have a spectral intensity of about  $-42.8$  dBm) which indicates that the Q-switched fiber laser is reliable and stable over time. It was confirmed that the Al<sub>q</sub><sub>3</sub>: PVA thin-film based organic SA had no sign of damage or melting and was still in good shape after the Q-switched operation lasted for more than 60 minutes. The fiber laser showed a stable performance without observable fluctuation in the output spectra.

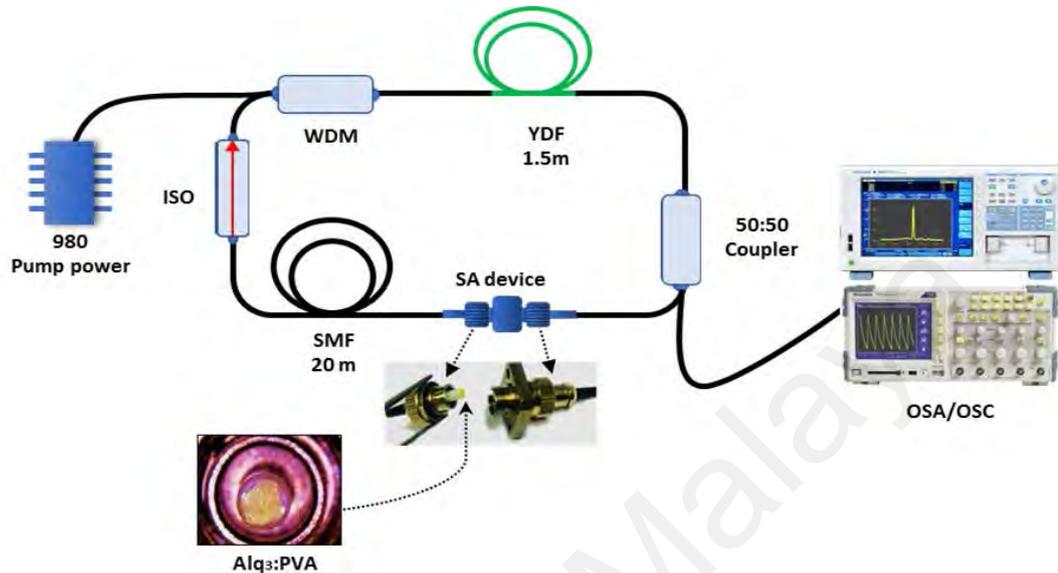


**Figure 4.22: Laser spectrum sampled at 10 min intervals for 1 h duration**

#### **4.5.2 High Pulse Energy Q-Switched Fiber Laser Operating at 1.0 $\mu\text{m}$ Operation**

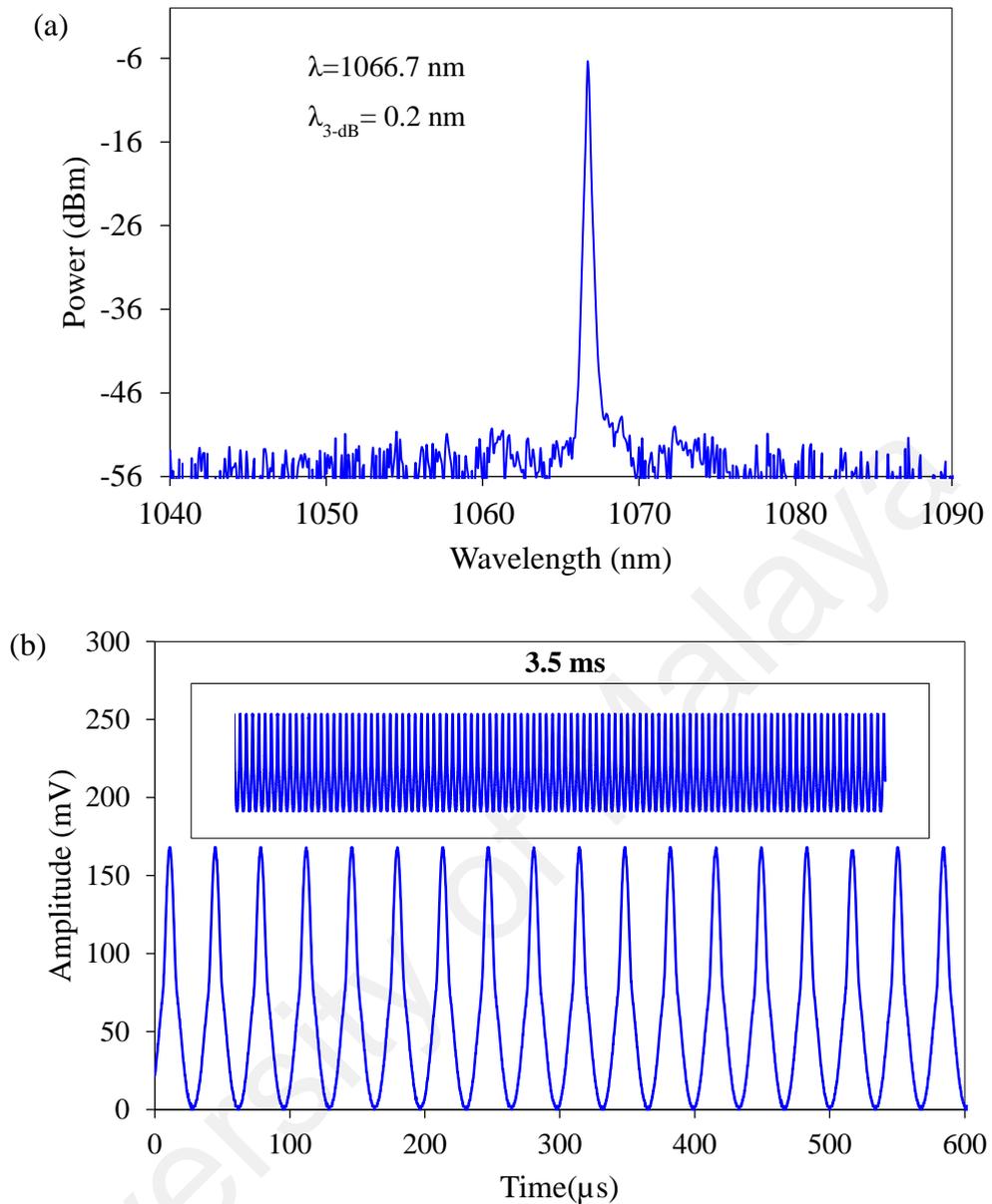
In this subsection, a Q-switched fiber laser operating in 1.0  $\mu\text{m}$  region is demonstrated by using  $\text{Alq}_3$  PVA thin-film as a SA. The SA device was incorporated into an ytterbium-doped fiber laser (YDFL) cavity. The cavity setup is presented in Figure 4.23. A 1.4 m long Ytterbium-doped fiber (YDF) was used as a gain medium. The YDF has Yb ions concentration of 1500 ppm, a core diameter of 4  $\mu\text{m}$  and a numerical aperture of 0.20. The cavity length was approximately 25 m. The ring cavity was coupled with a 980 nm LD through a 980/1064 nm WDM. A 50:50 optical coupler (OC) was spliced to another end of the YDF to allow half of the output to be extracted for performance monitoring. The remaining output was directed to the  $\text{Alq}_3$ : PVA thin-film which was sandwiched between two fiber ferrules. An optical isolator was used to eliminate back-reflection. The

characteristics of the laser were analyzed using a digital oscilloscope with 2 GHz photodetector, an optical spectrum analyzer and a 7.8 GHz RF spectrum analyzer.



**Figure 4.23: Configuration of the proposed Alq<sub>3</sub> based Q-switched YDFL**

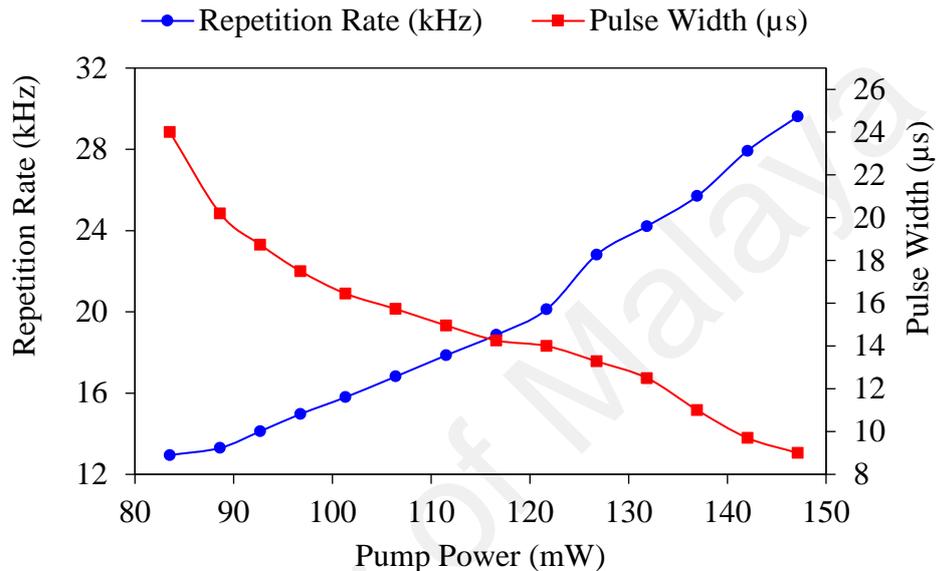
At first, a CW laser emission was produced at input LD power of 60 mW. A self-starting and stable Q-switched YDFL operation was recorded as the pump power was increased above the threshold of 84 mW. The Q-switching operation was maintained up to pump power of 147 mW where the relatively high energy and high output power were achieved. Figure 4.24 (a) shows the output spectrum of the Q-switched YDFL, which operated at a centre wavelength of 1066.7 nm with a 3-dB bandwidth of 0.2 nm. Figure 4.24 (b) shows the temporal characteristic of the Q-switched pulse train at the maximum input pump power; the inset Figure shows the pulse train in the time scale of 3.5 ms. The inset figure shows a uniform intensity distribution without amplitude modulation nor fluctuation (as almost all the pulses had an amplitude of  $168 \text{ mV} \pm 0.05 \text{ mV}$  when they were recorded at a sampling period of  $0.7 \mu\text{s}$ ), which indicates the fiber laser has stable performance.



**Figure 4.24: (a) Output spectrum of the Q-switched YDFL operating at 1066.7 nm and (b) typical pulse train at the maximum input LD power of 147 mW**

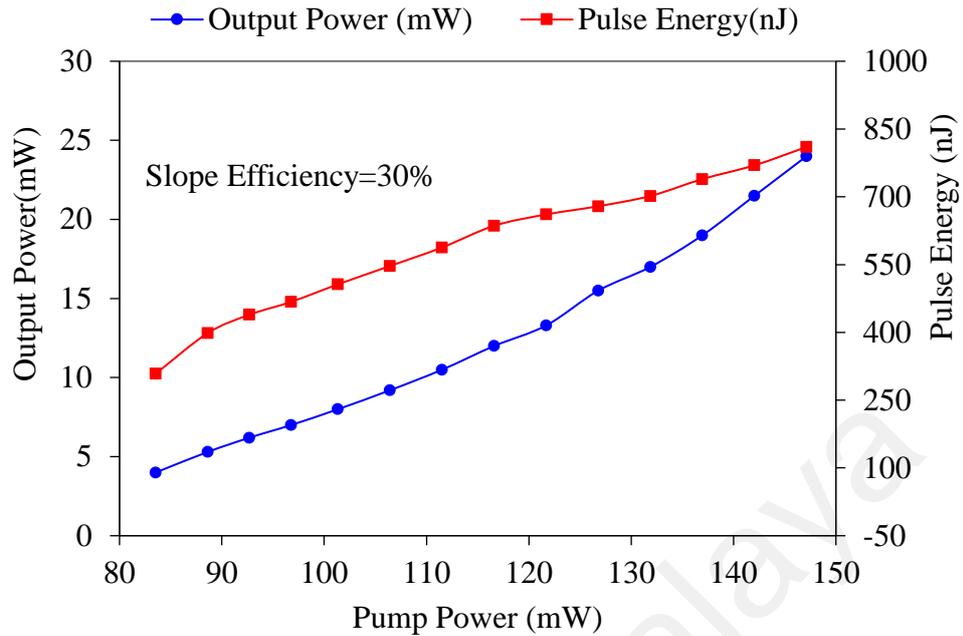
Figure 4.25 shows the repetition rate and pulse width of the YDFL versus pump power. It is obtained that the pulse repetition rate increased from 12.95 kHz to 29.62 kHz while the pulse width shortened from 24  $\mu\text{s}$  to 9  $\mu\text{s}$  as the input LD power was tuned from threshold to maximum value, i.e. from 84 mW to 147 mW. Increasing the input pump power (up to 249 mW) caused the pulse train to be unstable and then disappeared. That might be attributed to the SA being over-saturated, as similar behaviour has been reported previously (H Ahmad et al., 2017; Y. Chen et al., 2014; Q. Wang et al., 2015). The LD

power was increased up to the maximum power of 350 mW to investigate the damage threshold for the SA. As the LD power was gradually reduced within 84 mW to 147 mW, the Q-switched operation was observed again. Hence, we concluded that the thermal damage of the SA is higher than 350 mW.



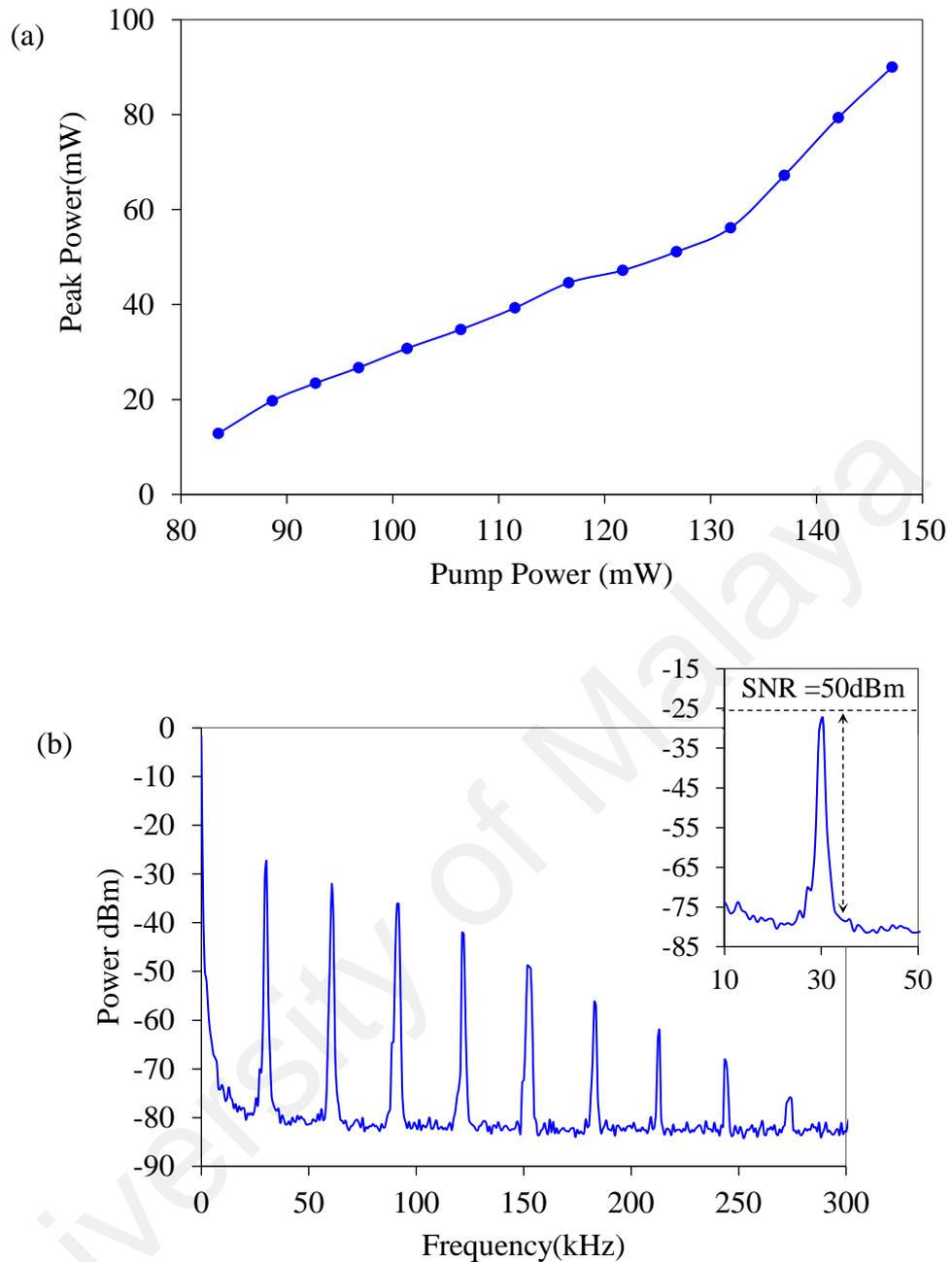
**Figure 4.25: Pulse repetition rate and pulse width as a function of input LD power**

Figure 4.26 shows the average output power and pulse energy against pump power. It is observed that the output power increased almost linearly from 4 mW to 24 mW with an increase in the input pump power from 84 to 147 mW. The average output power of the cavity was relatively high, that is due to the high performance and low insertion loss of the SA and low inter-cavity loss (Herda et al., 2008). Additionally, a large portion of signal power was extracted from the cavity due to the high tapping ratio of the OC. The slope efficiency of the YDFL was obtained at around 30%. The proposed YDFL also produced high pulse energy ranging from 0.3 to 0.81  $\mu\text{J}$  as the pump power was increased from 84 mW to 147 mW. The high pulse energy is attributed to the high average output power and low repetition rate.



**Figure 4.26: Average output power and pulse energy versus input LD power for Alq<sub>3</sub> based Q-switched YDFL**

The maximum pulse energy is 0.81  $\mu\text{J}$ , which is the highest compared to other passively Q-switched fiber lasers reported so far that operating in in 1  $\mu\text{m}$  region (Hou et al., 2018; K.-X. Huang et al., 2017; Rahman, Latiff, Rusdi, Dimiyati, & Harun, 2018; J. Wang et al., 2018). As shown in Figure 4.27 (a), the peak power also increased almost linearly from 12.9 to 90 mW within the given pump power range. To check the stability of Q-switched laser operation, the RF spectrum was measured with a span of 300 kHz, see Figure 4.27 (b). The inset Figure shows the fundamental RF peaks at 29.62 kHz with an SNR of about 50 dB, which indicates excellent Q-switching stability.

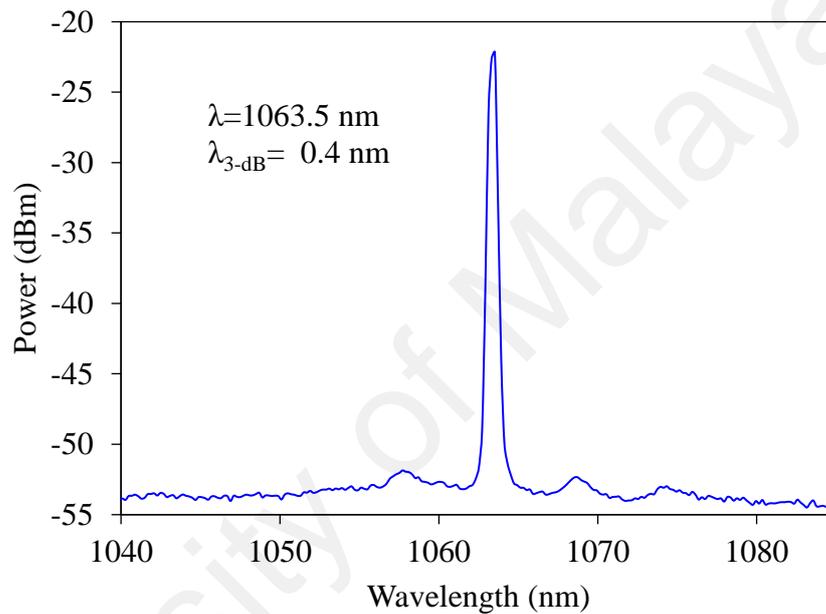


**Figure 4.27: Peak power against LD power for Alq<sub>3</sub> based Q-switched YDFL, and (b) RF spectrum of the Q-switched YDFL. Inset shows the fundamental frequency with SNR 50 dB at a span of 40 kHz.**

### 4.5.3 Short pulse width Q-switched YDFL

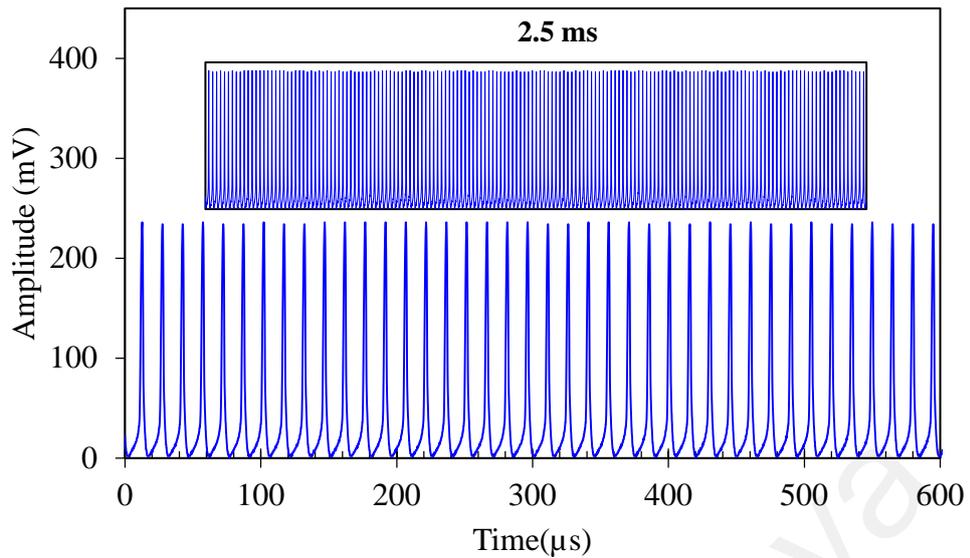
In this subsection, a Q-switched YDFL with a shorter pulse width is demonstrated by reducing the cavity length (as given in Figure 4.23) to 5 m. The proposed YDFL successfully produced a Q-switched pulse train with reduced pulse width and higher

repetition rate. Both pulse energy and peak power performances were also changed with this modification. In the new cavity, the CW YDFL emission was observed at the input LD power of 50 mW, while the threshold and maximum LD power for generating the Q-switched pulses were 87 mW and 147 mW, respectively. The optical spectrum showed almost similar shape in comparison with the previous cavity setup with slightly broader 3-dB bandwidth of about 0.4 nm, as can be seen in Figure 4.28.



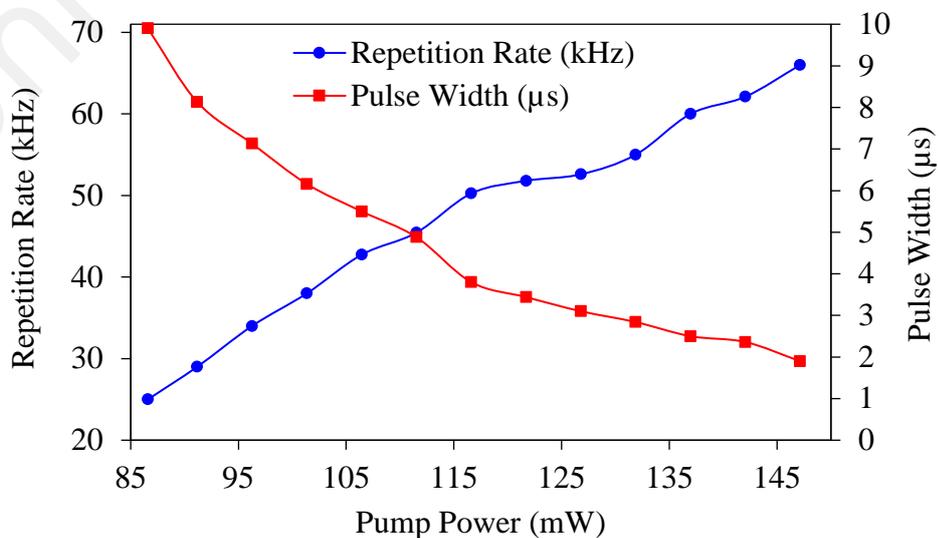
**Figure 4.28: Optical spectrum of the Q-switched YDFL with 5 m cavity length**

The centre wavelength slightly shifted to a shorter wavelength to compensate for the round-trip time difference due to the change in physical cavity length. Figure 4.29 shows the temporal characteristics at the maximum input LD power. The pulse train is more stable and uniform than the one in the previous setup which indicates the performance and stability of the fiber laser is enhanced. The inset presents the pulse train at a time scale of 2.5 ms.



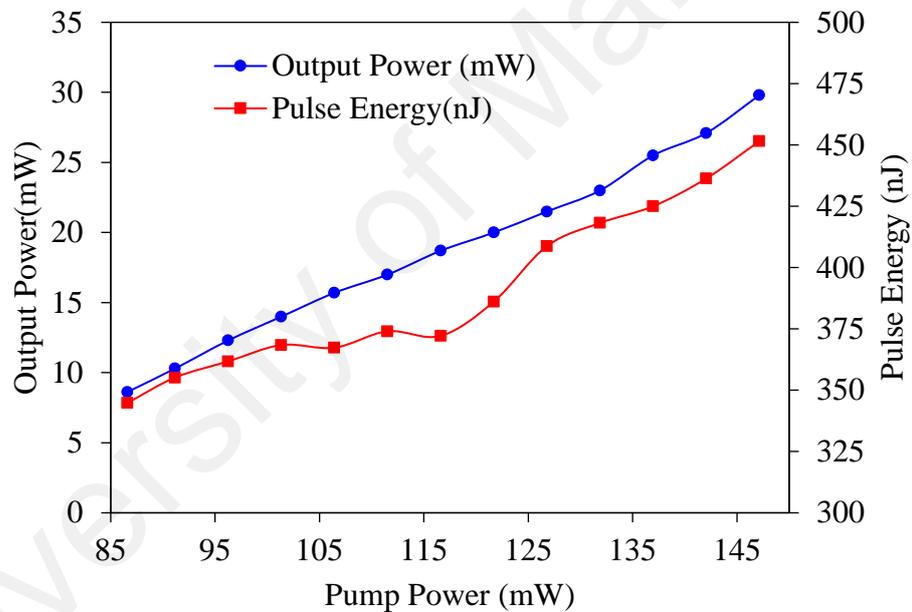
**Figure 4.29: Pulse train at the maximum LD power**

The repetition rate was raised from 25.2 kHz to 66.3 kHz and the pulse duration was shortened from 9.9  $\mu\text{s}$  to 1.9  $\mu\text{s}$  as the input LD power was raised from threshold to the maximum value, see Figure 4.30. The pulse repetition rate was increased as compared to the previous setup, as the decrease in the cavity length resulted in a shorter time for a pulse to travel through the cavity. Besides, the saturation of the SA is more rapid as compared to the 25 m cavity, resulting in a shorter pulse width and larger repetition rate.



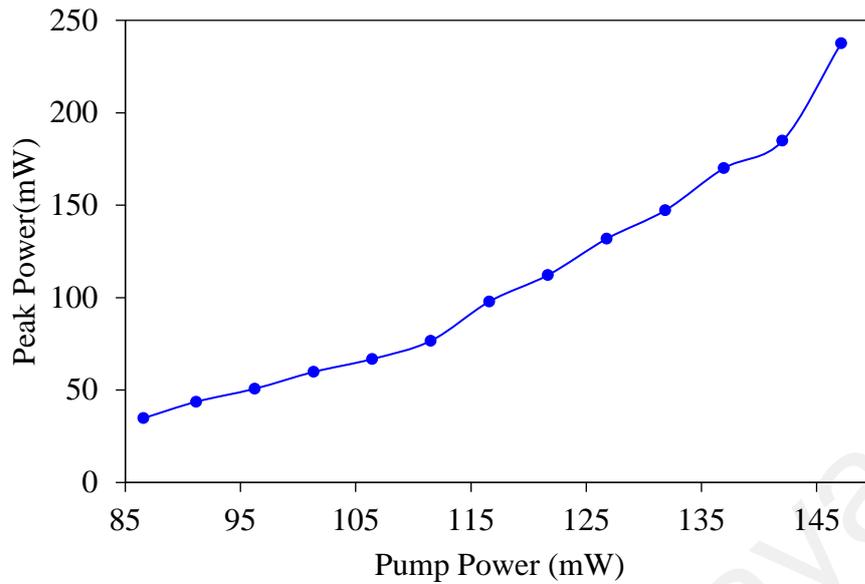
**Figure 4.30: Pulse repetition rate and pulse width duration versus input LD power**

The output power was enhanced in comparison to the previous setup due to lower optical loss with shorted cavity length. This setup produced an output power of 8.62 mW which is about twice the power produced in the previous experiment at the same input LD power. At the same time, higher slope efficiency was obtained, which is 33%. As can be seen from Figure 4.31, the output power reached the maximum value of about 30 mW at maximum input LD power. The maximum pulse energy achieved in this setup is 451.5 nJ which is about half of the value in the previous setup. That can be attributed to the pulse energy being inversely proportional to the pulse repetition rate. The increment in the pulse repetition rate resulted in a drop in the pulse energy.



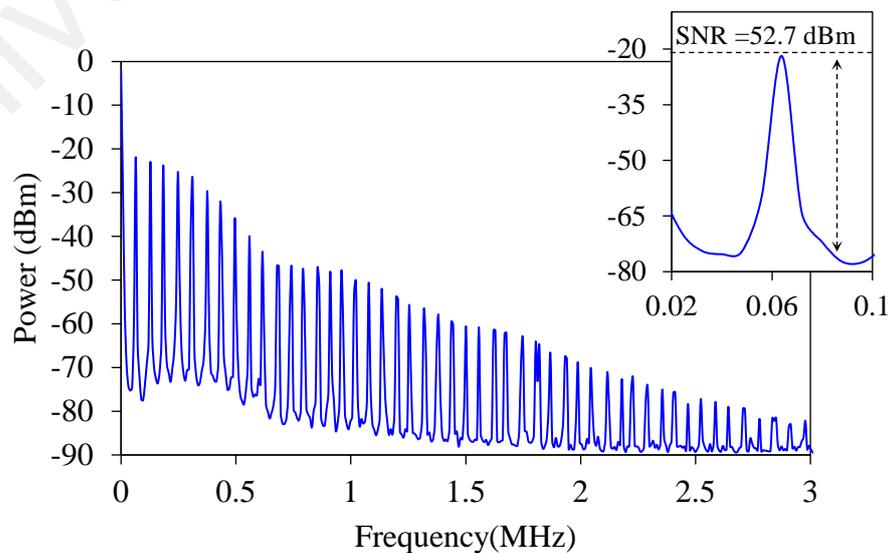
**Figure 4.31: Output power and pulse energy as a function of input LD power**

The peak power was enhanced tremendously in this setup reaching linearly to a maximum value of 237.62 mW at maximum input LD power of 147 mW, see Figure 4.32. That can be attributed to the peak power being correlated inversely with the pulse width and proportionally with the pulse energy.



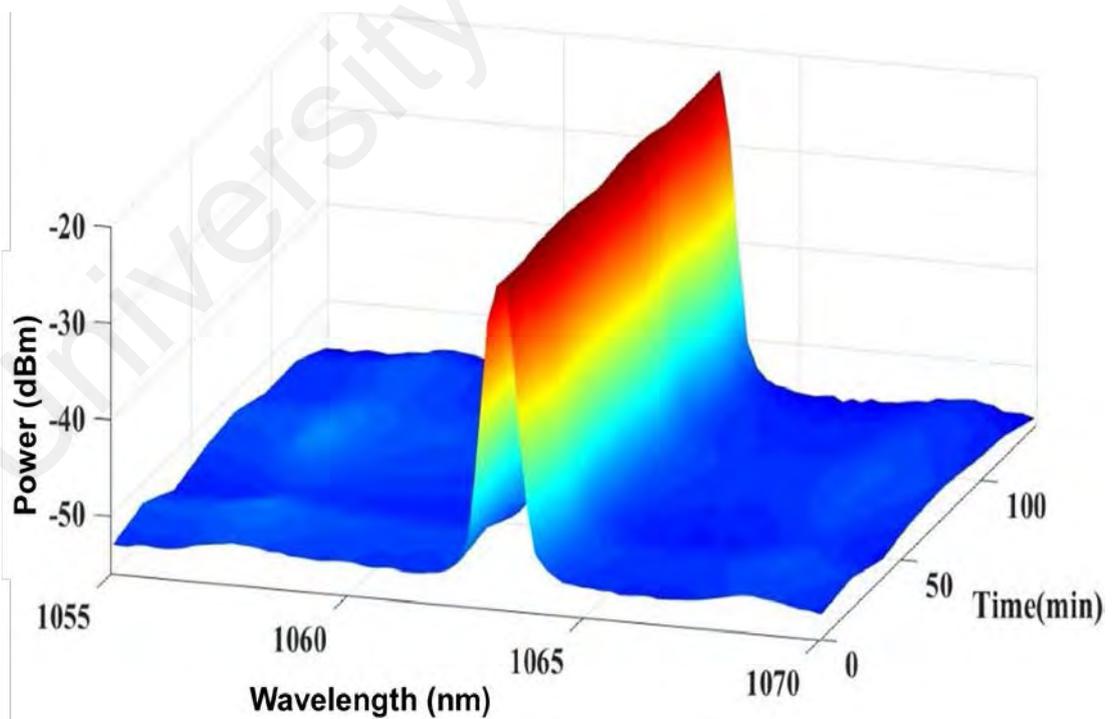
**Figure 4.32: Peak power versus input LD power**

In this setup, the pulse width was decreased by about eight times while the pulse energy was reduced only to the half as compared with the previous setup under similar considerations. Figure 4.33 shows an improvement in Q-switching stability. The SNR was recorded to be 52.78 dB, see the inset Figure. Additionally, the RF spectrum shows more harmonics up to 3 MHz which further indicates better stability of fiber laser.



**Figure 4.33: RF spectrum, inset Figure shows SNR at a span of 40 kHz**

The reduction in RF intensities (in Figure 4.27 (b) and Figure 4.33) confirms the Q-switching operation, which has a relatively bigger pulses width than the mode-locking. To check the long-term stability of the fiber laser, we sampled the output spectrum every  $\sim 12$  minutes for the total duration of 138 minutes, see Figure 4.34. The samples were almost identical, as the peaks were located at 1063 nm with an optical power intensity of  $-22 \text{ dBm} \pm 0.4 \text{ dBm}$ . This further confirms the stability of the pulsed laser and shows that the SA can withstand long term operation. An optimized Q-switching operation can be obtained by further reducing the cavity length and/or enhancing the cavity design (Degnan, 1995; Herda et al., 2008). To the best of our knowledge, this work has achieved (by using SA based compact all-fiber ring cavity and through two different setups) very high pulse energy and very high peak power at 1  $\mu\text{m}$  region. Additionally, this is the first demonstration of passive Q-switched fiber laser using an organic material as SA operating in 1  $\mu\text{m}$  region.



**Figure 4.34: Output optical spectrum evolution for the total duration of 135 minutes**

## 4.6 Summary

Q-switched fiber lasers have been successfully demonstrated using the newly developed organic material based SAs operating in both 1.5 and 1.0  $\mu\text{m}$  wavelength regions. At first, Firpic SA was used to produce pulses train operating at a central wavelength of 1560.4 nm. The laser's pulse width reduces from 9.5  $\mu\text{s}$  to 3.4  $\mu\text{s}$  while the repetition rate increases from 39.22 to 87.4 kHz as the pump power was increased from 30 to 208 mW. The SA was also used to produce a dual-wavelength output operating at 1558.1 nm and 1562.6 nm by incorporating a microfiber device inside the cavity.  $\text{Znq}_2$  was then used to produce Q-switching pulses. The laser produced pulse width and repetition rate in a range of 6.6- 2.8  $\mu\text{s}$  and 45-86 kHz as the pump power was varied from 38 to 198 mW. Tunable Q-switched EDFL was also realized by using  $\text{Znq}_2$  SA as a Q-switcher. The central wavelength of the tunable laser can be shifted continuously from 1520.0 nm to 1563.3 nm, covering almost all C-band region by tuning the band-pass filter inside the laser cavity. Finally, the developed  $\text{Alq}_3$  SA was successfully used to produce Q-switched EDFL operating at 1559 nm, which the pulse width is tunable from 6.6 to 1.2  $\mu\text{s}$  and the repetition rate is tunable from 31.65 to 144.5 kHz. The SA also used to produce Q-switching pulsing with high-output power and high pulse energy at 1.0  $\mu\text{m}$  region. The Q-switched YDFL produced high pulse energy of 0.8  $\mu\text{J}$  and peak power of 90 mW at cavity length of 25 m. As the laser cavity was reduced to 5 m, the peak power increased to 237.62 mW, the pulse energy dropped to 451.5 nJ and the minimum pulse width was reduced from 9  $\mu\text{s}$  to 1.9  $\mu\text{s}$ . These results indicate that the proposed organic materials can be easily used to generate different types of Q-switched fiber lasers with a low-cost, simple and straightforward fabrication process. All the produced laser output had a very high SNR of around 56 dB and very stable performance in terms of long-term operation and stable pulse train output. Table 4.1 summarize the performance of the Q-switched fiber lasers developed by using Firpic,  $\text{Znq}_2$  and  $\text{Alq}_3$  as saturable absorbers. This work

presents the first demonstration of these materials in fiber laser technology to the best of our knowledge.

**Table 4.1: Performance of Q-switched fiber lasers by using the developed SAs**

Section in the thesis	Pulse width ( $\mu\text{s}$ )	Repetition rate (kHz)	Wavelength (nm)	Pulse energy (nJ)	SNR (dB)	SA
4.2.2	9.5 – 3.4	39.22 – 87.4	1560.4	122.6	58.36	Flrpic
4.3.2	8.4 – 3.6	32.15 – 78	1558.1 and 1562.6	110.13	55.36	
4.4.1	6.6 – 2.8	45 – 86	1560	95.35	57.4	Znq <sub>2</sub>
4.4.2	–	-	1520 – 1563.3	-	53.2	
4.5.1	6.6 – 1.2	31.65 – 144.5	1559	63.8	46	Alq <sub>3</sub>
4.5.2	24 – 9	12.95 – 29.62	1066.7	810	50	
	9.9 – 1.9	25.2 – 66.3	1063.5	451.5	52.7	

## CHAPTER 5: MODE-LOCKED PULSE GENERATION BY USING ORGANIC MATERIALS

### 5.1 Introduction

Ultrafast mode-locked fiber lasers have gained a great research interest in recent years due to their unique advantages, such as low cost, flexibility and good reliability (X. Liu, 2011; L. Zhang et al., 2011). Additionally, they have widespread applications in telecommunications, supercontinuum generation, material processing, nonlinear microscopy and frequency comb generation (Fermann & Hartl, 2013; Nicholson, Yablou, Westbrook, Feder, & Yan, 2004; Smirnov, Kobtsev, & Kukarin, 2014). The mode-locked lasers are normally produced by a passive technique by utilizing many types of saturable absorbers (SAs), such as transition metal-doped bulk crystals (D. Lin et al., 2010) and semiconductor saturable absorber mirrors (SESAMs) (M. Wang, Chen, Li, Huang, & Chen, 2014). However, doped crystal SAs are mainly utilized in solid-state laser while SESAMs are costly and work only in a narrow wavelength range and also require a complex fabrication to improve their damage threshold (Saraceno et al., 2011). SA based mode-locked fiber lasers have also been demonstrated using carbon nanotubes (CNTs) and graphene (H. Zhang et al., 2010). CNTs have the advantages of easy and low-cost fabrication, while graphene has the advantage of ultrafast recovery time (Q. Bao et al., 2009), wide operation spectral range (B. Y. Zhang et al., 2013) and large absorption per layer (Nair et al., 2008). Yet, both have some limitations, as CNTs often require band gap engineering (Q. L. Bao et al., 2009), and graphene suffers from the absence of band gap. Recently, transition metal dichalcogenides (TMDs), such as tungsten disulfide ( $WS_2$ ) (Yan et al., 2015) and molybdenum disulfide ( $MoS_2$ ) (Hao Liu et al., 2014), have been extensively investigated and demonstrated as SAs, as they show unique absorption property (B. Chen et al., 2016). But, TMDs suffer also from a low optical damage threshold (Horiguchi et al., 2008; Latiff et al., 2017), and a complicated and costly

fabrication process. Recently, black phosphorus (BP) has been reported as a promising SA (Jaroslaw Sotor et al., 2015). BP has a broadband nonlinear optical response (Lu et al., 2015) and it can be easily fabricated as it consists of only one element (phosphorus). However, BP is a hydrophilic material (J. Li et al., 2016) which is easily damaged when exposed to water and oxygen.

Nevertheless, these nanomaterials might be dangerous on human health in the case of long-term exposure (Nel et al., 2006). Besides, there are several attempts more recently to develop SA from new material. In that respect, organic materials might have the potentials in fiber laser technology. They can be utilized as a high-performance, bio-compatible and environment-friendly SAs. organic materials have the advantages of ultrafast nonlinear response and broad spectral tunability (Boulet et al., 2015; Clark & Lanzani, 2010; Williams, 1983). Additionally, They could increase the applications in sensing and in-situ biomedical laser processing (Khazaeinezhad et al., 2017). Furthermore, they are mechanically flexible, light-weight, and can be produced at a low cost (Someya et al., 2004). Due to their unique characteristics, organic materials are used in several applications, such as an organic thin-film transistor (J.-M. Kim et al., 2012), organic light-emitting diodes (O. Y. Kim & Lee, 2012; Yook & Lee, 2012), organic solar cells (J. Y. Kim et al., 2007; Peumans et al., 2011), and organic bistable memory devices (Briseno et al., 2006).

In this chapter, various mode-locked erbium-doped fiber lasers (EDFLs) are demonstrated using the newly developed organic material-based SA devices. Three organic materials; Bis[2-(4,6-difluorophenyl)pyridinato- $C^2,N$ ](picolinato)iridium(III) (FIRpic), bis(8-hydroxyquinoline)zinc ( $Znq_2$ ) and Tris(8-hydroxyquinoline)aluminum ( $Alq_3$ ) are used in this work for the generation of stable mode-locked laser pulses. FIRpic was successfully used as mode-locker and produced a soliton wavelength. The produced pulsing was stable and showed long term operation. On the other hand,  $Znq_2$  was

successfully used to produce ultrafast pulsing with dark pulsing, white pulsing and supercontinuum operations. Alq<sub>3</sub> was successful as well in generating mode-locking pulses in both regions of 1.0  $\mu\text{m}$  and 1.5  $\mu\text{m}$  regions. Different pulse widths were generated as the parameters of the cavity were changed. It should be mentioned that dark pulses are less affected by intra-pulse-stimulated Raman scatters and stable in the presence of fiber loss and noise (L.-Y. Wang et al., 2012; X. Wang, Zhou, Wang, Xiao, & Liu, 2014). That makes them very attractive in applications of optical-processing system and telecommunications.

## 5.2 Mode-Locked Erbium-Doped Fiber Laser with FIrpic SA

In this section, a soliton mode-locked EDFL is demonstrated using FIrpic PVA film as a SA. Figure 5.1 shows the configuration of the proposed mode-locked laser, which is almost similar setup to the Q-switched laser (Figure 4.1) as described in section 4.2.1 of the previous chapter except that the output coupler (OC) used was 99:1 and a spool of single-mode fiber (SMF-28) was added between the SA device and isolator.

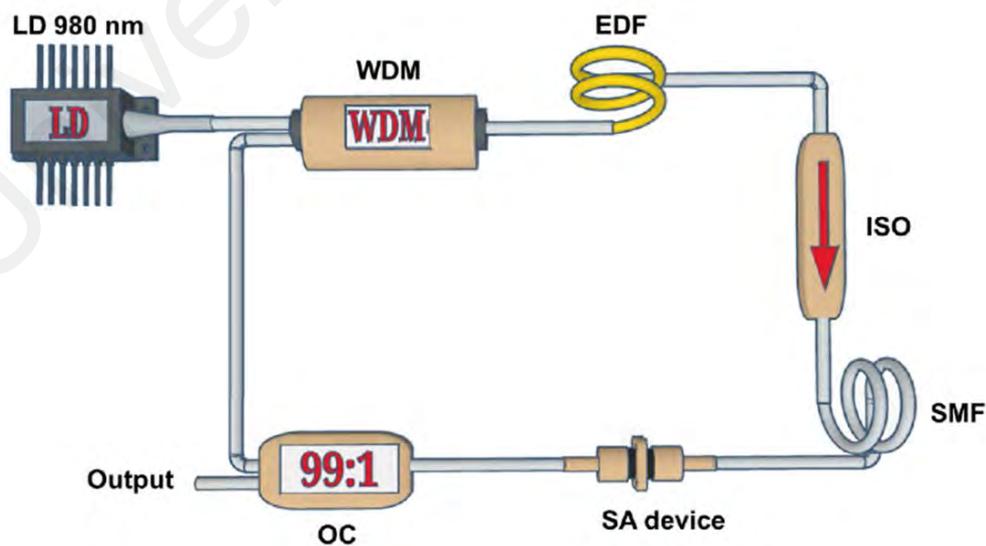
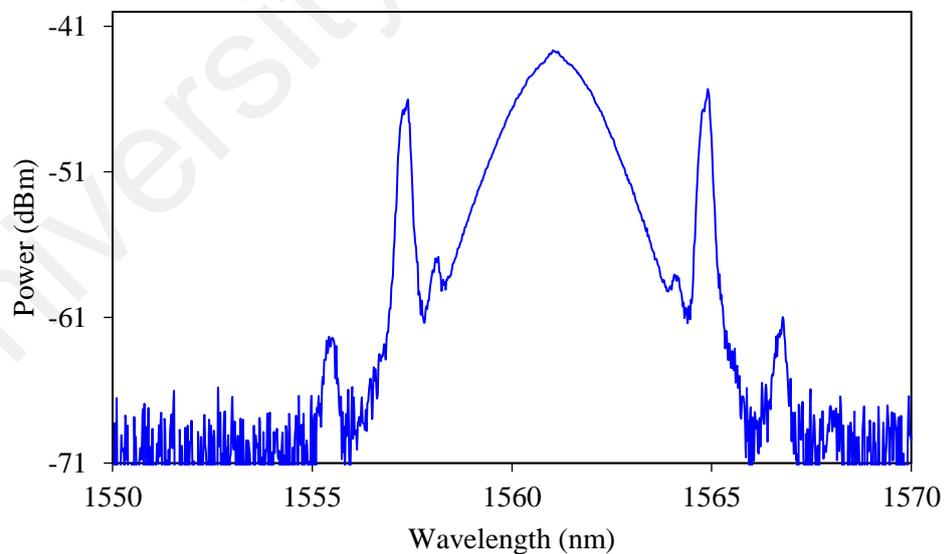


Figure 5.1: Configuration of the mode-locked EDFL

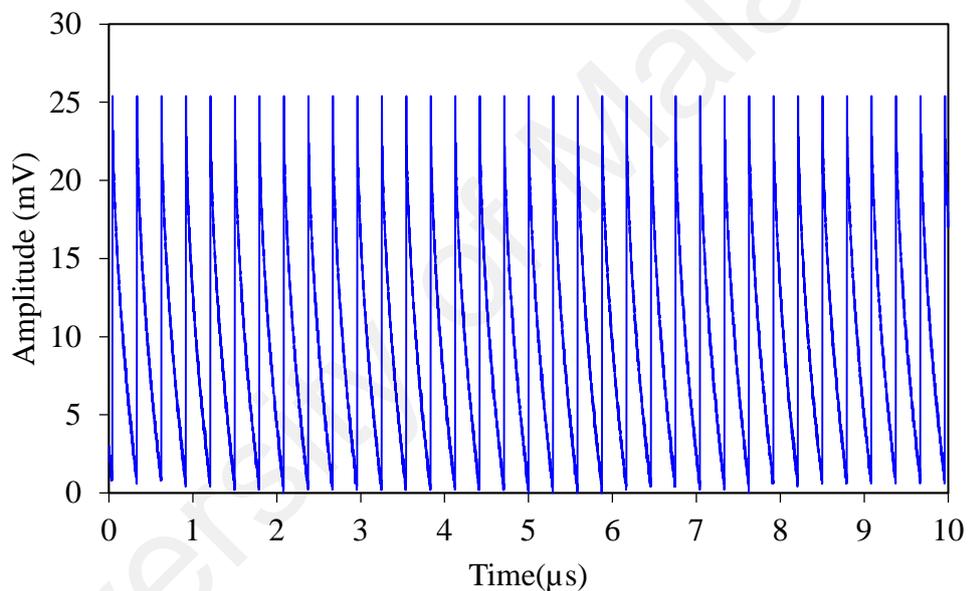
The balance between the nonlinearity effect and group velocity dispersion (GVD) inside the ring cavity was managed through the addition of SMF which allows the generation of a soliton mode-locking. The addition of 52 m SMF causes the cavity to operate in anomalous dispersion of about  $-1.216 \text{ ps}^2$ , the total cavity length was about 60 m. The wavelength division multiplexer (WDM) coupler, SMF and EDF have GVD of  $-48.5$ ,  $-21.7$ , and  $27.6 \text{ ps}^2/\text{km}$ . A 99:1 OC was also used to maintain a high-power oscillating inside the laser cavity. The laser self-started mode-locking pulses at the pump power of 35 mW and the mode-locking operation were maintained up to the maximum pump power of 188 mW. The laser has an optical spectrum centred at 1562.57 nm with a 3-dB bandwidth of 1.8 nm, as shown in Figure 5.2. The mode-locking pulses had a soliton shape spectrum with Kelly sidebands that can be easily observed due to the periodic perturbation in the cavity. The employment of SMF into the cavity led to the increase in the net dispersion, birefringence and nonlinearity of cavity which helps the mode-locking operation.



**Figure 5.2: Optical spectrum of the soliton laser**

Additionally, the saturation absorption effect from FIrpic based SA and the non-linearity of the EDF also contributed to the formation of soliton mode-locked pulses train.

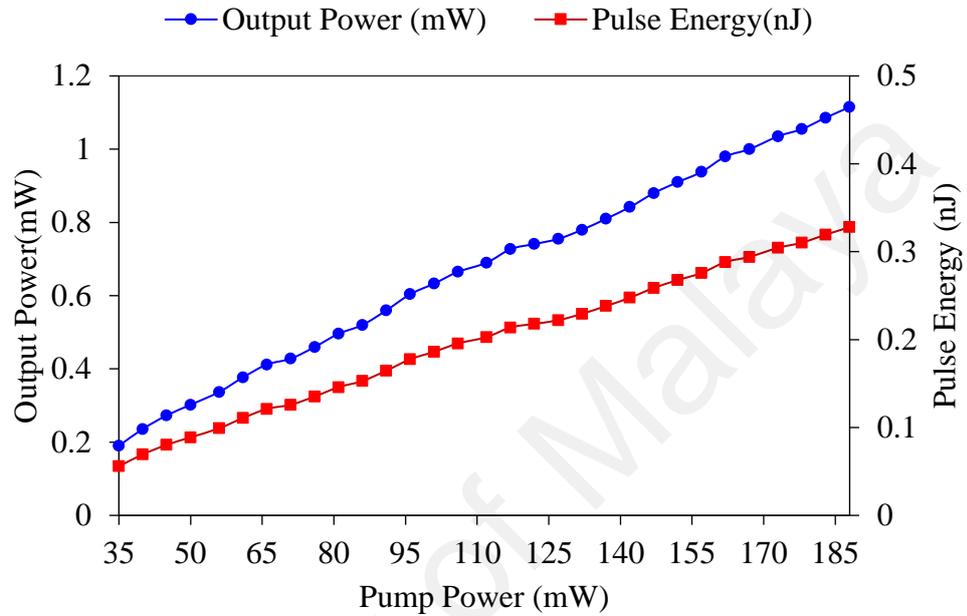
Figure 5.3 shows the output pulse train of the soliton laser in the time domain at the maximum pump power of 188 mW. It shows a very uniform pulse amplitude with a pulse width of about ~120 ns. An attempt was made to measure the pulse width with an autocorrelator, and it was unsuccessful due to the limited specifications of the autocorrelator as it could only detect a pulse width narrower than 50 ps. The interval between adjacent pulses is about 291.5 ns which represents a repetition rate of 3.43 MHz. The obtained repetition rate matches the fundamental frequency and the total cavity length of about 60 m.



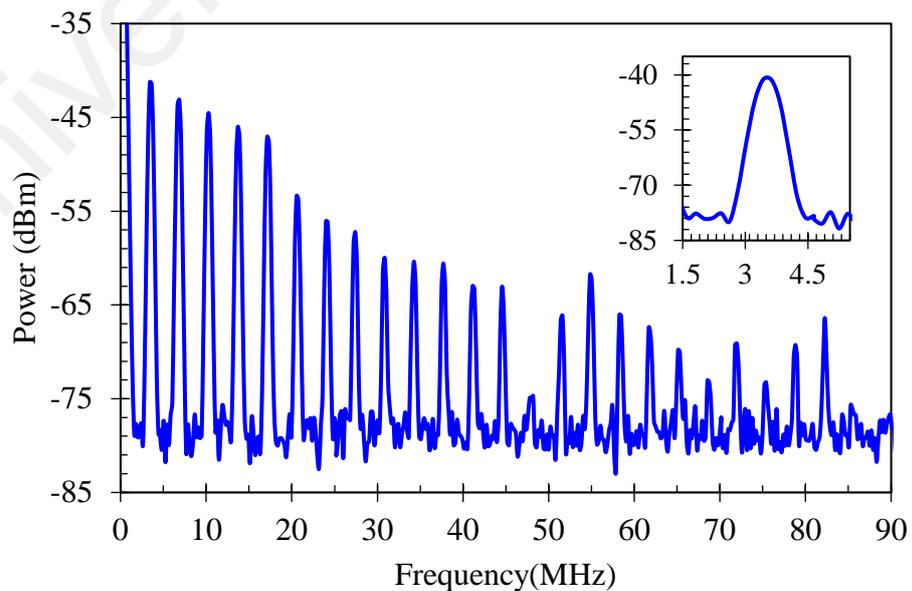
**Figure 5.3: Optical pulse train in the time domain**

The output power and pulse energy versus input pump power are also investigated in the proposed mode-locked EDFL and the results are shown in Figure 5.4. As the pump power was tuned from 35 mW to 188 mW, the output power and pulse energy increased from 0.19 to 1.1 mW and from 0.06 to 0.33 nJ, respectively. The RF spectrum was also measured to check the stability of the mode-locking operation in a span of 85 MHz with a resolution bandwidth (RBW) of 10 Hz, as shown in Figure 5.5. The figure shows the fundamental frequency with many harmonics. The fundamental frequency operates at

3.43 MHz with an SNR of 38.3 dB as shown in the inset figure. It is observed that the number of harmonics is limited, which further verifies that the mode-locked laser is operating in a nanosecond regime.

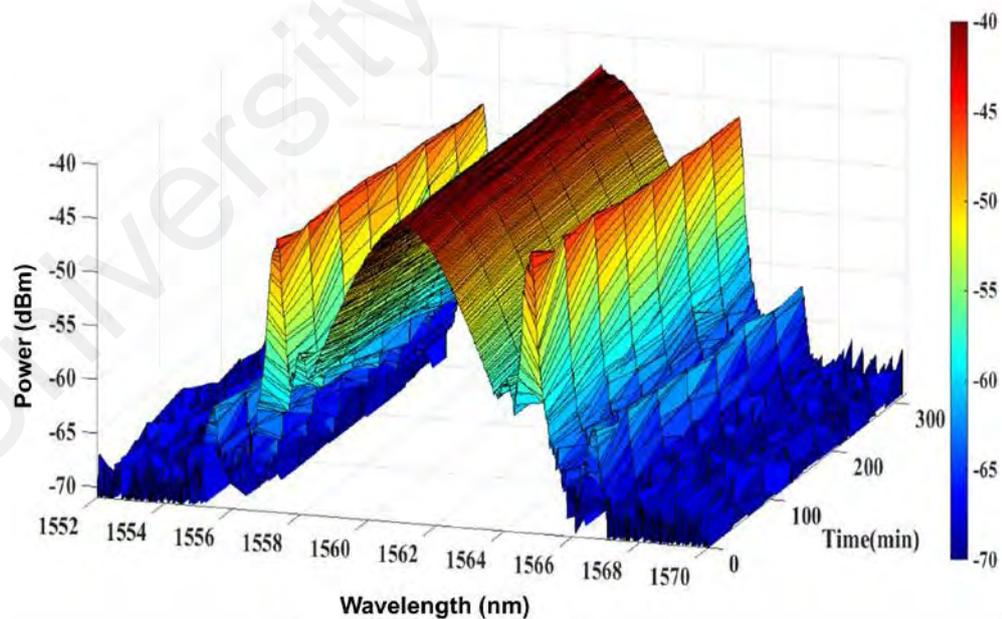


**Figure 5.4: Output power and pulse energy versus input pump power**



**Figure 5.5: RF spectrum**

Finally, the output optical spectrum was sampled at an interval of ~45 min for a total duration of 320 minutes, see Figure 5.6. The figure shows the samples have almost the same shape, pattern and optical power intensity (about  $-42.7 \text{ dB} \pm 0.17 \text{ dB}$ ) which reveals the very good performance of the SA device. In summary, a soliton mode-locked laser was successfully achieved in anomalous dispersion cavity through the addition of 52 m SMF. The mode-locked laser has a fixed repetition rate of 3.43 MHz and 120 ns pulse width. To the best of the authors' knowledge, this is the first report of the organic material being used as a SA in a fiber laser cavity. These results indicate that FIRpic is suitable for use in producing pulsed lasers operating in the  $1.5 \mu\text{m}$  wavelength region. The generated pulses laser has comparable performance to those of other SA materials such as CNTs, graphene, TMDs, MoS<sub>2</sub>, WS<sub>2</sub> and BP (Babar, Harun, Yasin, & Ahmad, 2017; Cheng et al., 2013; B. Gao et al., 2018; N. Razak et al., 2017; Rosol et al., 2017; Yang, Yang, Li, & Lin, 2019)

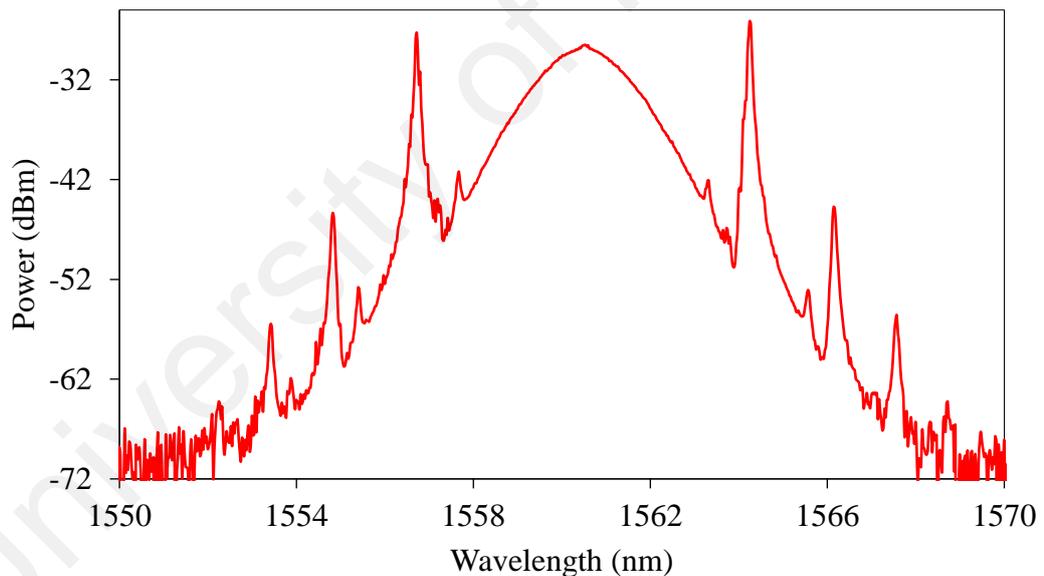


**Figure 5.6: Output optical spectrum sampled at an interval of ~45 minutes for the total duration of 320 minutes. The colour-bar on the left is a reference for the intensity**

### 5.3 Mode-Locked Laser by Using Znq<sub>2</sub> as SA at 1.5 μm Region

#### 5.3.1 White Pulse Operation

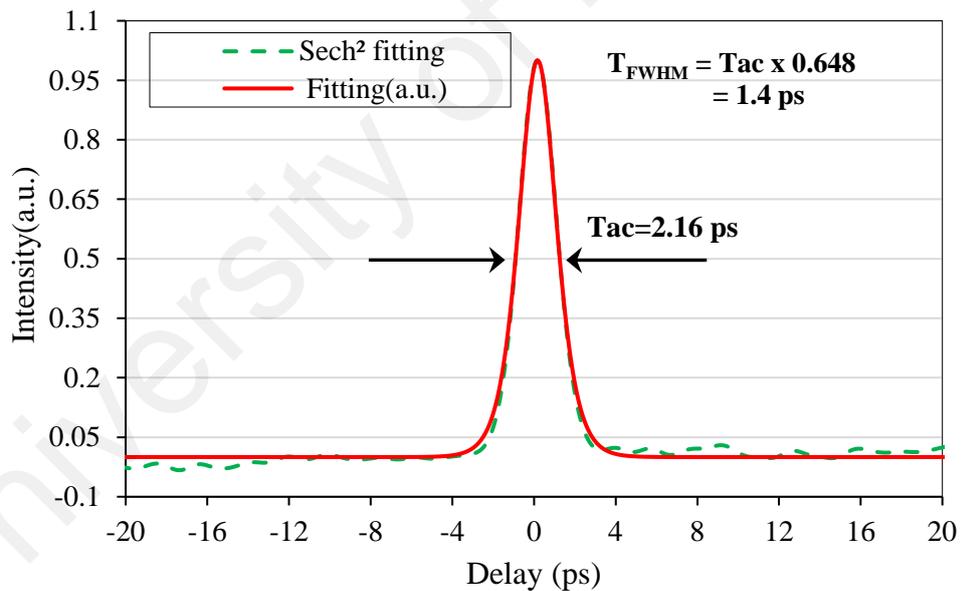
In this section, a mode-locked EDFL is demonstrated using Znq<sub>2</sub> as SA to operate at 1.5 μm region. A stable soliton mode-locked pulse train was produced as the LD intensity gradually increased to 15 mW. Figure 5.7 - Figure 5.13 summarize the performance of the mode-locked laser. The cavity configuration used in this experiment is similar to the one presented in Figure 5.1, except the cavity length and optical coupler (OC) used in this experiment is 59.5 m and 90:10, respectively. The laser produced a soliton optical spectrum at a central wavelength 1560.6 nm with a 3-dB bandwidth of 2 nm, see figure 5.7. The soliton mode-locked operation is due to the interplay of self-phase modulation and group velocity dispersion, which is facilitated by the anomalous cavity dispersion.



**Figure 5.7: Optical spectrum of mode-locked operation based on Znq<sub>2</sub> SA**

This is confirmed by the presence of Kelly sidebands on the spectrum (Lazaridis, Debarge, & Gallion, 1995). The spectrum presents symmetrical Kelly sidebands centred at 1560.6 nm. The measured distance ( $\Delta d$ ) between the central wavelength and the 1<sup>st</sup>, 2<sup>nd</sup> and 3<sup>rd</sup> order Kelly sidebands are  $\pm 3.65$  nm  $\pm 5.9$  nm and  $\pm 6.97$  nm, respectively

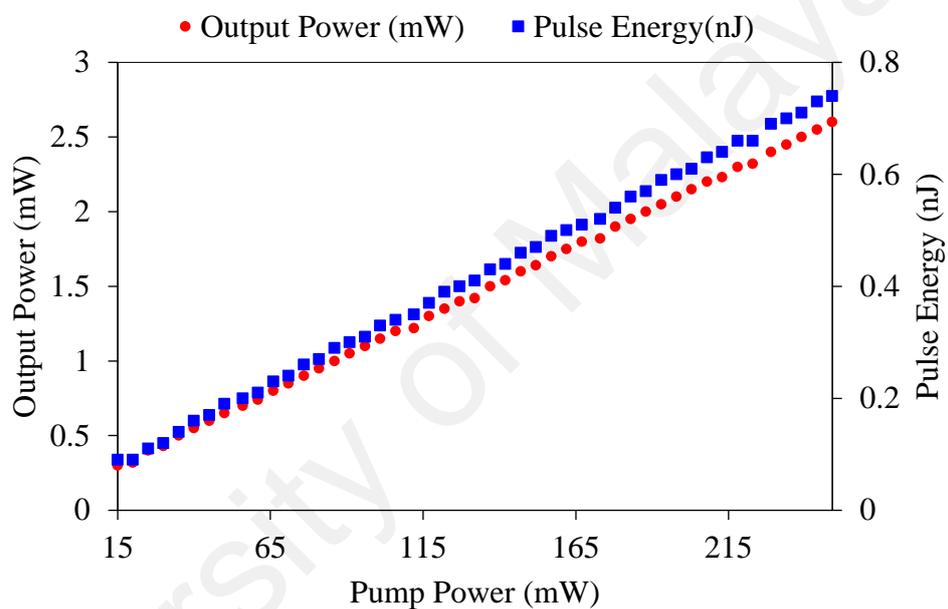
These values are very close to the theoretical values, which were obtained at 3.7 nm, 5.7 nm and 7 nm, respectively. They were calculated based on the relation between the ( $\Delta d$ ), central wavelength, pulse duration and total net dispersion of the cavity as described in Ref. (Smith, Blow, & Andonovic, 1992). A highly-sensitive auto-correlator was used to accurately measure the pulse width. The autocorrelation trace with  $\text{sech}^2$  fitting is given in Figure 5.8. The pulse has a  $T_{\text{FWHM}}$  of 1.46 ps, as the full width at half maximum (FWHM) duration of the autocorrelation trace ( $T_{\text{ac}}$ ) is 2.16 ps. The time-bandwidth product (TBP) is calculated to be 0.359, which is larger than the transform-limited value of 0.315, indicating that the generated pulses are slightly chirped. The calculated pulse width is 1.27 ps, which is very close to the measured value.



**Figure 5.8: Autocorrelation trace of the mode-locking operation based on Znq<sub>2</sub> SA**

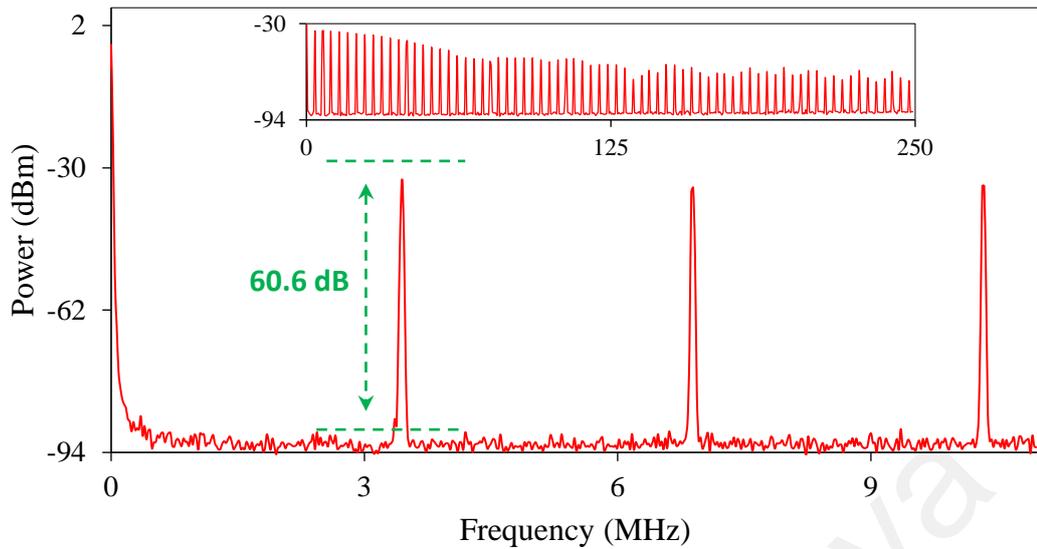
The ultrafast pulsing was produced as the LD intensity was increased from 15 mW to 249 mW. As the LD intensity is lowered below 15 mW, the pulsing operation was disappeared and only continuous wave (CW) operation was produced as observed on the oscilloscope. Similarly, the pulsing operation was disappeared when we raised the LD

intensity above 249 mW and up to the maximum available of 350 m. That is attributed to the bleaching of the SA. When we reduced the LD intensity below 250 mW, the laser restored the mode-locking operation. Hence, we concluded that the SA damage threshold is higher than 350 mW. If the SA was damaged, no mode-locking operation could be produced. The average output intensity and pulse energy are increased linearly from 0.3 mW and 0.1 nJ to 2.6 mW and 0.74 nJ, respectively, see Figure 5.9.



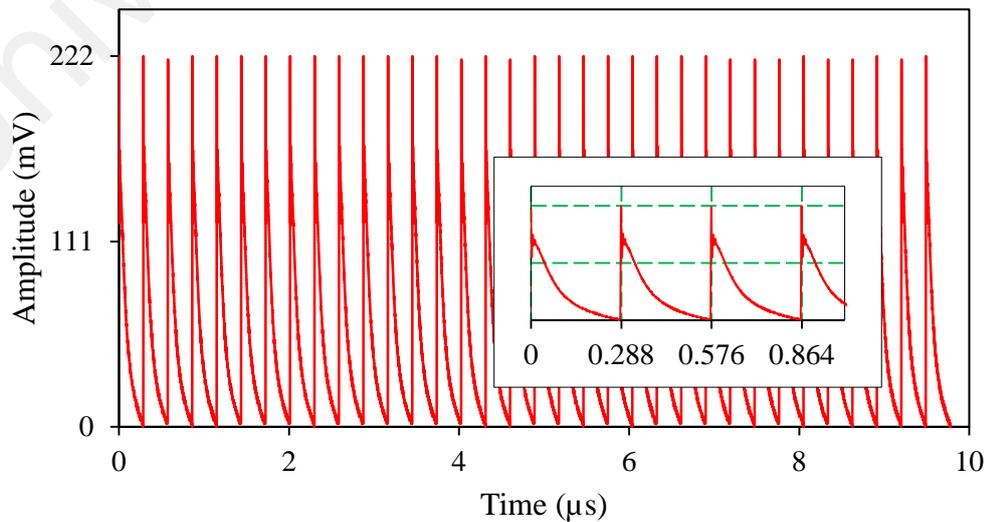
**Figure 5.9: Output power and pulse energy as a function of LD power**

The RF spectrum with a span of 250 MHz and 11 MHz is given in Figure 5.10. It indicates that the laser has a repetition rate of 3.5 MHz. The spectrum also shows a high number of cavity harmonics that the intensity decreased in relative to the fundamental frequency, which suggests a good and stable mode-locking operation with a small pulse width. Additionally, the laser has an excellent signal to noise ratio of 60.6 dB, while the short-span RF spectrum showed no Q-switching instabilities. This proves the stable mode-locking operation.



**Figure 5.10: RF spectrum at a span of 11 MHz. The inset figure shows the spectrum in a span of 250 MHz**

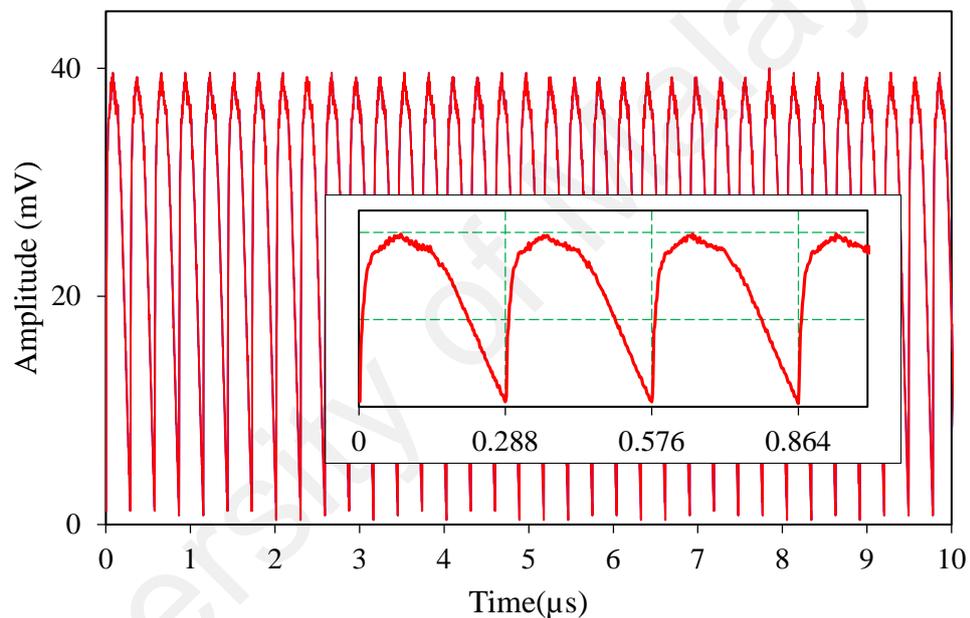
Figure 5.11 shows the typical pulse train of the mode-locked laser within 10  $\mu\text{s}$  span. The pulse width measured by the oscilloscope is 102 ns, which is due to the limited resolution. The pulse train produced a stable operation in which no instabilities nor is distortion observed. The inset Figure shows the pulse train in a shorter span of 1  $\mu\text{s}$ . As illustrated in the inset Figure, the pulses have a pulse period of  $\sim 288$  ns, which it coincides with the repetition rate and the cavity length.



**Figure 5.11: Output pulse train of the mode-locked laser based on Znq<sub>2</sub> SA**

### 5.3.2 Dark Pulse Operation

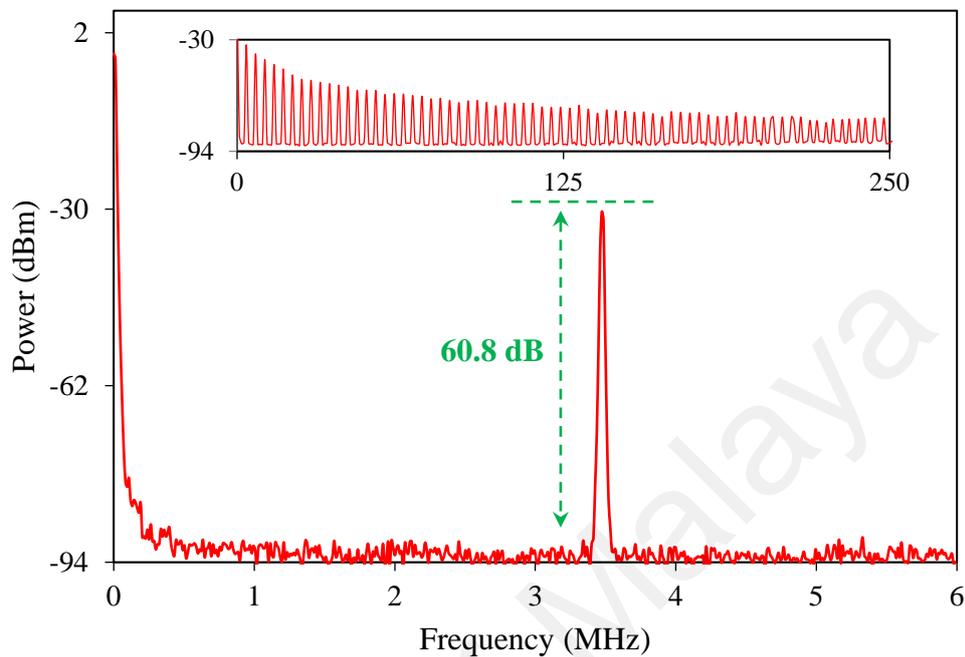
In this experiment, the mode-locked fiber laser could generate not only the bright pulses but also dark pulses, by appropriately managing the polarization state of the cavity. The oscilloscope trace of the dark pulse train is given in Figure 5.12. Obvious dark pulses have appeared. The pulse period is 288 ns, which is the same as in the bright pulses, determined by the cavity length. No bright pulses are observed on the pulse train, on the other hand, the saw-tooth like pulse train is attributed to the noise produced by the photo-detector.



**Figure 5.12: Output dark pulse train based on Znq<sub>2</sub> SA**

The RF spectrum of the dark pulses is presented in Figure 5.13. The laser maintained an excellent SNR of 60.8 dB. The experiment shows that Znq<sub>2</sub>-SA can act as a promising mode-locker, providing a bright and dark pulse operation. To confirm that the pulsing operation is attributed to the Znq<sub>2</sub>-SA, the Znq<sub>2</sub>-SA was removed from the cavity. In this case, no bright or dark pulsing operation was observed on the oscilloscope, no matter how the polarization of the cavity or LD intensity was changed. Further investigations of this

SA in ultrafast lasers are needed, as this is the first demonstration of bright and dark pulsing operation using Znq<sub>2</sub>-SA.



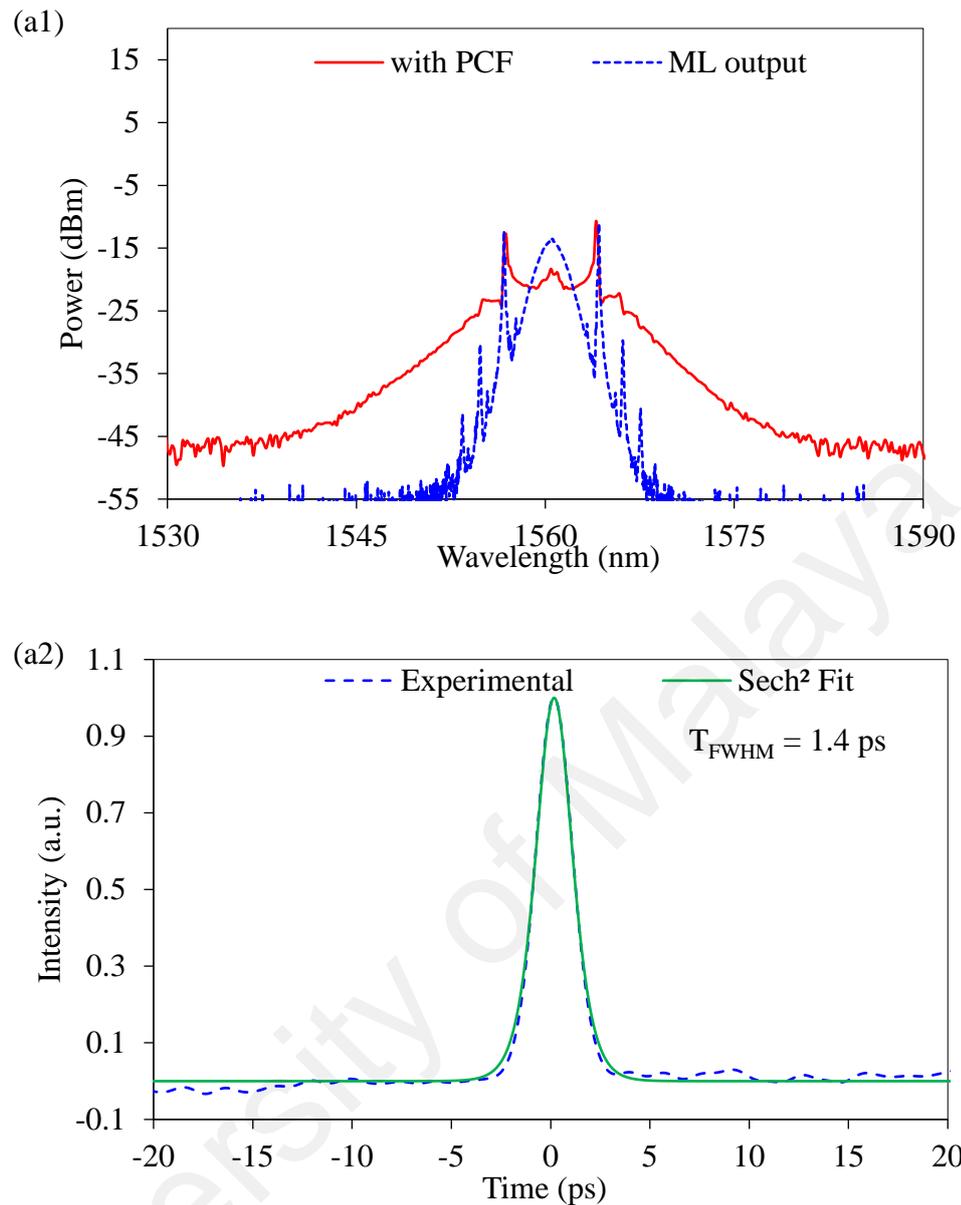
**Figure 5.13: RF spectrum of dark mode-locked operation at a span of 6 MHz. The inset figure shows the spectrum at a span of 250 MHz**

### 5.3.3 Supercontinuum Pulse Generation with Znq<sub>2</sub> Based EDFL

In this subsection, the developed Znq<sub>2</sub> based EDFL is used as a pump to generate a supercontinuum (SC) light. SC generation has attracted much of research interest in recent years due to its various applications, such as spectroscopy, optical communications, optical frequency metrology and mobile clockwork, (Keren, Brand, Levi, Levit, & Horowitz, 2002). SC can be generated by using noise-like pulses (NLPs) (A.-P. Luo et al., 2015), short pulses (Han Zhang, Tang, Zhao, Wu, & Tam, 2009) and ultra-short pulses (Sumimura, Genda, Ohta, Itoh, & Nishizawa, 2010), and nonlinear propagation media like standard fiber (SMF) (Zaytsev et al., 2013), zero-dispersion fiber, tapered fiber (Liao et al., 2012), highly nonlinear fiber (HNLF) (Liao et al., 2012) and photonic crystal fiber (PCF) (Travers, Rulkov, Cumberland, Popov, & Taylor, 2008).

In this work, output pulses from the developed  $\text{Znq}_2$  based EDFL were launched into a 100 m long PCF to generate SC. Figure 5.14 (a1) shows the output spectrum of the pulses before and after the PCF at the maximum input pump power of 249 mW. It is observed that the output spectrum was significantly broadened after the PCF. Figure 5.14 (a2) and (a3) shows the autocorrelation trace of the laser, which was obtained before and after the PCF respectively. The  $T_{\text{FWHM}}$  was reduced to 640 fs after the supercontinuum generation as observed after the PCF. To improve the performance of supercontinuum, an erbium-doped fiber amplifier (EDFA) device is used to amplify the input mode-locked laser, which functioned as the pump. The average output power was increased to 32 mW after amplification. Figure 5.14 (b1) compares the output optical spectrum before and after amplification. It is observed that the output spectrum was also broadened after the amplification.

Figure 5.14 (b2) shows the output spectrum of the amplified pulses at a wider span, which indicates the spectral broadening has covered a wider range from 1200 nm to 1750 nm. The OSA used in this experiment had a wavelength range up to 1750 nm. Thus, it is believed that the spectrum is widened beyond 1750 nm. Figure 5.14 (b3) shows the autocorrelator trace of the pulses, which indicates  $T_{\text{FWHM}}$  of about 2.78 ps. The pulse width obtained was broadened as compared to unamplified input pulses and the previous SC generation after PCF (without amplification). To enhance the SC operation, the amplified pulses were injected into the PCF. The spectrum had an enhanced broadening, see Figure 5.14 (c1) and (c2). While the pulse width is reduced to 260 fs as shown in Figure 5.13(c3)



**Figure 5.14 (a1) Output spectrum for normal mode-lock (ML) operation and when 100 m PCF attached to the output of the cavity, (a2) autocorrelation trace for normal ML operation, (a3) autocorrelation trace when the 100 PCF attached, (b1) output spectrum for normal ML operation and when EDFA attached to the output of the cavity at a span of 60 nm, (b2) output spectrum for normal ML operation and when EDFA attached to the output of the cavity at span 550 nm, (b3) autocorrelation trace for normal ML operation and when EDFA attached to the output of the cavity, (c1) output spectrum for normal ML operation and when EDFA and 100 PCF are attached to the output of the cavity at span of 60, (c2) output spectrum for normal ML operation and when EDFA and 100 PCF are attached to the output of the cavity at a span of 550 nm, and (c3) autocorrelation trace when EDFA and 100 PCF are attached to the output of the cavity**

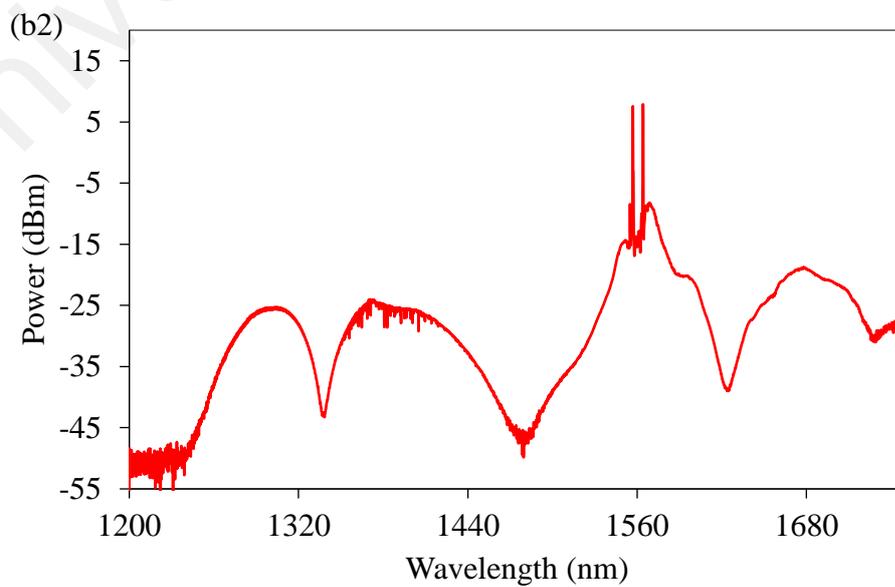
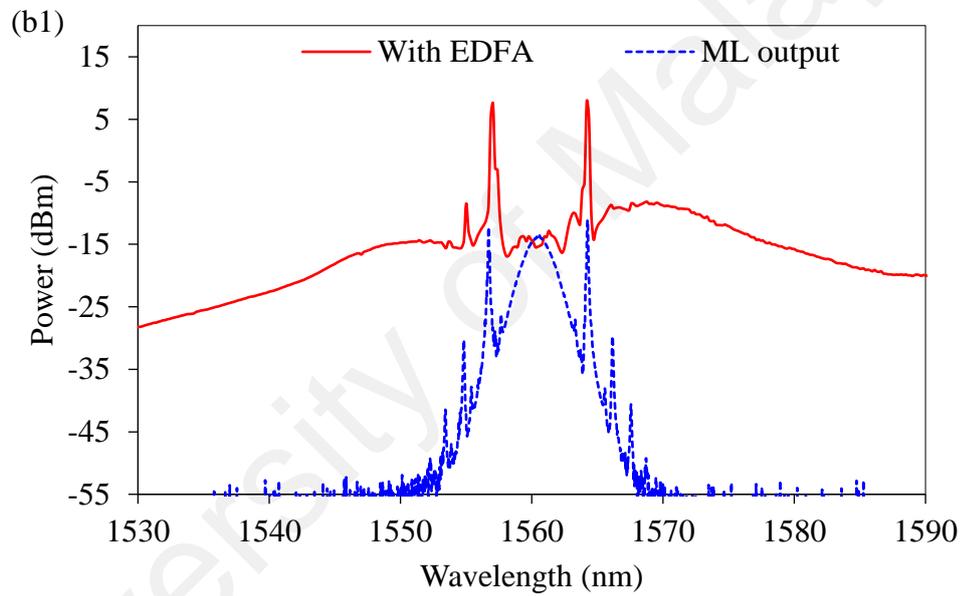
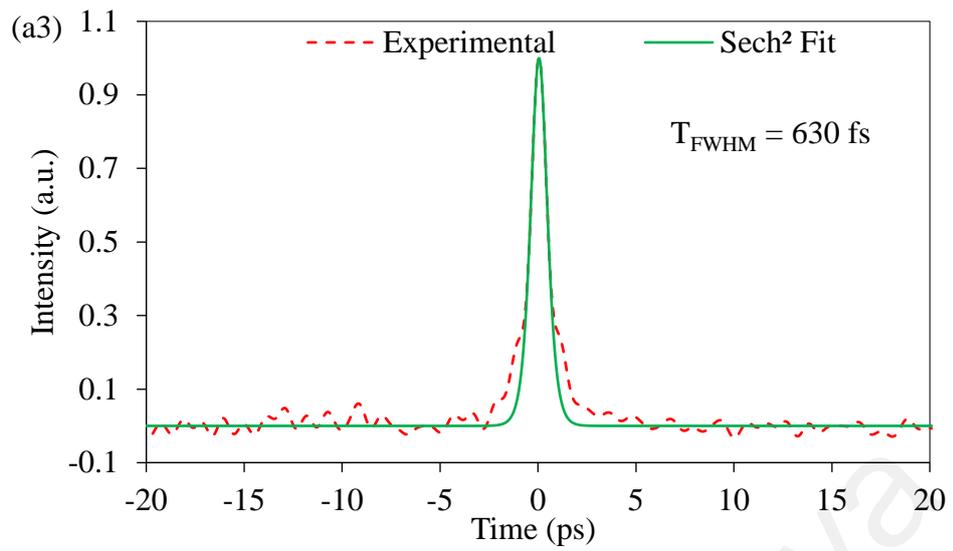


Figure 5.14, continued

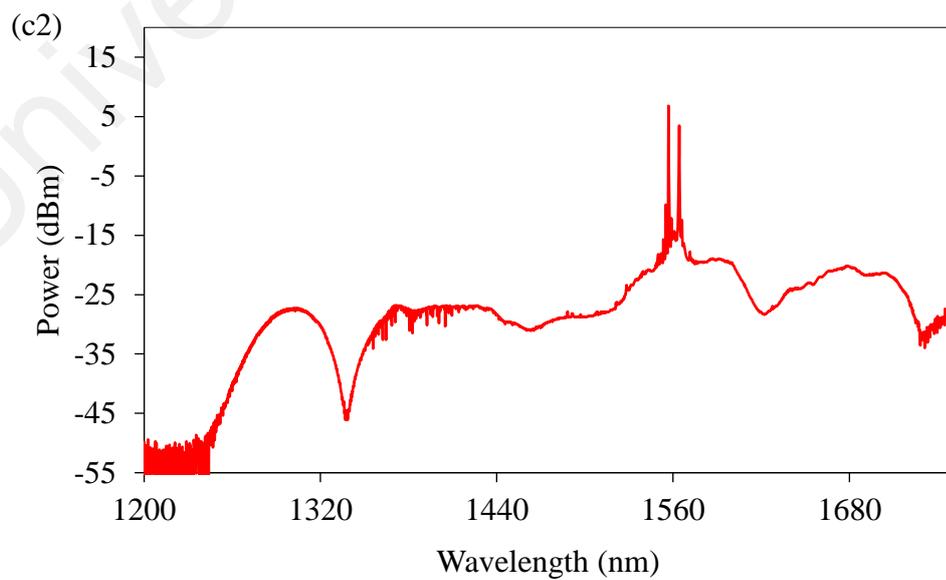
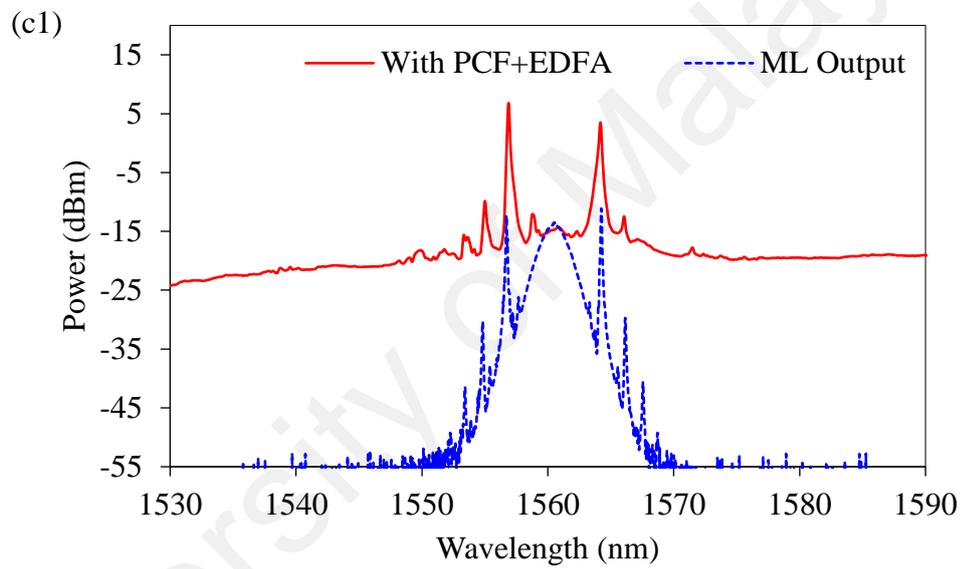
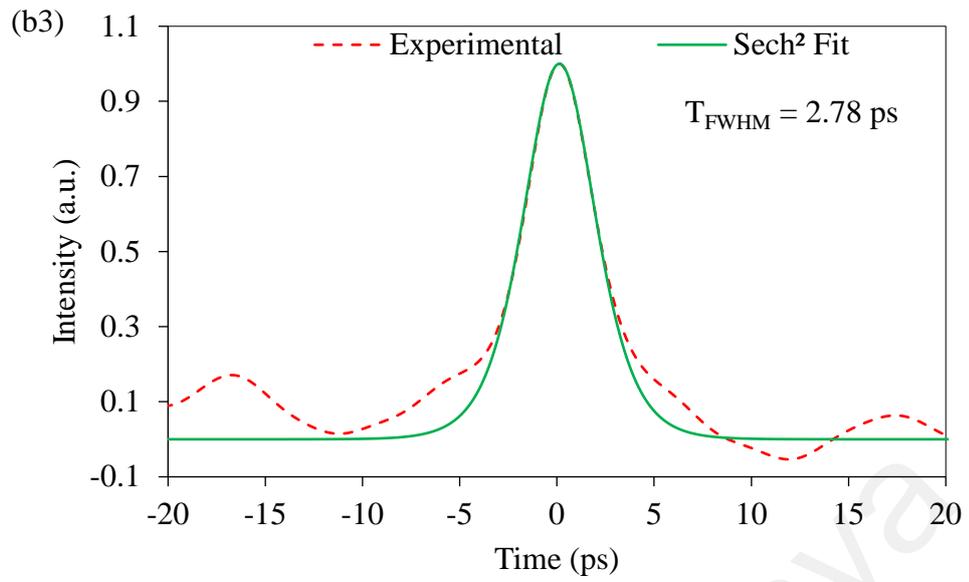
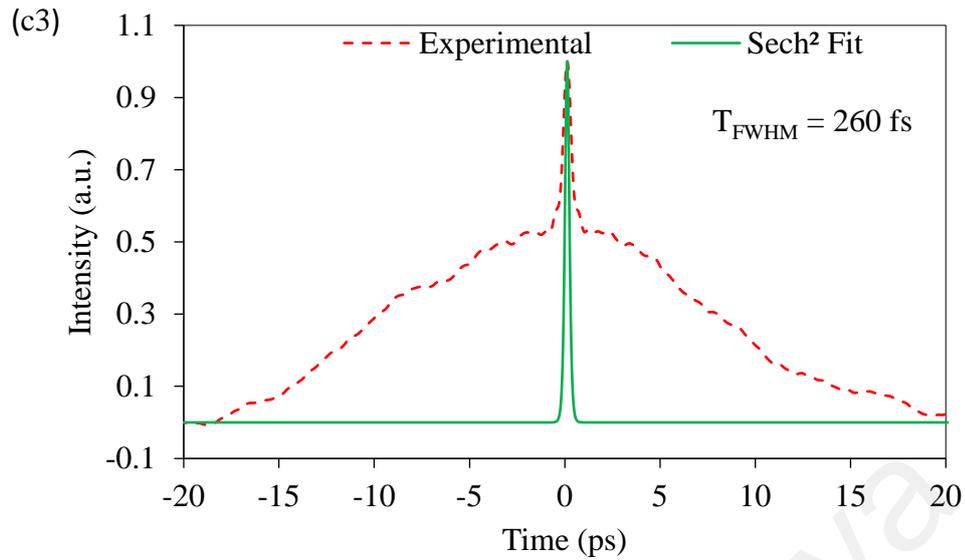


Figure 5.14, continued

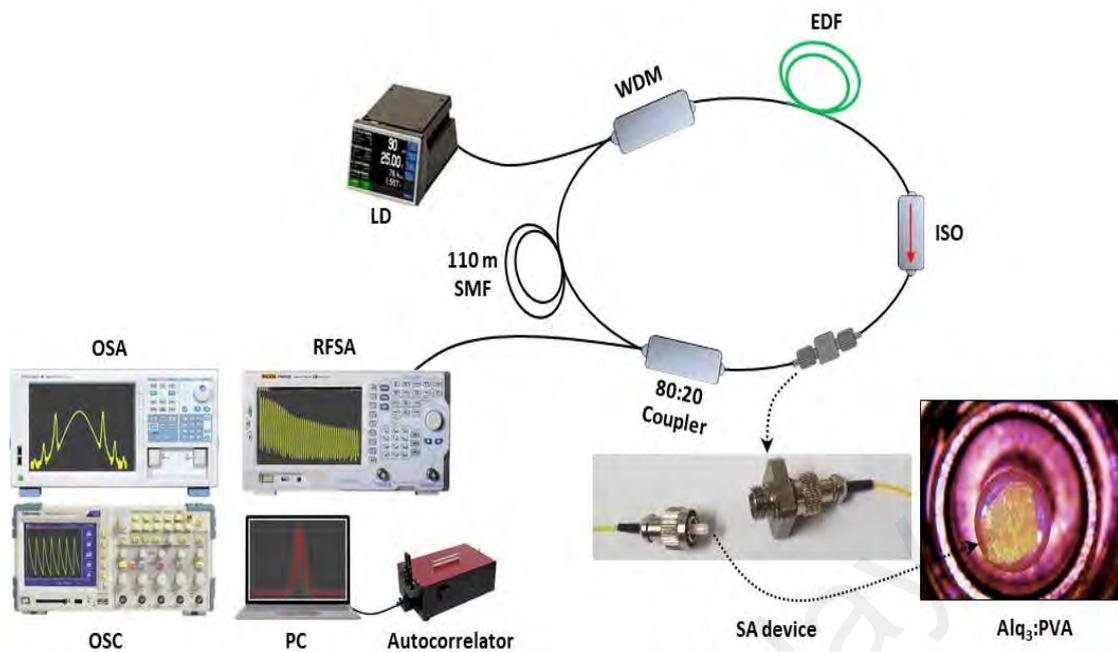


**Figure 5.14, continued**

## 5.4 Mode-Locked EDFL with Alq<sub>3</sub> Saturable Absorber

### 5.4.1 Experimental Setup

The configuration of the proposed fiber laser is shown in Figure 5.15. The fiber laser cavity consists of a 2 m long Erbium-doped fiber (EDF) as a gain medium, 0.5 m long WDM fiber and 110 m long standard SMF. The EDF has a core diameter of 4  $\mu\text{m}$ , NA of 0.16, and erbium ion absorption of 23  $\text{dB m}^{-1}$  at 980 nm. The cavity length was 112.5 m with net cavity dispersion of  $-2.356 \text{ ps}^2$ . The group velocity dispersion of the EDF, WDM and SMF were 27.6,  $-48.5$  and  $-21.7 \text{ ps}^2/\text{km}$ , respectively. Pump light at 980 nm was injected from a laser diode (LD) into the cavity via the WDM. An isolator (ISO) was used to prevent light reflection, and an 80:20 optical coupler was used to couple out 20% of intra-cavity power. The output optical spectrum, RF spectrum, oscilloscope traces and pulse width were observed by optical spectrum analyzer (OSA) with a spectral resolution of 0.07 nm, RF spectrum analyzer (RFSA) (Anritsu MS2683A), real-time oscilloscope (OSC) (GW INSTEK GDS-3352, 350 MHz bandwidth with 1.2 GHz photodetector) and an autocorrelator, respectively.

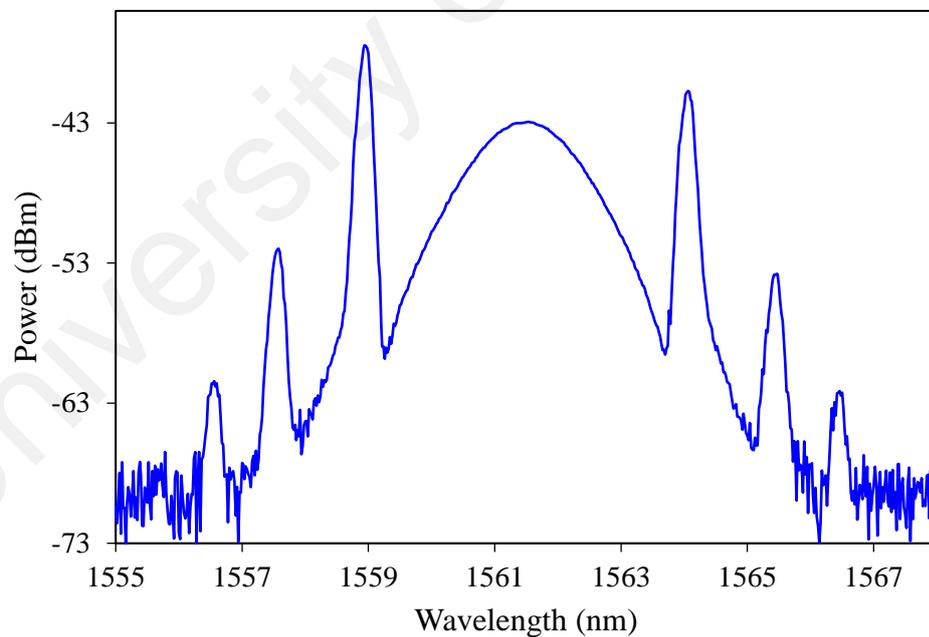


**Figure 5.15: EDFL cavity setup with Alq<sub>3</sub> SA as a mode-locker**

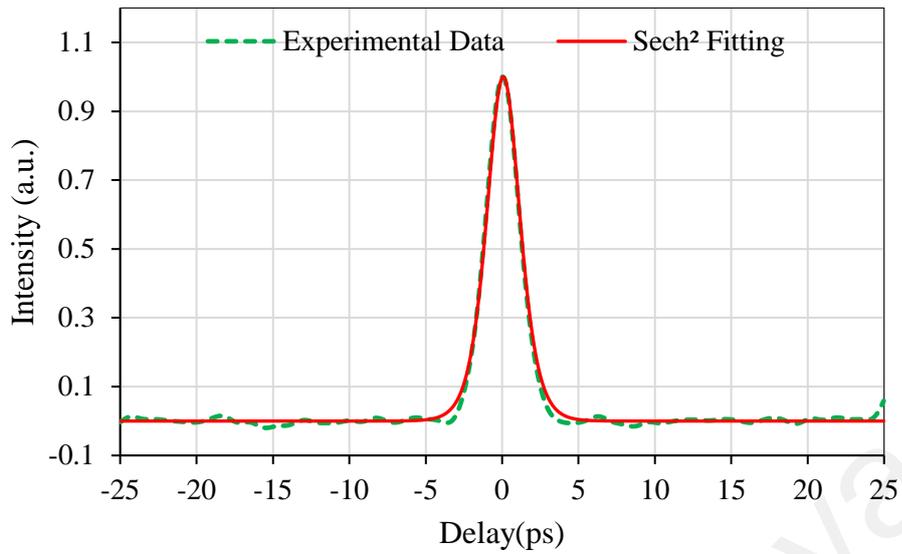
#### 5.4.2 1.6 Picosecond Mode-Locked Pulse Generation

The proposed Alq<sub>3</sub> based EDFL produced a CW operation at an input LD power of 10 mW, while the mode-locked operation was obtained and sustained as the LD power increased from 25 mW to 274 mW. When the LD power raised to its maximum of 350 mW, the mode-locked operation was disappeared, i.e. the CW operation was observed again in which no pulsing was observed in the oscilloscope and the soliton shape turned into single peak centred at 1561.5 nm wavelength. As we reduced the LD power below 274 mW, the mode-locked pulsing was produced again. Hence, the damage threshold of the proposed soliton laser was confirmed to be above 350 mW. It is believed that if the LD power has damaged the SA, there is no mode-locking operation can be produced when the LD power was reduced back in the range between 25 mW to 274 mW. However, this is not the case in our observation. The reason for the switching in the laser regime is attributed to the SA was fully saturated. In another experiment, the Alq<sub>3</sub>: PVA film has been removed from the cavity to verify that Alq<sub>3</sub> caused the pulsing operation. In this case, no pulsing was observed despite tuning the LD power over a wide range and

changing the polarization inside the cavity by twisting and bending optical fibers. By inserting the Alq<sub>3</sub>: PVA back into the fiber laser cavity, the mode-locking pulses was observed again. It is concluded that Alq<sub>3</sub> was purely responsible for the mode-locking operation rather than other components. The output optical spectrum of the pulsing operation showed that the fiber laser produced a soliton mode-locking regime, see Figure 5.16. The spectrum shows a typical soliton-like shape with symmetrical Kelly sidebands centred at 1561.5 nm. The separation between the central wavelength and the 1<sup>st</sup>, 2<sup>nd</sup> and 3<sup>rd</sup> order Kelly sidebands are  $\pm 2.55$  nm,  $\pm 3.95$  nm and  $\pm 4.94$  nm, respectively. The autocorrelation trace of the mode-locking operation is given in Figure 5.17. The pulse width has  $T_{FWHM}$  of 1.6 with an estimated TBP of 0.350. The TBP is higher than the transform-limited prediction of 0.313, showing that the pulses were chirped. The minimum possible pulse width duration (calculated mathematically) is  $\sim 1.43$  ps.

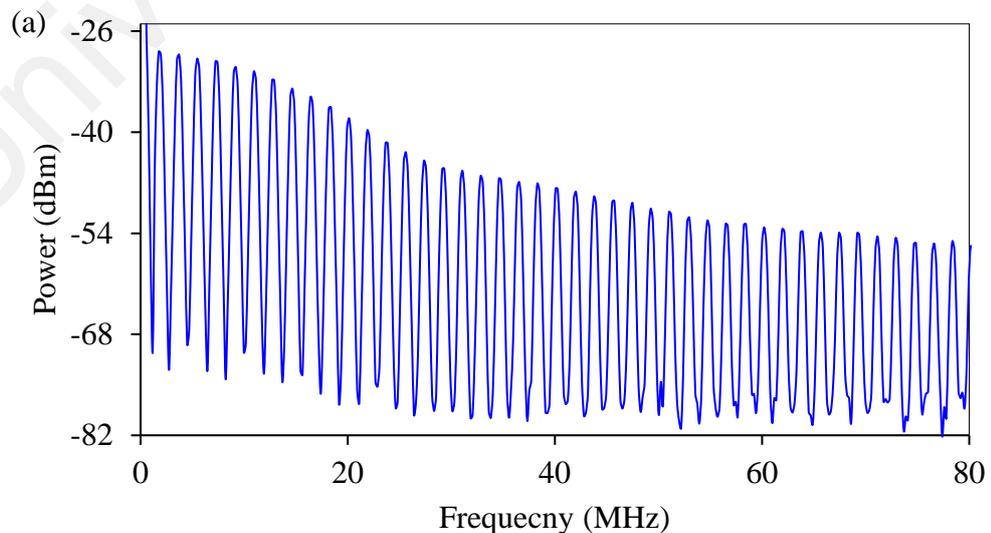


**Figure 5.16: Output spectrum from the proposed Alq<sub>3</sub> based mode-locked EDFL**

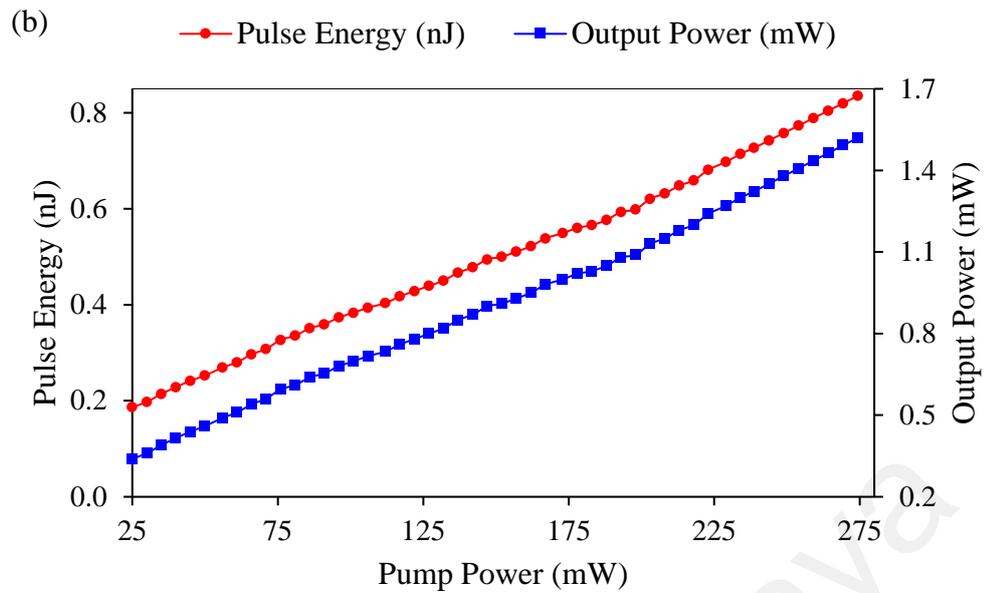


**Figure 5.17: Autocorrelator trace of the mode-locked pulses from the Alq<sub>3</sub> based EDFL**

Figure 5.18 (a) shows the measured RF spectrum within a span of 80 MHz, which indicates the fundamental frequency operates at a repetition rate of 1.8 MHz with a signal to background noise ratio (SNR) of 41.5 dB. The spectrum also shows a high cavity harmonic as well as a high spectral purity without significant spectra modulation, which proves the stability of the mode-locking operation. The output pulse energy and output power against the input LD power are given in Figure 5.18 (b).

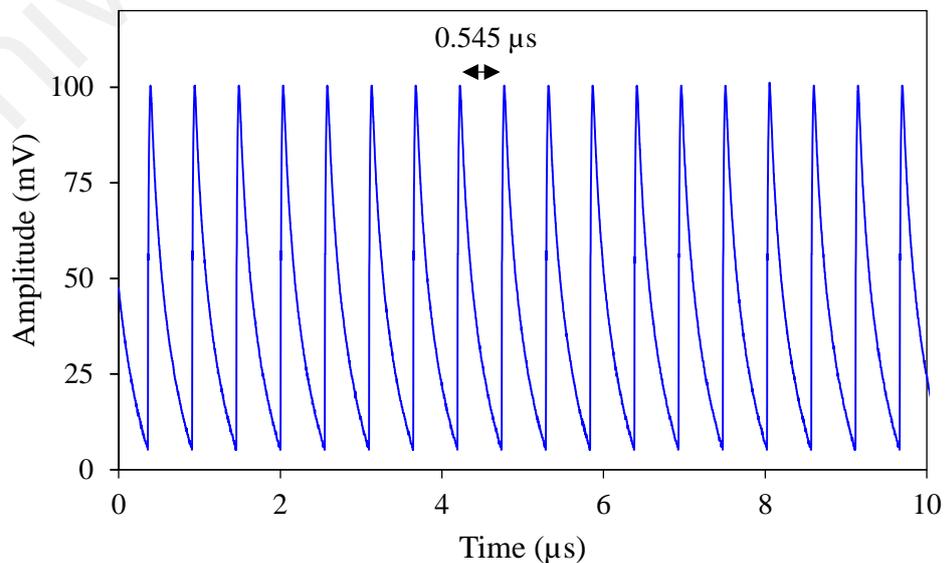


**Figure 5.18: (a) RF spectrum of the proposed Alq<sub>3</sub> based mode-locked EDFL and (b) pulse energy and output power as a function of input LD power**



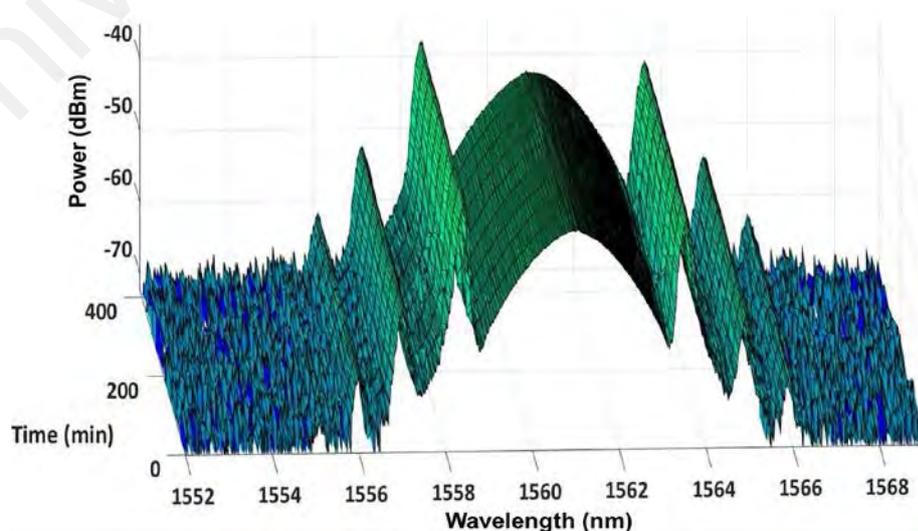
**Figure 5.18, continued**

As the LD power was tuned from 25 mW to 274 mW, the pulse energy can be raised linearly from 0.19 nJ to 0.84 nJ and the output power increased from 0.34 mW to 1.52 mW. The oscilloscope was used to check the temporal pulse train as shown in Figure 5.19. The pulse train has a very uniform distribution, which indicates that the laser operation was very stable. The pulse period was measured to be 545 ns, which it is in coincident with the pulse repetition rate and cavity length.



**Figure 5.19: Output pulse train of mode-locked laser based on Alq<sub>3</sub> as SA**

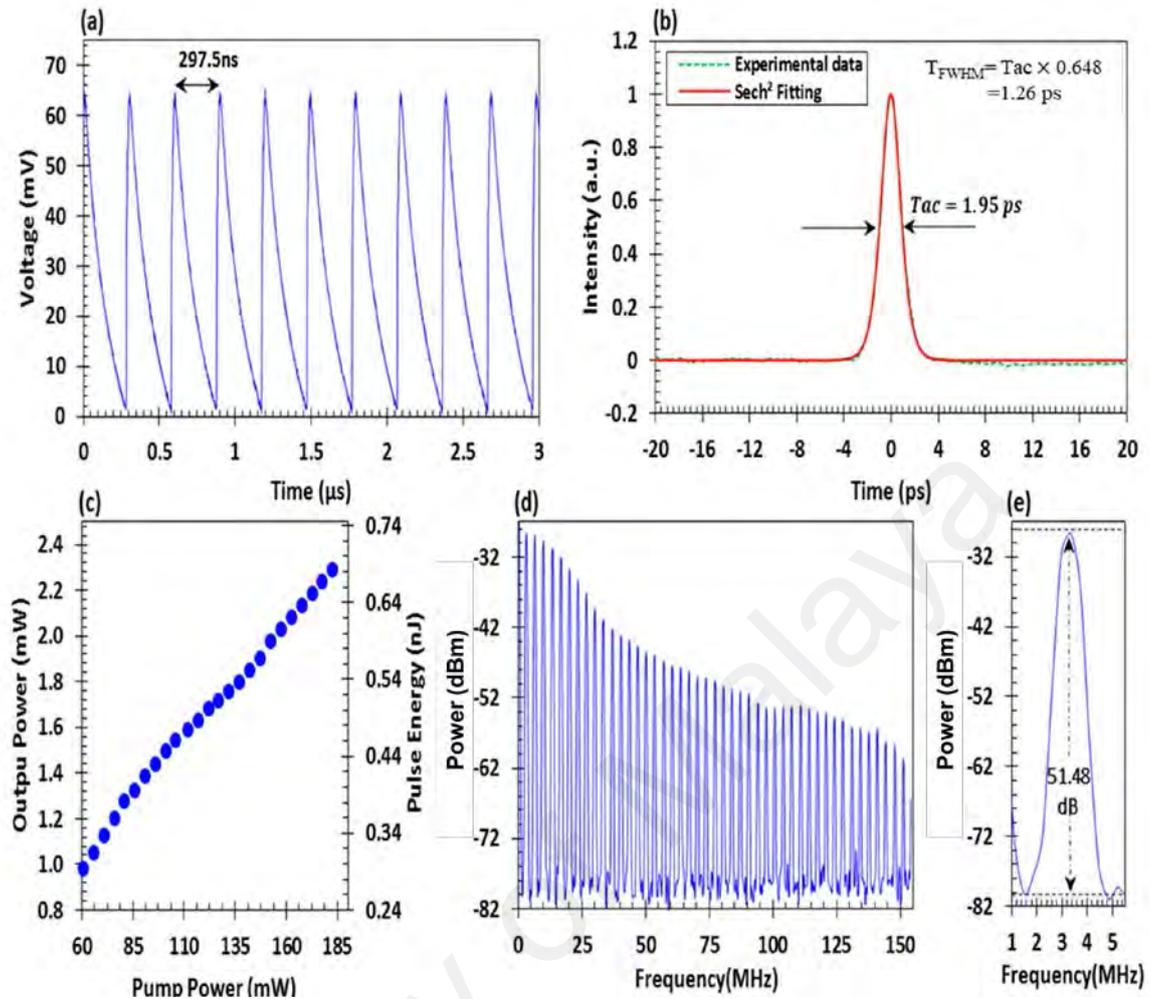
The pulse train showed a sawtooth-like aspect, this is attributed to the noise produced by photo-detector attached to the oscilloscope. This assumption agrees with the high SNR and the short pulse width produced by the fiber laser. To check the long-term operation of Alq<sub>3</sub>: PVA SA, the optical spectrum was sampled every ~ 42 minutes for 420 minutes, as shown in Figure 5.20. The figure depicts a very stable operation in which all the samples had the same central wavelength and the amplitude of 1561.5 nm –42.9 dBm, respectively. Additionally, the samples show that the Kelly sidebands kept the same optical intensity and wavelengths throughout the sampling process, in which the 1<sup>st</sup> order had intensities of –37.6 dBm and –40.83 dBm (at 1558.95 nm and 1564.05 nm), the 2<sup>nd</sup> order had intensities of –52.0 dBm and –53.83 dBm (at 1557.55 nm and 1565.45 nm) and 3<sup>rd</sup> order had intensities of –61.6 dBm and –62.26 dBm (at 1556.56 nm and 1566.44 nm), respectively. This further proves the stability of the mode-locking operation and indicates that Alq<sub>3</sub>: PVA can provide the same performance and can also withstand a long-term illumination. It should be mentioned that a mode-locked operation with a wider pulse width duration and lower repetition rate was achieved with a longer cavity length. It is believed that by further decreasing the cavity length, an enhanced fiber laser operation can be obtained.



**Figure 5.20: Output optical spectrum sampled for 420 minutes**

### 5.4.3 1.26 Picosecond Mode-Locked Pulse Generation

In this subsection, the performance of the Alq<sub>3</sub> based mode-locked EDFL is investigated by reducing the cavity length from 112.5 m to 61.5 m. It was obtained by removing 50 m long SMF from the previous experimental setup. A self-starting soliton mode-locking was produced at 61 mW and the operation was sustained as the input power is increased up to 183 mW. Figure 5.21 (a) illustrates the output pulse train of the fiber laser. The peak to peak spacing is about 297.5 ns, which is equivalent to the cavity round-trip time and pulse repetition rate of 3.36 MHz and coincides with the estimated fundamental frequency of the cavity. The oscilloscope gave a much wider pulse duration (107.1 ns) than the actual value, which is due to the limited resolution of the oscilloscope. It can be observed that the pulse train has a very uniform pulse amplitude which indicates a stable mode-locking operation. To measure the pulse duration accurately, we used a highly-sensitive auto-correlator to measure the autocorrelation trace, see Figure 5.21 (b). The pulse width has  $T_{FWHM}$  of 1.26 ps as estimated from the FWHM duration of the autocorrelation trace ( $T_{ac}$ ) of 1.95 ps and assuming  $\text{sech}^2$  curve fitting. The TBP is calculated to be 0.324, which is slightly deviated from the transform-limited TBP of 0.315, suggesting that the pulse was slightly chirped in which the theoretical pulse-width was calculated to be 1.22 ps. Figure 5.21 (c) illustrates the relation between LD power versus average output power and output pulse energy. The result depicts that as the LD power increases, the average output power raises linearly from 0.98 to 1.87 mW. The figure also shows the linear increment of pulse energy; it was raised from 0.29 to 0.56 nJ as the input power was increased. As the LD power was increased to exceed 183 mW up to the maximum available power of 350 mW, the mode-locked EDFL became unstable and disappeared. This is due to the over-saturation of the Alq<sub>3</sub>: PVA film. When the pump power was decreased within 61 mW to 183 mW, the mode-locked pulsing was re-produced again, indicating that thin-film was not damaged.



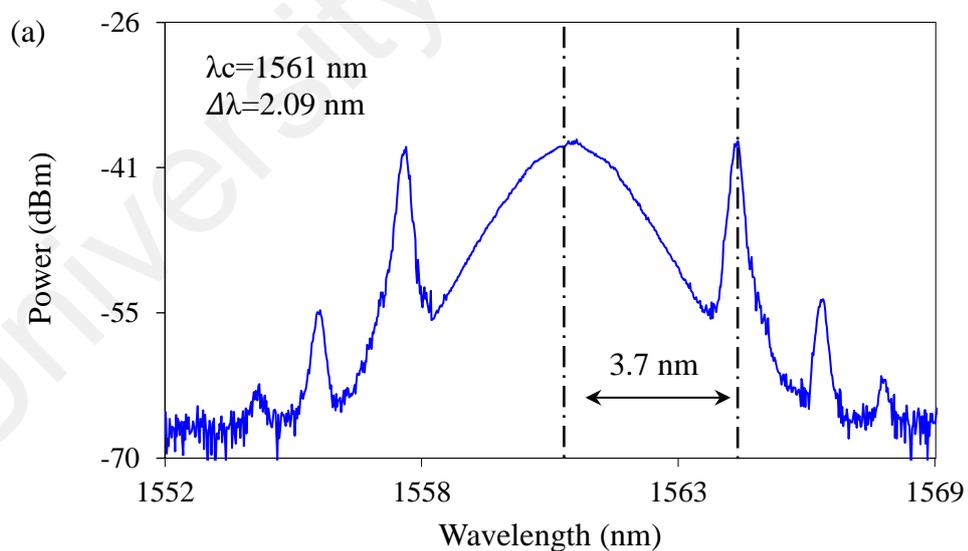
**Figure 5.21: Picosecond pulsing laser in 61.5 m cavity length with 80:20 OC: (a) oscilloscope trace of the soliton laser; (b) autocorrelator trace showing a pulse duration of 1.26 ps ; (c) pulse energy and average output power against LD power; RF spectrum with a span of (d) 148 MHz, and (e) 4.5 MHz**

To further check the stability of the laser, the RF spectrum was recorded over a span of 148 MHz, as shown in Figure 5.21 (d). The Figure depicts high cavity harmonics without any noticeable instabilities. Additionally, the harmonics decreased relative to the fundamental frequency, proving good mode-locking performance. Figure 5.21 (e) shows the fundamental frequency of 3.36 MHz has a very good SNR of 51.48 dB which further indicates a stable and excellent pulsing operation. The net cavity dispersion in this experiment is anomalous, resulting in soliton mode-locked pulses generation through the interplay of self-phase modulation and group velocity dispersion. This is confirmed by the presence of Kelly sidebands on the spectrum (Lazaridis et al., 1995), see Figure 5.22

(a). The spectrum has a central wavelength of 1561 nm with a 3-dB bandwidth of 2.09 nm. The distance between the centre wavelength and the 1<sup>st</sup> order Kelly sideband ( $\Delta d$ ) is 3.7 nm. The theoretical value for the distance was calculated to be 3.653 nm. This value was obtained or calculated by using the relation between the central wavelength and  $m$ th order of the Kelly sideband position as described in the following equation (Smith et al., 1992);

$$\Delta d = \frac{(1.7627)\lambda^2}{2\pi C \tau} \sqrt{\frac{4m\pi}{|\beta|} \left(\frac{\tau}{1.7627}\right)^2 - 1} \quad (5.1)$$

where  $\lambda$ ,  $\tau$  and  $\beta$  are central wavelength, pulse duration and total net dispersion of the cavity, respectively. Figure 5.22 (b) shows the  $\Delta d$  as a function of the pulse duration for 1<sup>st</sup>, 2<sup>nd</sup> and 3<sup>rd</sup> Kelly sideband orders. There is a slight difference between the measured and calculated values of  $\Delta\lambda$  for the 1<sup>st</sup> order of the Kelly sideband.



**Figure 5.22: Picosecond pulsing laser in 61.5 m cavity length with 80:20 OC: (a) output spectrum; (b)  $m$ th order Kelly sidebands distance to the centre wavelength versus pulse width at a central wavelength of 1561 nm; and (c) optical spectrum sampled for 220 minutes**

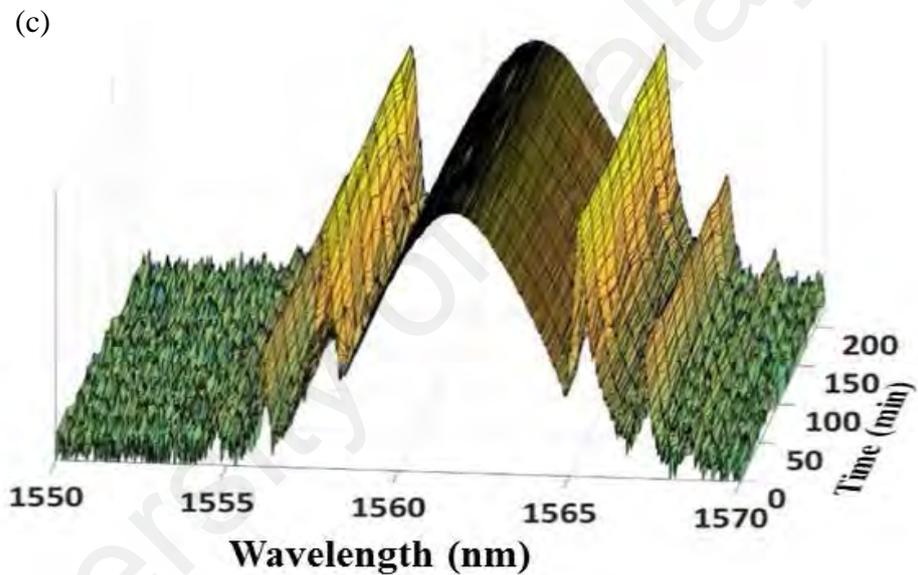
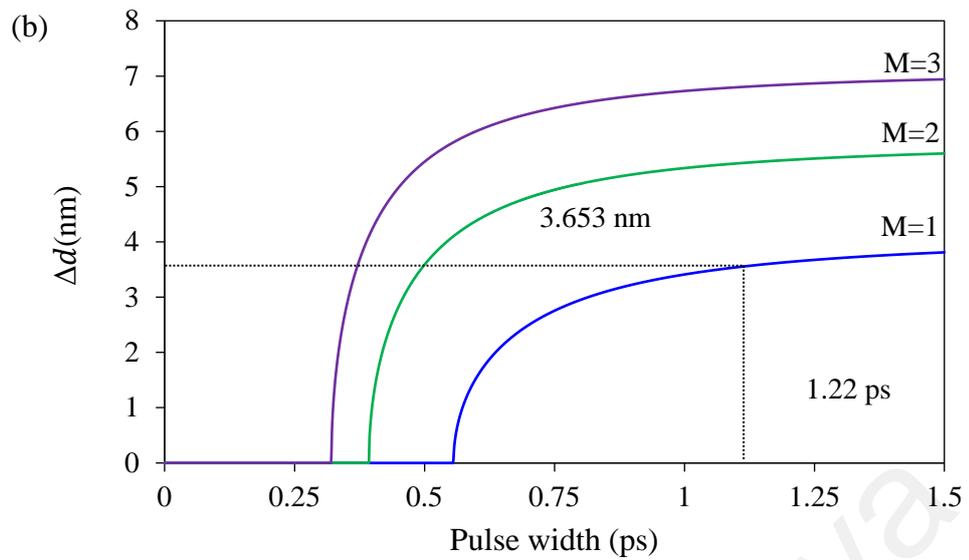


Figure 5.22, continued

The calculated and measured 3-dB values are 2.033 nm and 2.09 nm, which are almost equal. The 3-dB is calculated through

$$\Delta\lambda = \frac{0.315 \lambda^2}{C \tau} \quad (5.2)$$

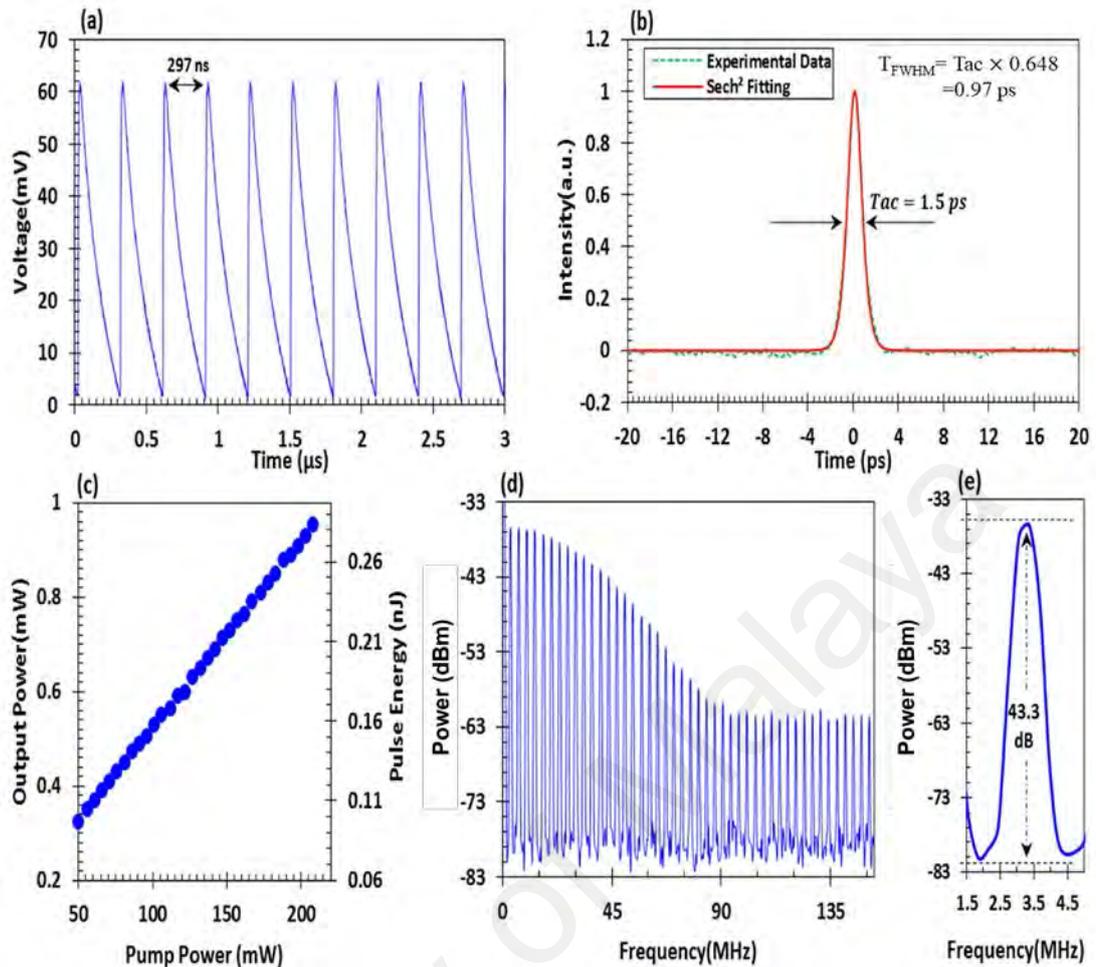
Figure 5.22 (c) shows the long-term performance over 220 minutes at input LD power of 183 mW. The shapes of the plotted spectra are almost like one another, further indicating a very stable mode-locked operation.

It should be noted that no mode-locking was generated when we extracted the SA from the cavity or when we replaced the SA with pure PVA film, even though the LD power was tuned over a wide range and the fiber was twisted and bent accordingly in both cases. We conclude that Alq<sub>3</sub> is responsible for the passive mode-locking operation.

#### 5.4.4 970 Femtosecond Mode-Locked Laser Operation

In this experiment, only 80:20 OC was replaced with 95:5 OC, while the rest of the cavity configurations were as the previous setup. The system maintained almost the same performance except that the pulse width and SNR are reduced to 970 fs and 43.3 dB, respectively, see Figure 5.23. The pulse train maintained the same performance, in which no instabilities are observed, as shown in Figure 5.23 (a). In comparison with the previous experiment, the pulse train revealed by oscilloscope had the same pulse width of about 106 ns and the same pulse period of 297 ns. The pulse period also coincides with the fundamental frequency of 3.36 MHz and equivalent to the cavity round-trip, as the cavity length was the same as in the previous experiment.

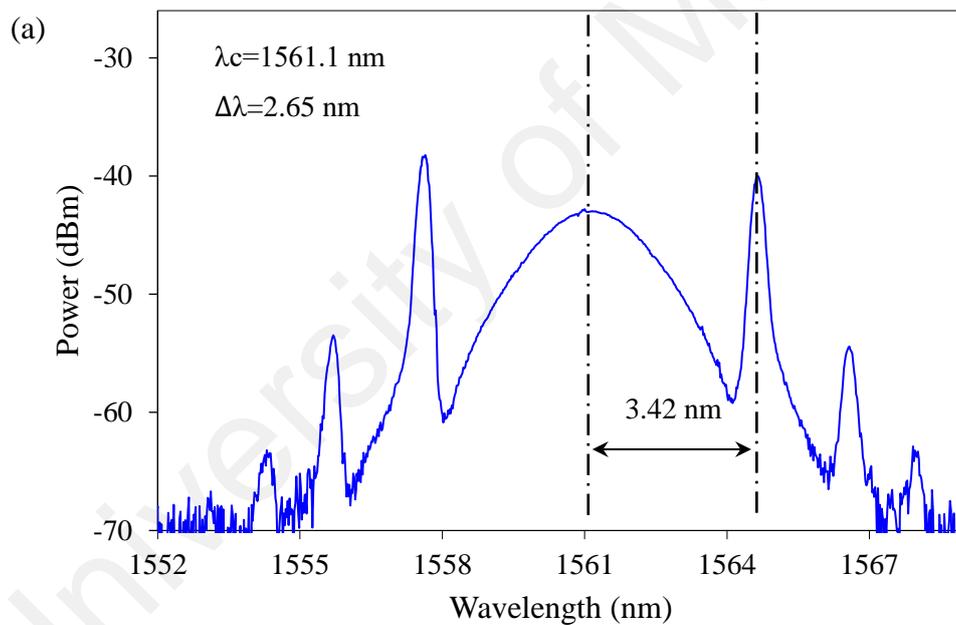
The pulse width  $T_{FWHM}$  was reduced to 970 fs; this reduction in the pulse width is attributed to the utilization of the 95:5 OC. The calculated pulse width is 966 fs, which is almost the same as the measured value as the TBP in this cavity is 0.316. The 95:5 OC extracted a small portion of the signal and increased the oscillating power within the cavity. Correspondingly, the number of modes that can pass through the intensity-dependent transmission mode-locker (the SA) were increased. Hence, the pulse width was reduced, as it is inversely correlated the number of modes that are locked together inside the cavity. Figure 5.23 (b) shows the autocorrelation trace. Like the previous setup, the experimental result is perfectly matched with  $\text{sech}^2$  fitting, which indicates excellent and stable performance.



**Figure 5.23: Femtosecond pulsing laser in 61.5 m cavity length with 95:5 OC: (a) output pulse train; (b) autocorrelator trace showing a pulse duration of 970 fs ; (c) average output power and pulse energy versus LD power; RF spectrum with a span of (d) 153 MHz, and (e) 3.5 MHz**

The output power was reduced as compared with the previous setup since this OC allowed a small portion of the signal to be extracted from the cavity. Correspondingly, the pulse energy was also reduced, as the pulse energy is correlated with average output power. The maximum average output power and maximum pulse energy are about 0.95 mW and 0.28 nJ, respectively, see Figure 5.23 (c). On the other hand, the SNR is reduced to 43.4 dB, which might be attributed to the increase in the non-linearity that is introduced by the 95:5 OC. However, the cavity still produced high harmonics up to 153 MHz, see Figure 5.23 (d). Figure 5.23 (e) shows the fundamental frequency with a span of 3.5 MHz. The output optical spectrum is given in Figure 5.24 (a). In comparison with the previous

experiment, the spectrum is centred at the same wavelength of 1561.1 nm but with a wider 3-dB bandwidth of 2.65 nm. The pulse width is inversely correlated with the 3-dB bandwidth, as indicated by Equ (5.2). As expected, the spectrum showed a reduction in the  $\Delta d$  for the 1<sup>st</sup> order Kelly sideband, since the pulse width was reduced while the cavity dispersion and central wavelength were maintained as in the previous experiment, see Figure 5.24 (b). The measured and calculated values of  $\Delta d$  were 3.42 nm and 3.357 nm. Figure 5.24 (c) shows the long-term stability of the mode-locking operation. The spectrum was sampled every ~18 minutes for the total duration of 176 minutes. The samples also show a very stable operation.



**Figure 5.24: Femtosecond pulsing laser in 61.5 m cavity length with 95:5 OC: (a) output spectrum; (b) mth order Kelly sidebands distance to the centre wavelength versus pulse width at a central wavelength of 1561 nm; and (c) optical spectrum sampled for 176 minutes**

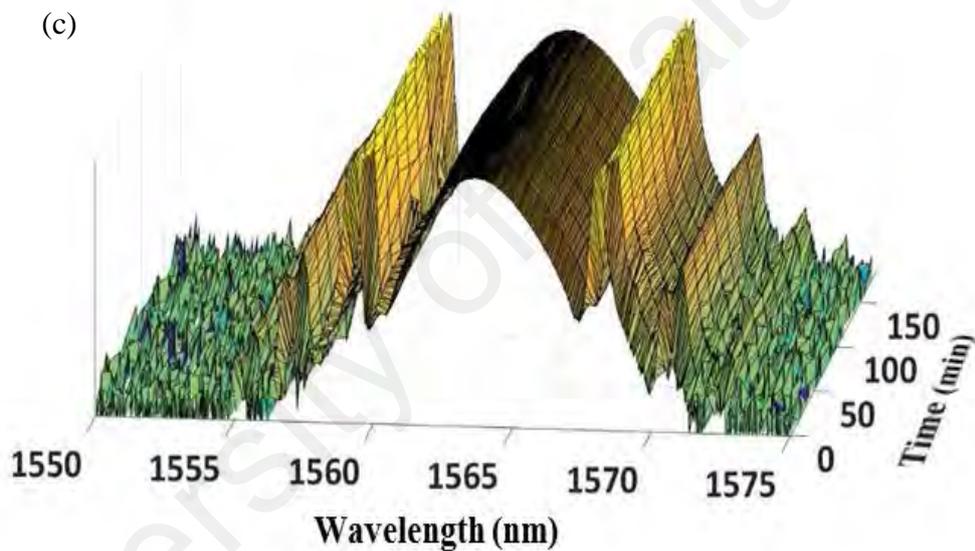
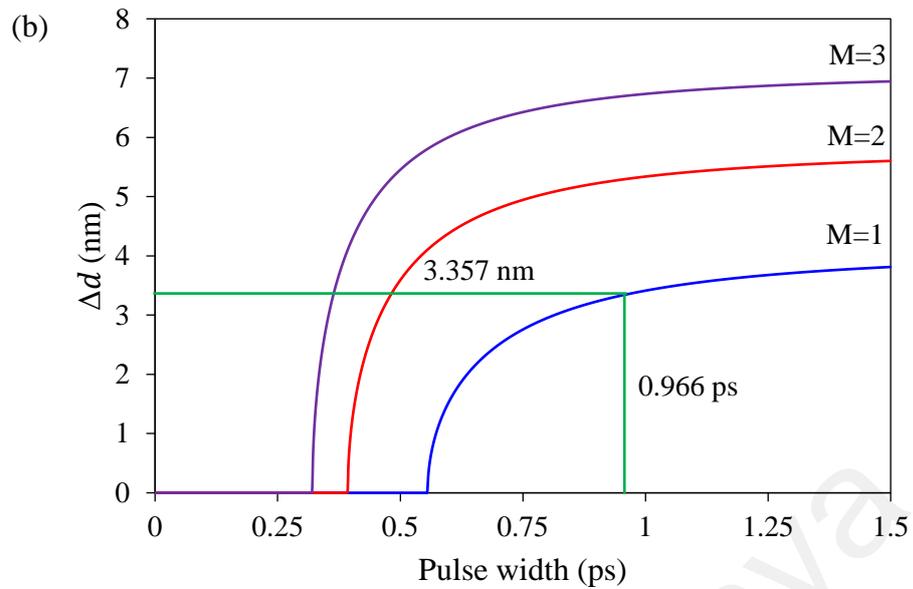
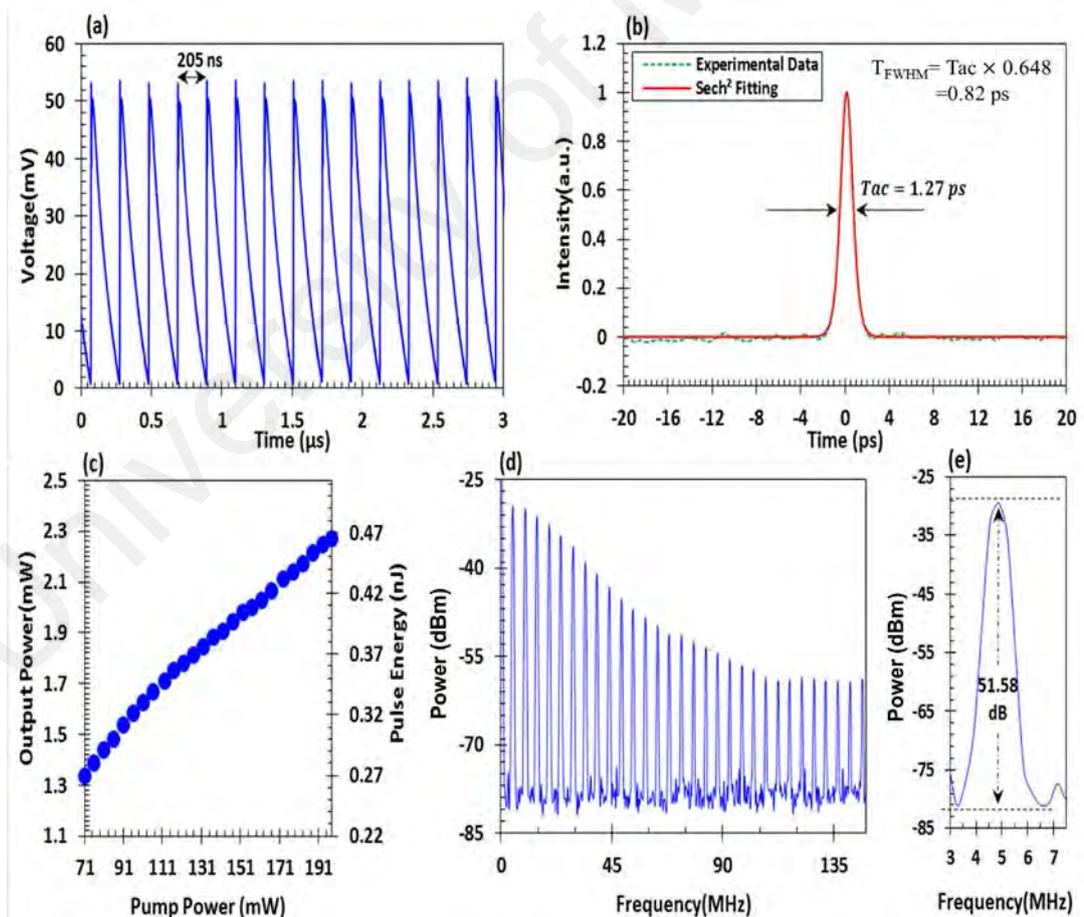


Figure 5.24, continued

#### 5.4.5 820 Femtosecond Mode-Locked Laser Operation

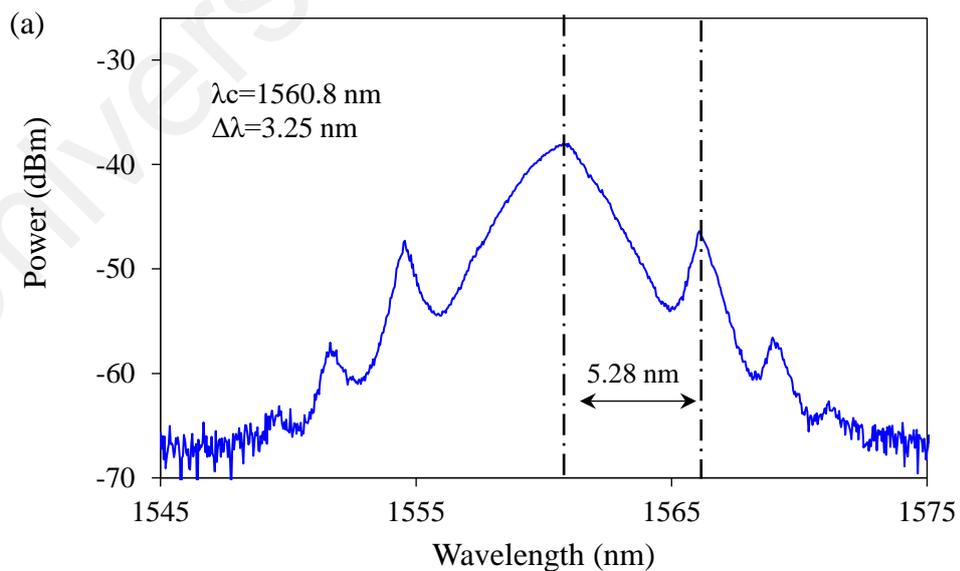
In this experiment, a shorter round trip was facilitated by further reducing the length of SMF spool from 52 m to 32.5 m, and by using 80:20 OC. The total cavity length was 42 m with an estimated dispersion of  $-0.8262$  ps<sup>2</sup>. Correspondingly, a soliton mode-locked laser with a wider spectrum, shorter pulse width and higher repetition rate was produced. The mode-locked operation was generated at a pump power range from 71 to 198 mW. Figure 5.25 (a) shows a typical pulse train with uniform pulse shape and period.

The oscilloscope recorded pulse width of 102 ns due to the limited resolution. It should be mentioned that the sawtooth-like pulse train produced by the oscilloscope is attributed to the noise produced by photo-detector. The pulse period is reduced to 205 ns, which correspond to a repetition rate of 4.9 MHz. The pulse width in this experiment has  $T_{FWHM}$  of 820 fs, as depicted in Figure 5.25 (b). The output pulse energy and average output power of this laser are presented in Figure 5.25 (c). At LD power of 198 mW, the pulse energy and average output power are 0.46 nJ and 2.27 mW, respectively. The RF spectrum within a span of 148 MHz is given in Figure 5.25 (d), the spectrum reveals a high cavity harmonic. The fundamental frequency of 4.9 MHz is presented in Figure 5.25 (e) with a span of 4.5 MHz. The figure shows a very high SNR value of 51.58 dB.

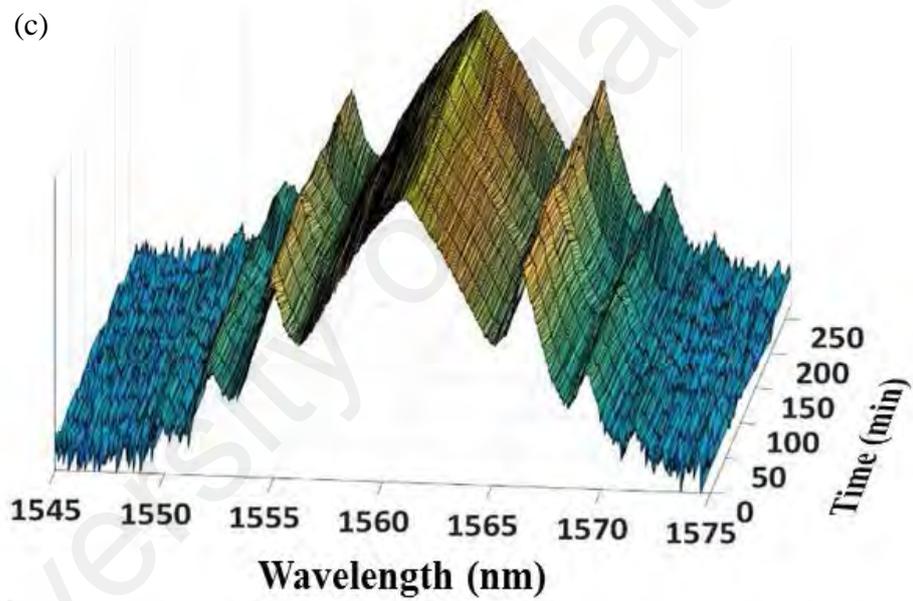
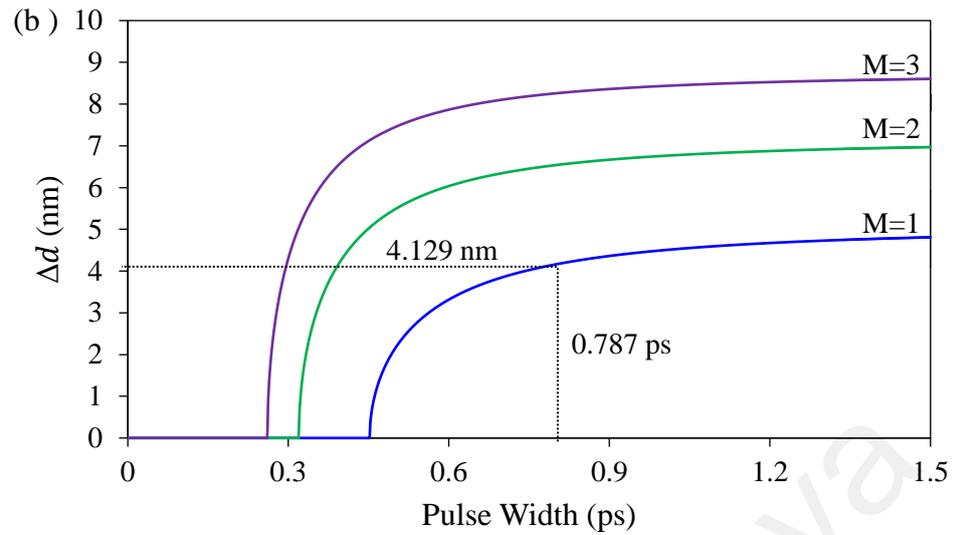


**Figure 5.25: Femtosecond pulsing laser in 42 m cavity length with 80:20 OC: (a) oscilloscope trace of the soliton laser, (b) autocorrelator trace, (c) LD power against average output power and pulse energy, (d) RF spectrum with a span of 148 MHz and (e) RF spectrum with a span of 4.5 MHz**

Figure 5.26 (a) shows the optical spectrum of the femtosecond mode-locked laser centred at 1560.8 nm, which is almost the same central wavelength as in the previous experiments. The TBP in this experiment is 0.328 which indicates that the output pulses were chirped in which the theoretical pulse duration is calculated to be 0.787 ps, this chirping also results in a difference between the theoretical and measured values of  $\Delta d$  and 3-dB bandwidth (Ausschnitt, Jain, & Heritage, 1979). The  $\Delta d$  value for the 1<sup>st</sup> order Kelly sideband position was calculated to be 4.129 nm, which differs compared to that of the measured value of 5.28 nm, see Figure 5.26 (b). A wider  $\Delta d$  is obtained in this experiment (even though the pulse duration was reduced), that is due to the increment in the cavity dispersion as compared to the previous experiments. The measured 3-dB bandwidth in this experiment is 3.25 nm, while the calculated value is 3.122 nm. The slight discrepancy is probably due to a small error in the autocorrelation measurement. The optical spectrum was sampled every ~ 30 minutes for 270 minutes, see Figure 5.26 (c).



**Figure 5.26: Femtosecond pulsing laser in 42 m cavity length with 80:20 OC: (a) output spectrum, (b) mth order Kelly sidebands distance to the centre wavelength versus pulse duration at a central wavelength of 1560.8 nm, and (c) optical spectrum sampled for 270 minutes.**



**Figure 5.26, continued**

Like the previous experiments, the mode-locked laser showed a very stable performance in which the intensity, peak locations and spectral bandwidth were very stable. It is believed that shorter pulse duration, higher repetition rate and higher pulse energy can be delivered if shorter cavity length is addressed along with a higher gain medium. This work demonstrated that  $\text{Alq}_3$  has great potentials in ultrafast fiber lasers.

## 5.5 Mode-Locked Laser at 1066 nm by Using Alq<sub>3</sub> as SA in a YDFL Cavity

As explained in chapter 3, the SA was synthesized through the drop-casting method, in which the Alq<sub>3</sub> powder was embedded into PVA. The produced SA thin-film had modulation depth, saturation intensity, linear transmission and thickness of 8.1%, 3 MW/cm<sup>2</sup>, 98% (in the range from 900–1600 nm) and 50 μm, respectively. A small piece of the thin-film was then inserted between two FC/PC to form the mode-locker device. The experimental setup of the proposed Alq<sub>3</sub> based ytterbium-doped fiber laser (YDFL) is shown in Figure 5.27. The SEM image of the SA is shown in the inset of Figure 5.27. A 980 nm LD was used to launch power to the cavity through a 980/1064 WDM coupler. A 1.5 m long single-mode ytterbium-doped fiber (YDF) (with an ion-absorption of 280 dB/m at 920 nm and cut-off wavelength of 1010 nm) was used as a gain medium. A 20-meter SMF was added to the cavity to manage the dispersion regime. The total length of the cavity was around ~28 m. A 50:50 optical coupler was used to extract 50% of the signal from the cavity, and an optical isolator (ISO) was addressed to prevent light reflection. Additionally, a polarization controller (PC) was used in the cavity. The optical spectrum, RF spectrum and optical pulse train were monitored using the OSA, RF spectrum analyzer, and digital oscilloscope, respectively.

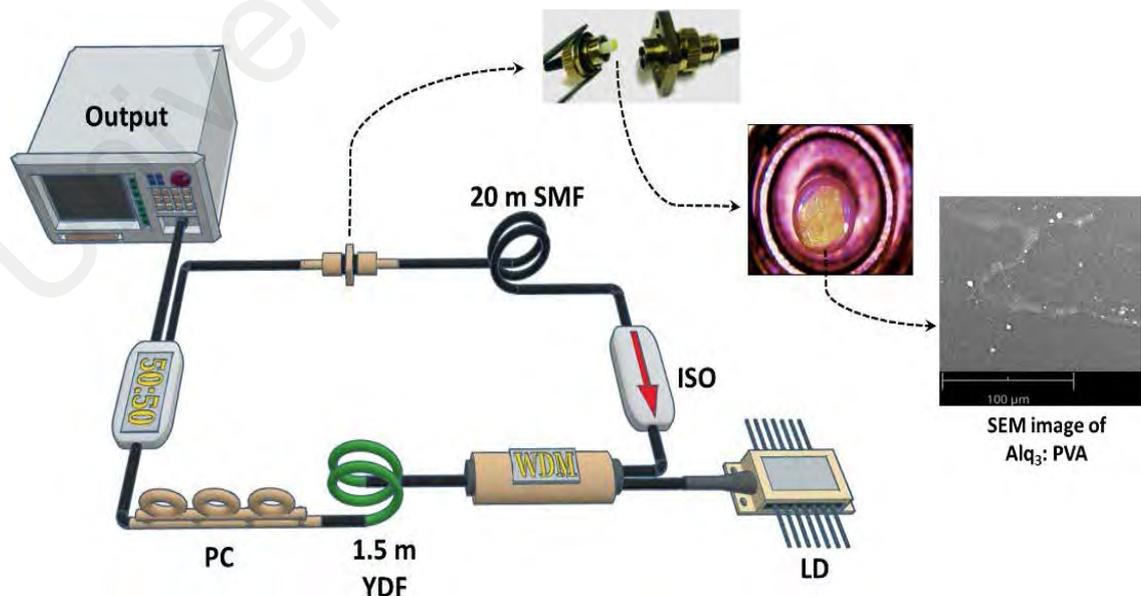
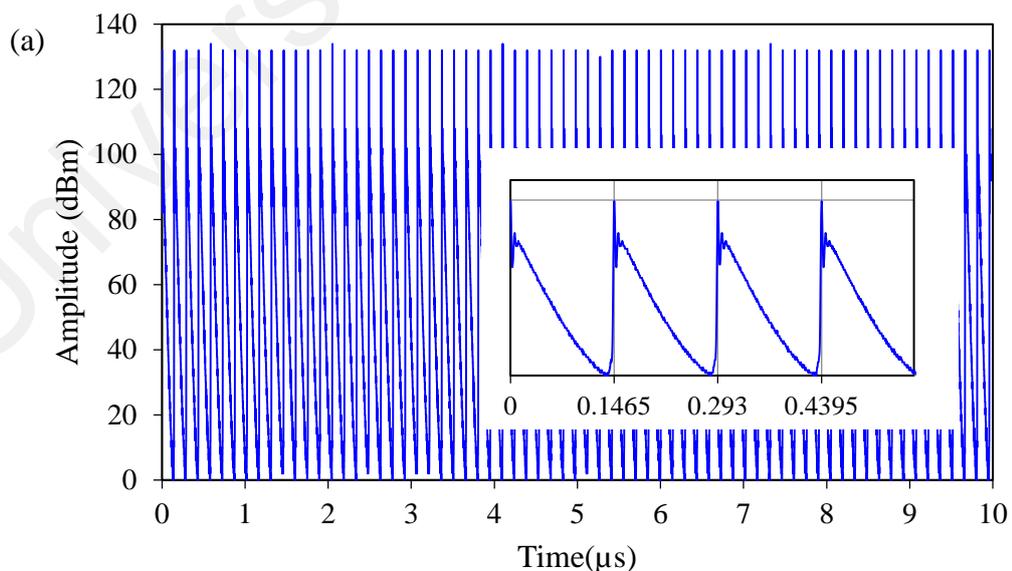
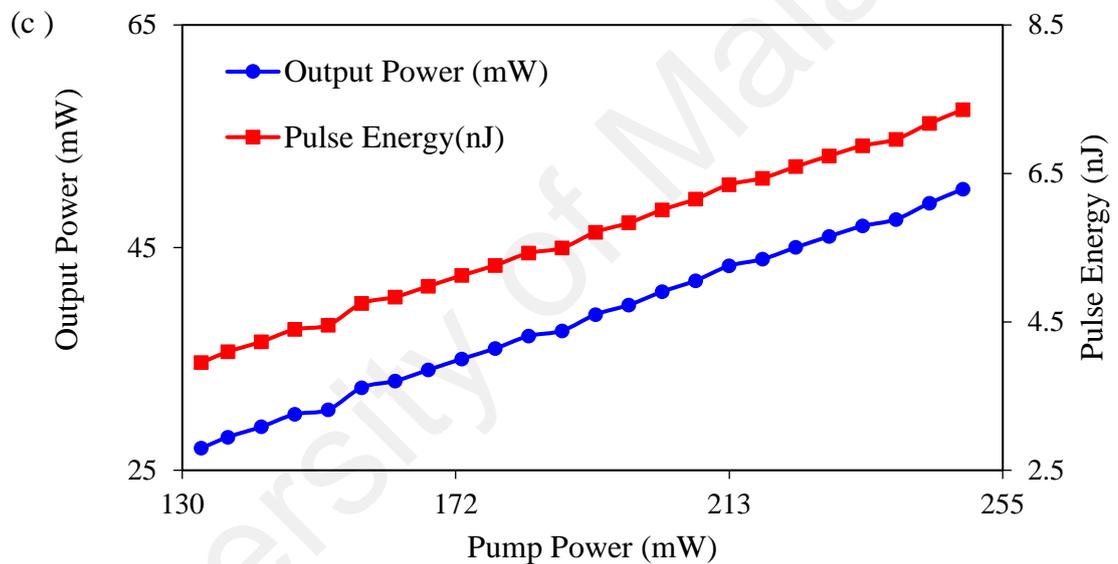
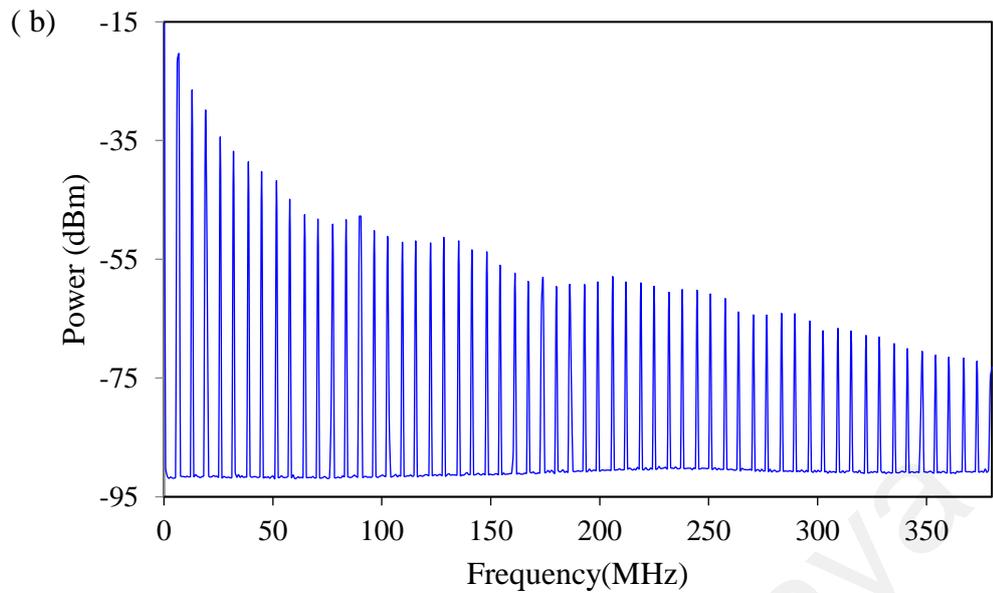


Figure 5.27 Fiber laser cavity

The mode-locked operation was easily achieved when the SA was incorporated into the cavity. At first, a continuous lasing was produced at a threshold power of 50 mW. Then by increasing the power to range between 133 mW - 249 mW, a stable and self-started mode-locked operation was observed with a pulse repetition rate, central wavelength and 3-dB bandwidth of 6.84 MHz, 1066.2 nm and 0.2 nm, respectively. Figure 5.28 (a) shows the output pulse train at maximum power, the pulse width given by the oscilloscope is  $\sim 62$  ns. Which is much wider than what we expect, this is attributed to the limited resolution of the oscilloscope. The pulse train indicates a very stable performance, in which there is no instabilities, nor distortion on the pulse train. The inset figure shows a pulse train with a shorter span of  $0.57 \mu\text{s}$ . As shown in Figure 5.28, the period is  $\sim 146.5$  ns, which is in coincident with the pulse repetition rate and cavity length. The pulse-width can be found by using the TBP formula. The calculated pulse width is 5.96 ps with TBP of 0.315 for  $\text{Sech}^2$  pulse profile. Figure 5.28 (b) presents the RF spectrum with span up to 377 MHz and a resolution bandwidth of 300 kHz. The fundamental frequency is located at 6.84 MHz with an SNR of 74 dB, which indicated excellent stability of the fiber laser. The output power and pulse energy of the fiber laser were relatively high, see Figure 5.28 (c).



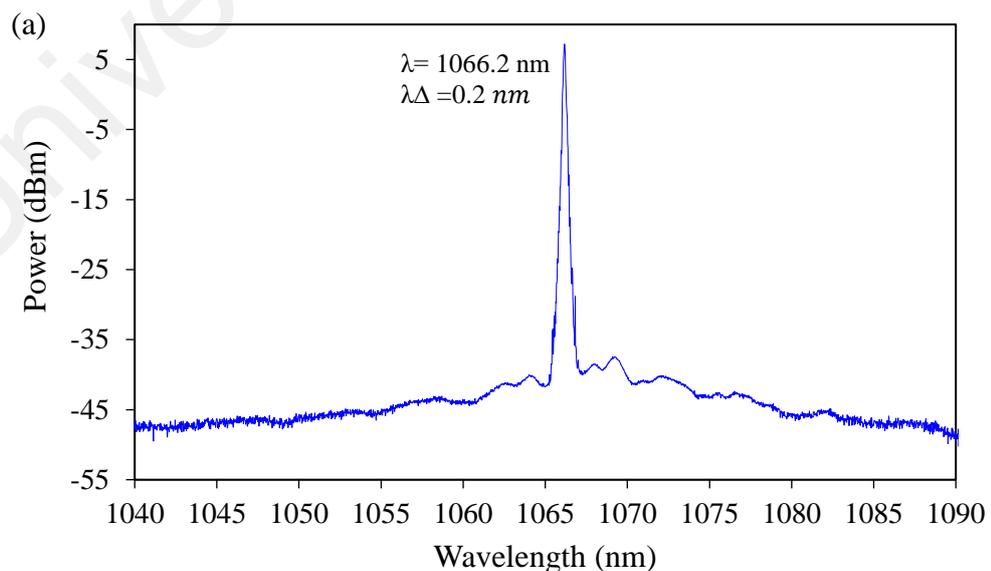
**Figure 5.28: (a) Typical pulse train and (b) RF spectrum of the laser and (c) output pulse energy and average output power versus LD power**



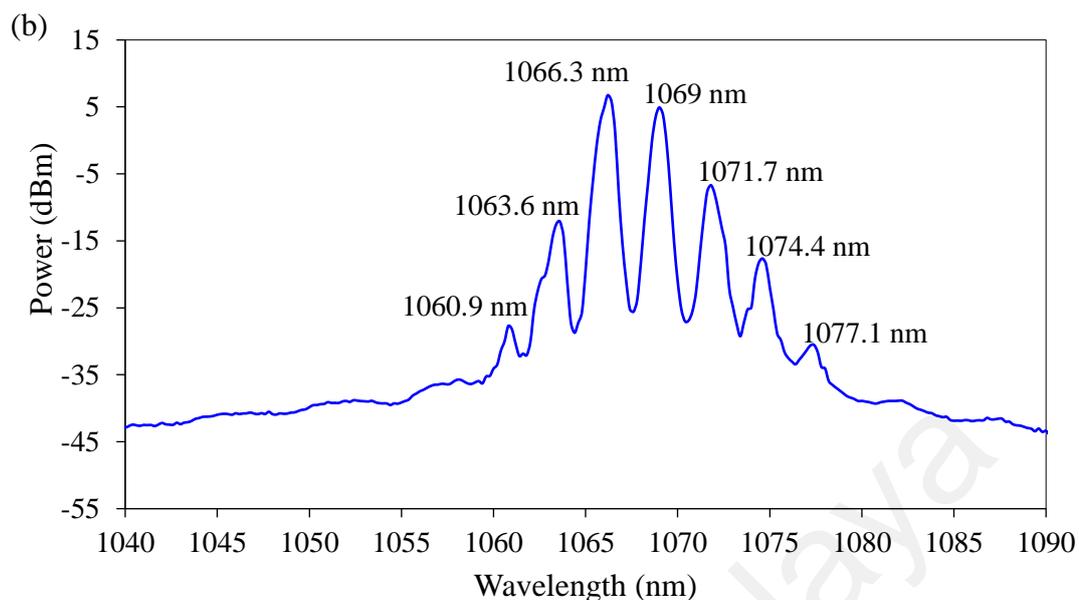
**Figure 5.28, continued**

The average output power raised linearly from 27mW to 50.2mW with a slope efficiency of 19.7 %. While the highest pulse energy was 7.35 nJ. The output spectrum of a single wavelength mode-locking operation is shown in Figure 5.29 (a). The figure shows the optical spectrum has a peak at 1066.2 nm with a 3-dB spectral bandwidth of 0.2 nm. The system produced the same performance during ~6 hours experiment, which further indicates the stability of the system. A polarization-dependent loss could be generated by squeezing and tuning the PC. Combining that with that cavity birefringence,

a spectral filter could be developed, which could be used to achieve a multi-wavelength pulsing operation (H. Zhang, Tang, Wu, & Zhao, 2009). Therefore, a multi-wavelength operation was obtained by carefully tuning the PC. Figure 5.29 (b) shows the spectrum of a multi-wavelength mode-locked laser. The peaks are located at  $\sim 1060.9$  nm, 1063.6 nm, 1066.3 nm, 1069 nm, 1071.7 nm, 1074.4 nm and 1077.1 nm with intensities of  $-27.7$  dBm,  $-12.1$  dBm,  $6.7$  dBm,  $4.9$  dBm,  $-6.7$  dBm,  $-17.64$  dBm and  $-30.5$  dBm, respectively. The peaks are  $2.7$  nm ( $\pm 0.05$  nm) apart. The operation can be sustained for a long time, given that no environment perturbations applied to the laser. The output pulse train maintained the same performance on the oscilloscope in terms of pulse width and pulse period. The motivation of this work is to draw attention to the potentials that organic materials have as mode-locker in  $1.0$   $\mu\text{m}$  region. As this is first-time, to the best of our knowledge, that  $\text{Alq}_3$  is used as mode-locker in  $1.0$   $\mu\text{m}$  region. The all-fiber based ring cavity produced a very high output power and high peak power of  $57$  mW and  $1.4$  kW, respectively. The RF spectrum showed the laser had a very high SNR of  $74$  dB, which shows an excellent performance by the laser.



**Figure 5.29: Laser output spectrum (a) narrow spectrum and (b) broadened spectrum.**



**Figure 5.29, continued**

## 5.6 Summary

Different types of mode-locked lasers were successfully demonstrated by using organic materials as the SA. In the first part, a mode-locked laser was produced by using FIrpic as SA. The repetition rate and pulse width are 3.43 MHz and 120 ns. Then Znq<sub>2</sub> was used to produce another mode-locked laser with enhanced performance. In this case, the pulse width and repetition rate were 1.46 ps and 3.5 MHz. A dark pulses mode-locking operation was also realized by appropriately managing the polarization state of the cavity. Furthermore, a supercontinuum light generation was also successfully demonstrated by launching the output pulses from the Znq<sub>2</sub> based mode-locked EDFL into a 100 m long PCF. This process resulted in reducing the pulse width to 640 fs. Ultrafast lasers were also demonstrated by using Alq<sub>3</sub> SA in four different configurations. These lasers produced pulse width/ repetition rate of 1.6 ps / 1.8 MHz, 1.26 ps / 3.36 MHz, 970 fs / 3.36 MHz and 820 fs / 4.9 MHz by using cavity length /optical coupler (OC) of 112.5 m / 80:20 OC 61.5 m / 80:20 OC, 61.5 m / 95:5 OC and 42 m / 80:20 OC, respectively. All

configurations produced a soliton laser with a very stable mode-locking operation operating at a central wavelength of ~1561 nm. The results presented in this chapter show that the developed organic materials based SAs have a very high potential to be used in fiber laser technology. In the last part of this chapter, high output power mode-locked YDFL was also demonstrated to operate at 1066.2 nm with a pulse width and repetition rate of 5.96 ps and 6.84 MHz, respectively. The SNR produced by this laser is very high (74 dB). In short, different types of mode-locked fiber lasers were successfully produced in this chapter with a simple and straightforward fabrication process as this work is intended to draw the attention of organic materials in fiber laser technology. This is the first time that FIrpc, Znq<sub>2</sub> and Alq<sub>3</sub> have been used in fiber laser technology, to the best of our knowledge. Table 5.1 presents the performance of different mode-locked lasers by using the developed SAs.

**Table 5.1: Performance of mode-locked fiber lasers by using the developed SAs**

Section in the thesis	Cavity length (m)/ coupler	3-dB (nm)	Wave-length (nm)	Pulse Width (ps)	Repetition rate (MHz)	SNR (dB)	SA
5.2	60/99:1	1.8	1562.57	120 ns	3.43	38.3	FIrpc
5.3.1	59.5/90:10	2	1560.6	1.46	3.5	60.6	Znq <sub>2</sub>
5.4.2	112.5/80:20	1.6	1561.5	1.6	1.8	41.5	Alq <sub>3</sub>
5.4.3	61.5/80:20	2.09	1561.5	1.26	3.36	51.48	
5.4.4	61.5/95:05	2.65	1561.1	0.97	3.36	43.3	
5.4.5	42/80:20	3.25	1560.8	0.82	4.9	51.58	
5.5	28/50:50	0.2	1066.2	5.96 ps	6.84	74	

## CHAPTER 6: CONCLUSION AND FUTURE WORK

### 6.1 Conclusion

There is strong motivation to find new materials to be used as a high-performance SAs, as the rapid development of SAs has provided several new opportunities for nonlinear optics. In that regard, organic materials have the advantages of large and ultrafast nonlinear response and broad spectral tunability. Moreover, they offer mechanical flexibility, light-weight and low production cost, additionally, organic materials could increase the applications in biomedical laser processing and sensing as they are (in general) non-hazardous and environmentally-friendly materials. These materials, in comparison to inorganic materials, require simple fabrication and offer versatile molecular design. Despite numerous works reported on the use of organic materials in the linear regimes and in a relatively low power domain, the applications of organic materials in nonlinear optics, especially in the fiber lasers pulse generation, have yet to be fully explored.

The goal of this research work is to draw attention to the potentials that organic materials have in fiber laser technology. Organic materials used in this work produced very interesting results for different types of pulsed fiber lasers. This work involved the fabrication and characterization of newly developed passive SAs based on organic materials, these materials are FIrpic, Znq<sub>2</sub> and Alq<sub>3</sub>. In chapter 3, the fabrication and characterization of these materials have been thoroughly described. The fabrication process was straight forward, simple and low-cost where PVA was used to form thin-films. PVA is a suitable host to produce high-performance SA due to its favourable physical properties such as biocompatibility and good chemical resistance. Additionally, PVA has excellent film-forming, mechanical and adhesive properties. Then these SAs thin-films were characterized in terms of FTIR, SEM image, linear and non-linear absorptions. The FIrpic, Znq<sub>2</sub> and Alq<sub>3</sub> have a saturation intensity of 5.5 MW/cm<sup>2</sup>, 103.6 MW/cm<sup>2</sup>, 3 MW/cm<sup>2</sup> and modulation

depth of 12.8%, 17.6% and 8.1% and linear absorption of ~ 2 dB, 1.2 dB and 2 dB at the region of 1550 nm, respectively.

Different types of passive Q-switched fiber laser were successfully developed at 1.5  $\mu\text{m}$  region. FIrpic based SA was used to generate Q-switched pulsing operation with a single and dual-wavelength operation. A stable Q-switched laser operating at 1560.4 nm was achieved at a threshold pump power of 30 mW. The pulse repetition rate increased from 39.22 to 87.4 kHz and the pulse width decreased from 9.5 to 3.4  $\mu\text{s}$  as the pump power was gradually increased from 30 to 208 mW.  $\text{Znq}_2$  were used to demonstrate Q-switched pulsing operation covering almost all the C-band region. The produced pulse width was decreased from 6.6  $\mu\text{s}$  to 2.8  $\mu\text{s}$  as the LD power increased, while the repetition rate was increased from 45 kHz to 86 kHz as the pump power was increased to 198 from 38 mW. Then a tunable bandpass filter was used to induce a tunability on the output wavelength. Correspondingly, the wavelength was shifted continuously and covered almost all the C-band region from ~1520 nm to ~1563 nm. Furthermore,  $\text{Alq}_3$  was used to generate a Q-switched laser operating at 1559 nm. The generated pulse train was stable and has a pulse width decreased from 6.65 to 1.2  $\mu\text{s}$  and the pulse repetition rate increased from 31.65 to 144.5 kHz as the pump power increased from 20 to 122 mW. The potential of organic materials for generating Q-switching at 1067 nm was also confirmed.  $\text{Alq}_3$  was used in an ytterbium-doped fiber laser cavity and produced high pulse energy of 0.8  $\mu\text{J}$  and high peak power of 90 mW. When the laser cavity was reduced from 25 m to 5 m, the peak power increased and the pulse energy dropped to 237.62 mW and 451.5 nJ, respectively. Additionally, the minimum pulse width was reduced from 9  $\mu\text{s}$  to 1.9  $\mu\text{s}$ .

Chapter 5 was set to address the potentials of the proposed organic materials in generating ultrafast mode-locked fiber lasers. FIrpic based SA has successfully produced a soliton mode-locking pulses at a wavelength of 1562.57 nm. The mode-locked laser had a repetition rate of 3.43 MHz and a pulse width of 120 ns within the pump power of 35–

188 mW. On the other hand,  $\text{Znq}_2$  was used as a mode-locker to generate a dark and white mode-locking operation. The pulse width and the repetition rate in the white mode-locked laser were 1.2 ps and 3.5 MHz, respectively. Furthermore,  $\text{Znq}_2$  were used to generate a supercontinuum light by incorporating 100 m long PCF at the output of the cavity. Finally, several mode-locking operations were demonstrated by using  $\text{Alq}_3$  as SA. By incorporating  $\text{Alq}_3$  into the fiber laser cavity, a stable pulsing was achieved. The laser pulses have a pulse width of 1.6 ps with a soliton spectrum centred at 1561.5 nm. The pulses train exhibits a very stable output with a high signal to noise ratio (SNR) of 41.5 dB in 112.5 m cavity with 80:20 optical coupler. The pulse width / repetition rate changed to 1.26 ps/3.36 MHz, 970 fs/3.36 MHz and 820 fs/4.9 MHz by changing the cavity length /OC to 61.5 m/80:20 OC, 61.5 m /95:5 OC and 42 m/ 80:20 OC, respectively. The  $\text{Alq}_3$  was used to develop ultrafast fiber laser at 1066.2 nm with a pulse width and repetition rate of 5.96 ps and 6.84 MHz, respectively. The SNR produced by this laser is significantly high of 74 dB which proves the stability of the developed fiber laser.

## 6.2 Future Work

All the proposed objectives have been successfully implemented. However, the demonstration of the organic materials in pulsed fiber laser technology can be further explored. Further work should be devoted to enhancing the performance of the proposed fiber laser in terms of shorting the pulse width, increasing the repetition rate and the output power and pulse energy. This can be achieved by addressing a shorter cavity length and enhanced gain medium. Additionally, future work should focus on exploring the developed SAs in other wavelength regions such as 2  $\mu\text{m}$  and 3.5  $\mu\text{m}$  using a different gain medium, this will be a very interesting area to investigate. As to the best of our knowledge, there is no research addressing organic materials-based SA in these wavelength regions. On the other hand, developing a new set of organic material for fiber

laser application will be very interesting research work. As through this thesis, we found that these materials have very attractive performance in fiber laser technology.

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## LIST OF PUBLICATIONS

List of publications during the study:

1. **Salam, S.**, Al-Masoodi, A., Al-Hiti, A. S., Al-Masoodi, A. H., Wang, P., Wong, W. R., & Harun, S. W. (2019). *Flrpic thin film as saturable absorber for passively Q-switched and mode-locked erbium-doped fiber laser*. *Optical Fiber Technology*, 50, 256-262.
2. **Salam, S.**, Al-Masoodi, A., Wang, P., & Harun, S. (2020). *Hybrid organic small molecules as a saturable absorber for passive Q-switching in erbium-doped fiber laser*. *OSA Continuum*, 3(2), 177-185.
3. **Salam, S.**, Al-Masoodi, A. H., Wang, P., & Harun, S. W. (2020). *Femtosecond mode-locked erbium-doped fibre laser with Alq<sub>3</sub> saturable absorber*. *IET Optoelectronics*.
4. **Salam, S.**, Al-Masoodi, A. H. H., Yasin, M., & Harun, S. W. (2020). *Soliton mode-locked Er-doped fiber laser by using Alq<sub>3</sub> saturable absorber*. *Optics & Laser Technology*, 123, 105893.
5. **Salam, S.**, Azooz, S. M., Nizamani, B., Najm, M. M., & Harun, S. W. (2020). *Mode-locked laser at 1066 nm by using Alq<sub>3</sub> as saturable absorber in all-fiber based cavity*. *Optik*, 165179.
6. **Salam, S.**, Harun, S. W., Al-Masoodi, A. H., Ahmed, M. H., Al-Masoodi, A. H., Alani, I. A., . . . Yasin, M. (2019). *Tris-(8-hydroxyquinoline) aluminium thin film as saturable absorber for passively Q-switched erbium-doped fibre laser*. *IET Optoelectronics*, 13(5), 247-253.
7. **Salam, S.**, Sulaiman, S., Al-Masoodi, A., Al-Azzawi, A. A., & Harun, S. (2019). *Q-switched ytterbium-doped fiber laser by using Flrpic as a saturable absorber*. *OSA Continuum*, 2(7), 2145-2152.
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9. Nizamani, B., Jafry, A., **Salam, S.**, Najm, M. M., Khudus, M. A., Hanafi, E., & Harun, S. (2020). *Mechanical exfoliation of indium tin oxide as saturable absorber for Q-switched Ytterbium-doped and Erbium-doped fiber lasers*. *Optics Communications*, 126217.

10. Nizamani, B., Memon, F., Umar, Z., **Salam, S.**, Najm, M., Khudus, M. A., . . . Harun, S. (2020). *Q-switched erbium-doped fiber laser with silicon oxycarbide saturable absorber*. *Optik*, 165234.
11. Nizamani, B., **Salam, S.**, Jafry, A., Zahir, N., Jurami, N., Khudus, M. A., . . . Harun, S. (2020). *Indium Tin Oxide Coated D-Shape Fiber as a Saturable Absorber for Generating a Dark Pulse Mode-Locked Laser*. *Chinese Physics Letters*, 37(5), 054202.

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