

Graphene-polyvinyl alcohol polymer based saturable absorption at 2000 nm region

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ABSTRACT

A graphene-polyvinyl alcohol (PVA) composite saturable absorption is demonstrated at 2000 nm region. Graphene suspension is produced using low-cost electrochemical exfoliation process. The suspension is mixed with PVA host polymer in 1:1 ratio and left evaporated at room temperature which finally produced graphene-PVA thin film. Thulium doped fiber (TDF) gain medium has been shown to produce a stable Q-switched pulse with a highest repetition rate of 54 kHz, a short pulse duration of 2.89 μ s, a maximum peak power of 16 mW, and an estimated maximum pulse energy of 49 nJ. Apparently, at 2000 nm region, superior performances of graphene-PVA composite have been recorded which was largely contributed by meticulous composite preparation and homogenous mixture with PVA host.

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1. INTRODUCTION

Pulse laser generation employing carbon nanotubes and graphene based passive saturable absorber have been extensively investigated and reported due to their intrinsic properties [1]. Recently, topological insulator (TI), molybdenum disulfide (MoS_2) and black phosphorus (BP) have been demonstrated as passive saturable absorber either for Q-switching or mode-locking [1]. The main advantage of producing graphene based passive saturable absorber is matured process of dispersion and fabrication compared to dispersion and exfoliation of TI, MoS_2 and BP [1].

To date, several approaches have been adopted in graphene synthesis for saturable absorber applications. These include chemical vapour deposition, liquid phase exfoliation (LPE), micro-mechanical cleavage and reduced graphene oxide (rGO) [2]. However, these methods do have some drawbacks which include high processing temperature, time consuming and high cost [3], [4]. In 2008, Liu *et al.* [5] had demonstrated a process of graphene synthesis using electrochemical exfoliation. In their work, the process requires simple apparatus which include graphite electrodes, electrolyte and DC power supply. The same method of processing has been recorded in a work by Parvez *et al.* [6], Raj *et al.* [7], Bhullar and Liu [8] and Ljubek *et al.* [9] which indicate that this method of graphene synthesis are widely accepted due to its practicality and low-cost. In saturable absorption, few works have been based on graphene synthesis using

electrochemical exfoliation. In these works, graphene in a form of membrane or polymer composite is integrated in the laser cavity which is used to generate laser pulses. Work by Yang *et al.* [10] had produced a mode-locker at 1500 nm region by graphene flakes produced using electrochemical exfoliation of highly oriented pyrolytic graphite (HOPG) (anode) and a platinum (Pt) wire (cathode). Similarly, Lin *et al.* [11] had generated a mode-locker at 1500 nm region by graphene flakes syphoned into the photonic crystal fiber. By using graphene-polyethylene oxide (PEO) film, Haris *et al.* [12] managed to produce a mode-locker at 1.500 nm region. Meanwhile, in 1900 nm region, Saidin *et al.* [13], [14] had adopted an electrochemical exfoliation technique in graphene-PEO thin film fabrication which produced Q-switching pulse laser. Other efforts by Ahmad *et al.* [15], Yamashita [16] and Jiang *et al.* [17] had produced a Q-switcher by applying other methods which include graphene oxide particles dissolution and chemical vapor deposition. In light of graphene based material with polyvinyl alcohol (PVA) composite as a host, few works have been recorded which include our previous work on graphene nanoplatelets (GnP) at 1500 nm in [18], Apandi *et al.* [19] which also concentrated on GnP-PVA at 1500 nm, work on graphene oxide-PVA by Rosol *et al.* [20] at 1900 nm and graphene nanosheet-PVA by Ahmad *et al.* [21] at 1500 nm region. Based on these literatures, we strongly believe that feasibility of graphene-PVA composite saturable absorber at 2000 nm can still be researched with lower-cost fabrication approach of electrochemical exfoliation.

In this work, a graphene-PVA based saturable absorption for thulium doped fiber lasers (TDFL) has been demonstrated at 2000 nm region which yield Q-switched pulses. An electrochemical exfoliation technique is adopted in graphene suspension production. For stable binding, a mixing process with PVA which act as a host polymer will be optimized. Meticulous approaches in saturable absorber fabrication are the key contribution to be presented in this paper, which eventually improved the performance of Q-switched pulse laser.

2. MATERIALS PREPARATION

In this process of saturable absorber synthesis, few important steps have been established and optimized for good characterization performance of saturable absorber. Initially, graphene flakes in a form of suspension will be synthesized using an electrochemical exfoliation process. Based on our observation, 20 volts is the optimized potential that will speed-up the process of dedocyl sulphate ions accumulation at the positive terminal. Note that the graphite rods (positive and negative terminals) are placed in a 1% sodium dedocyl sulphate (SDS) electrolyte. The process setup is shown in Figure 1. It is expected that this accumulation will result in the freeing of graphene molecules from their bonds with graphite [22], [23].

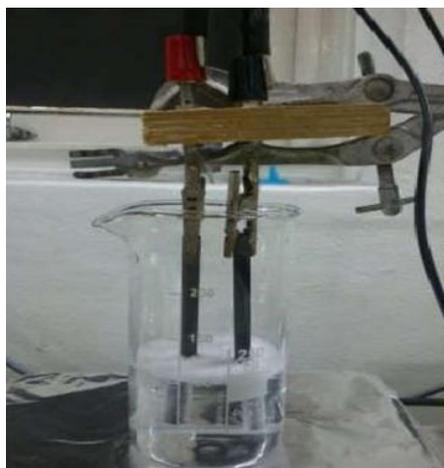


Figure 1. Electrochemical exfoliation of graphene

A complete process will take up to 2 hours which will produce a stable graphene suspension in electrolyte. A second step is centrifugation in which the obtained suspension will be centrifuged at 3,000 rpm for agglomerates removal. It will take up to 30 minutes prior to removal of supernatant section. In the third step, the graphene suspension will be diluted 1:1 with the previously prepared PVA solution. Finally, evaporation at room temperature produces free-standing films from the solution as shown in Figure 2. Gr PVA-based SA thin films are shown in Figures 2(a) and (b) before and after drying.

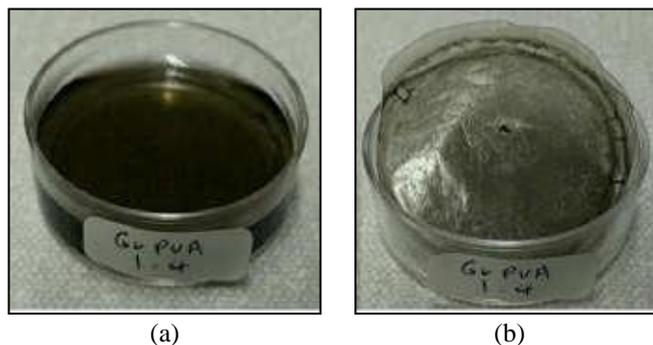


Figure 2. Graphene based SA thin film: (a) graphene solution and (b) free standing film

3. RESULTS AND DISCUSSION

For saturable absorber characterization, a thulium doped ring fiber laser setup has been developed. The experimental configuration is depicted in Figure 3, which shows the embedment of the graphene-PVA layer in the cavity. Gain medium is a 5 m long TDF and laser diode with 1,552 nm pumped laser diode employed in this experiment.

For monitoring, 10% of output light was extracted using 90/10 optical coupler. The InGaAs photodetector and optical power meter were used for spectrum monitoring while RF measurement used to measure the pulse repetition rate and pulse width. Further calculation of pulse energy can be carried out from these data. The graphene-PVA is placed after the 90% output coupler path and connected back to wavelength-division-multiplexing (WDM) to complete the ring cavity laser configuration. We have prepared a 4 mm² square graphene-PVA film and sandwiched it in between two FC/PC fiber connectors.

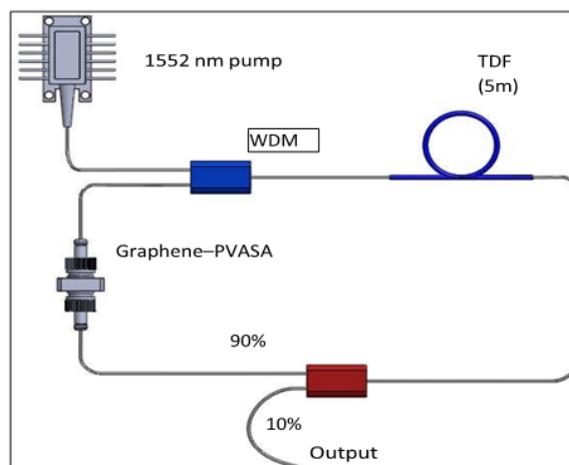


Figure 3. Cavity ring measurement setup

Without the saturable absorber, it is noted that 300 mW pump power will generate a stable continuous wave (CW) laser. Based on our observation, the between 400 mW and 650 mW of pump power, a Q-switched pulse train will be generated. It is worth to note that without saturable absorber at 650 mW pump power, the laser cavity produces continuous wave with central wavelength at 1934.2 nm. As illustrated in Figure 4, when saturable absorber was added to the cavity, it shifted the central wavelength to 1905 nm. A spectral broadening is recorded due to the loss introduced by the absorber and pulses are observed through oscilloscope.

Beyond 650 mW, the absorber could not withstand further pumping power as indicated by zero pulse. At 650 mW pump power, the laser cavity produces peak power of 16.21 mW. Figure 5 shows the repetition rate produced in the laser cavity with respective pump power. As demonstrated in Figures 5(a)-(d) the repetition rate increased from 25.7 kHz to 54 kHz.

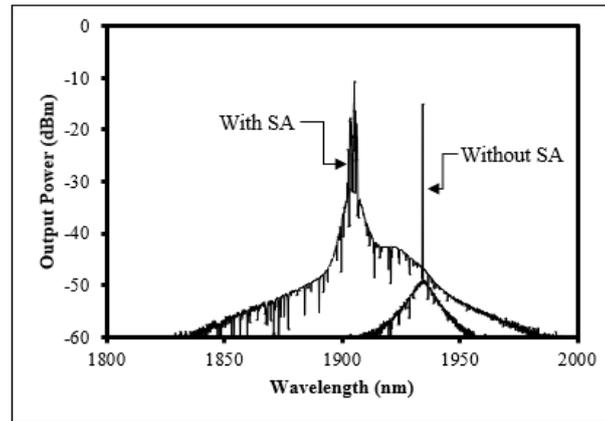


Figure 4. Optical spectrum of with and without saturable absorber at 650 mW pump power

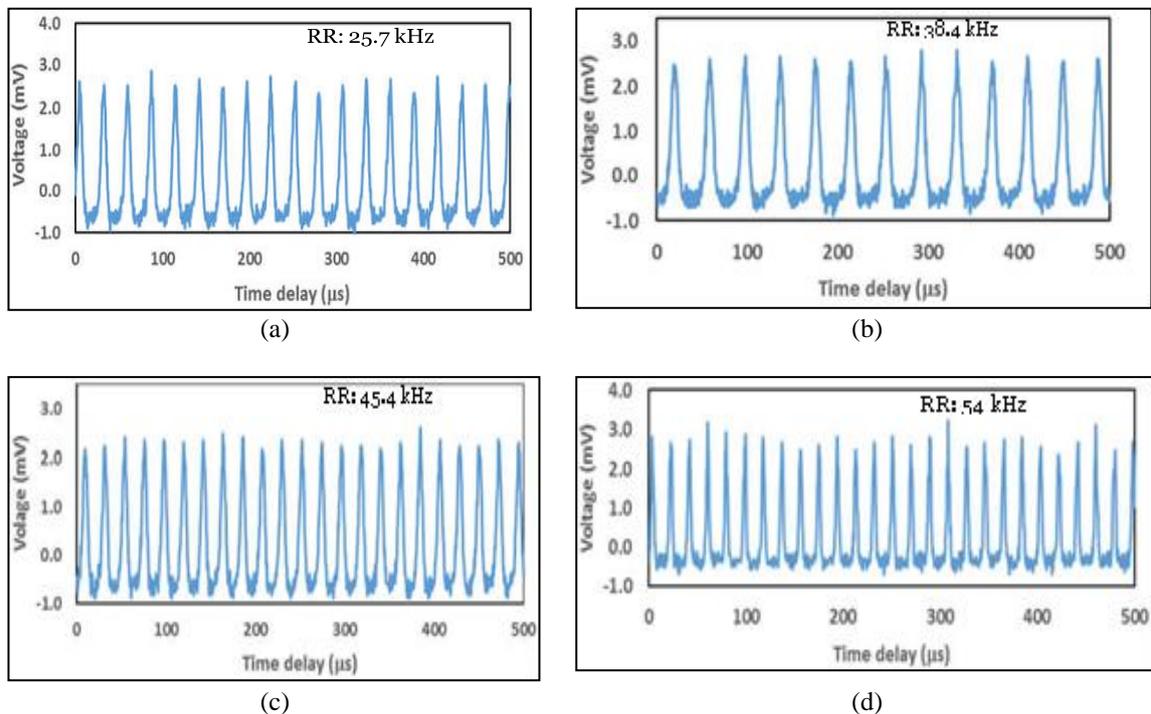


Figure 5. Pulse train at pump power: (a) 400 mW, (b) 450 mW, (c) 500 mW, and (d) 650 mW

As seen in the Figure 6, the pulse envelope of a single pulse and the frequency of its repetition are $2.89 \mu\text{s}$ and 54 kHz . The plot is based on a maximum pump power of 650 mW being applied. Repetition rate and pulse width variation with pump power are depicted graphically in Figure 7. As shown, repetition is measured to be between 25.7 kHz (threshold 400 mW) and 54 kHz (maximum 650 mW). Undoubtedly, this agrees with Q-switching characteristics for pulse repetition rate and pump power variation [24]-[26]. However, pump power appears to have an inverse relationship to pulse width. Figure 8 depicts the relationship between pump power, average output power, and pulse energy. When the pump's output is varied from $400\text{-}650 \text{ mW}$ as illustrated, the peak power climbs from 0.1 mW to 16 mW and the pulse energy from 1.2 nJ to 49 nJ .

The performance of our fabricated graphene-PVA SA in $2,000 \text{ nm}$ region is better than the result reported by Saidin *et al.* [13], [14] and Ahmad *et al.* [15]. We strongly believed that this maybe contributed by our meticulous preparation of the graphene-PVA film using electrochemical exfoliation technique. Ahmad *et al.* [15] employed optical deposition approach of graphene oxide particles and they reported a Q-switched pulse with maximum repetition rate of 16 kHz , shortest pulse width of $9.8 \mu\text{s}$ and maximum pulse

energy of 18.8 nJ. Saidin *et al.* [13], [14], demonstrated a graphene-PEO based passive saturable absorber with repetition rate of 7.5 kHz, 19.7 μs pulse width and maximum pulse energy of 66.6 nJ. In this work, we produce higher range of repetition rate, shortest pulse width compared to the work proposed by Saidin *et al.* [13], [14] and Ahmad *et al.* [15], and higher average output power than Ahmad *et al.* [14]. In comparison with work by Rosol *et al.* [20], our work is superior for all measured parameters of repetition rate, shorter pulse width and calculated pulse energy.

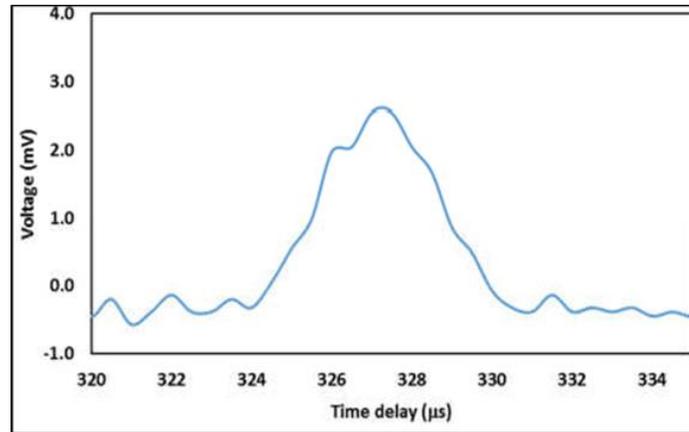


Figure 6. Single pulse envelope at maximum repetition rate 54 kHz with pulse width of 2.89 μs

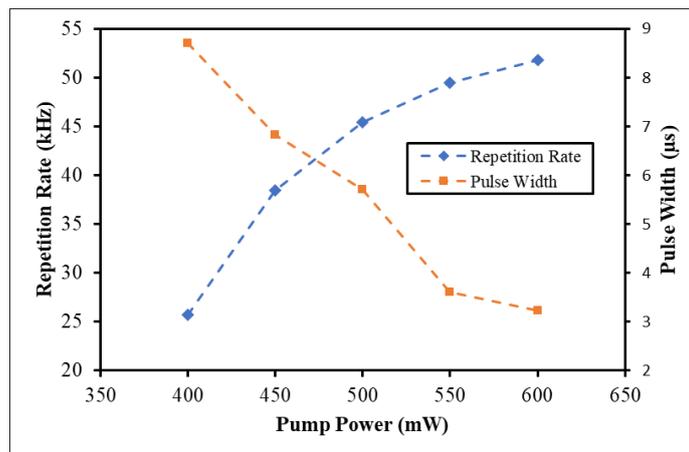


Figure 7. Pulse width and repetition rate versus pump power

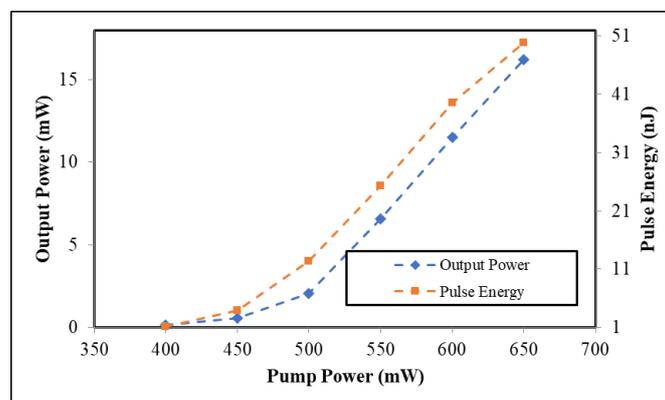


Figure 8. Pulse energy and peak power versus pump power

4. CONCLUSION

In this paper, a graphene-PVA composite based saturable absorption has been demonstrated at 2000 nm region. An electrochemical exfoliation technique in room temperature is applied for graphene flakes synthesis. The suspension is homogeneously mixed with polyvinyl alcohol (PVA) polymer as a host material and naturally dried to finally formed a graphene-PVA film. Using 5 meters TDF gain medium in approximately 14 meters cavity length, a Q-switched pulse train has been generated with maximum repetition rate of 54 kHz, with respect to maximum allowable pump power of 650 mW. From our measurement, the laser cavity produced maximum peak power of 16 mW, maximum pulse energy of 49 nJ and shortest pulse width of 2.89 μ s. Comparison with previous published work has shown superior performances of our fabricated SA in terms of repetition rate, pulse width and average output power. We believe that a meticulous preparation of the graphene-PVA saturable absorber using the electrochemical exfoliation and homogeneous mixture with PVA host is the key contribution to this success.

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