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Comparison of Polyethylene Oxide and Polyvinyl Alcohol as a Host for Graphene Saturable Absorber at 1.5 μm Region

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Abstract: This paper compares the performance of two polymeric substance, polyvinyl alcohol (PVA) and polyethylene oxide (PEO) as a host in graphene saturable absorber (SA) fabrication. By applying a low-cost solution casting method, graphene flakes which was prepared using the electro-chemical exfoliation technique will be mixed with PEO or PVA solvent as a host. Single concentration (12.04 wt.%) of graphene-PVA and graphene-PEO has been prepared for comparison. At 1.5 μm wavelength region, 2.8 meters erbium doped fiber (EDF) was applied as a gain medium in ring laser cavity setup. Generally, our measurement confirmed stable Q-switched pulses generation (>30 dB) for both SAs at 1.5 μm region. Evidently, the generated pulse by graphene-PVA at 1531.1 nm produced shorter pulse width of 1.85 μs as compared to 18.1 μs by graphene-PEO at maximum permissible pump power. Although smaller pulse energy was calculated for graphene-PVA, the ability of this type of SA to withstand higher pump-power proved its superiority as compared to its counterpart. Notably, this is the first ever effort that assess the performance of PVA and PEO host in graphene-based saturable absorption, particularly at 1.5 μm region.

Keywords: Graphene, Polyvinyl alcohol, Polyethylene oxide, Saturable absorber

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1. INTRODUCTION

Graphene has attracted wide interest of research communities due to its ideal optical characteristics. Features such as rapid recovery time and ultra-wideband absorption has led to its application as saturable absorber (SA) in pulse fiber laser generation [1-3]. It has been stated by Bonaccorso, et al. [4] that the compatibility of material to act as passive SA is hugely depending on their intrinsic properties which include ultrafast carrier relaxation, high nonlinearities and broadband operating wavelength.

In graphene-based SA fabrication, the use of binding material or host material is extremely important. Certainly, large area thin film with great optical homogeneity can be produced with suitable host material. In addition, good binding material will assist in fine controllability of saturable absorption. In view of binding material used in SA fabrication, few polymeric materials have been applied which include polyvinyl alcohol (PVA) [5-9] and polyethylene oxide (PEO) [10-12]. Note that, graphene derivatives with PVA-binded SA have been demonstrated in our previous work, particularly at 1.5 μm [13-14] and 2.0 μm [15]. Based on our thorough review, we believe that although the use of PVA and PEO have been well-established in graphene-based SA application, comparison between the two substances in terms of the fabricated SA performance is yet to be reported.

In this paper, we will report on the optimized solution casting method for SA fabrication and characterization using graphene in which two host materials, PVA and PEO will be applied. Based on our recent characterization [14], a single concentration (12.04 wt.%) of graphene-PVA and graphene-PEO will be prepared and tested for fabricated SA performance. We believe that the outcome of these findings will assist future related works in SA application.

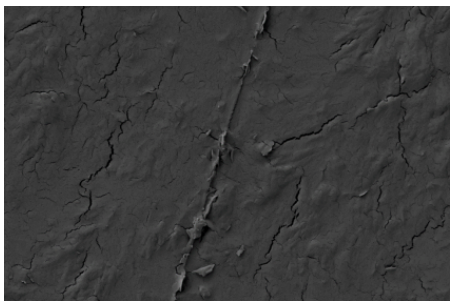
2. MATERIALS PREPARATION

Both PVA and PEO which were initially in a powder form need to be dissolved prior to its mixture with graphene flakes. For this purpose, a hot plate stirrer was applied to dissolve a pre-calculated weight of PEO or PVA in 120 ml of DI water. This process will take roughly three hours for complete dissolution. The PEO/PVA suspension will be used as a host in the mixing process. Graphene flakes will be prepared using electro-chemical exfoliation process, which has been demonstrated in our previous work [15]. Due to its low-cost and practicality, the same method of preparation has also been adopted in a work by Ljubek, et al. [16], Raj, et al. [17], Bhullar and Liu [18] and Parvez, et al. [19]. Based on our experiment, a potential difference

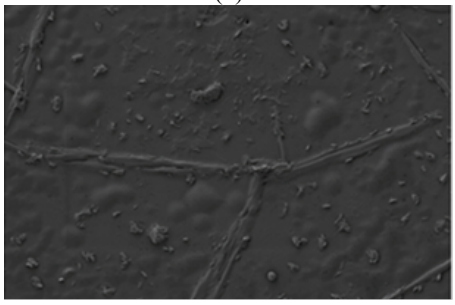
of 20 V was applied between 1 cm separation of graphite rods. The electrolyte used in this process was 1% sodium dedocyl sulphate (SDS) in deionized water. Due to this applied potential, anions and cations will be attracted to their respective positive and negative electrodes. Apparently, the anions of dedocyl sulphate will be accumulated at the positive terminal. This process of electro-chemical exfoliation will take almost two hours to finally produce a stable suspension.

In the centrifugation process, the optimized speed of 3000 rpm was applied for 30 minutes. The clear liquid portion of the suspension will be filtered out. The PVA/PEO solution which was prepared earlier will be mixed with the graphene solution at pre-determined ratio to produce single concentration (12.04 wt.%) of graphene-PVA and graphene-PEO. To produce stable composition of mixed solution, an ultra-sonic bath was applied for one hour. Finally, the solution was dried at room temperature to obtain free standing films.

For structural study, a Field-Emission Scanning Electron Microscope (FESEM) (Zeiss Crossbeam 340) was used. The sample shown in Figure 1(a) and Figure 1(b) depict homogeneous incorporation of graphene in the PEO and PVA polymer, respectively. Alternatively, we have applied a Raman spectroscopy technique to confirm the structural properties of composited graphene-PEO and graphene-PVA. A laser excitation at 532 nm was used to get the Raman spectra. Figs. 2(a) and 2(b) illustrate the Raman spectrum for graphene-PVA and graphene-PEO, respectively. The intensity peaks at Raman shifts of roughly 1355 cm^{-1} (D peak), 1590 cm^{-1} (G peak), and 2705 cm^{-1} (2D peak), are noted from the figures. As observed by Chen, et al. [20], G to 2D peak ratio of greater than 0.5 indicates the presence of multilayer graphene. Hence, our Raman's result for graphene-PEO and graphene-PVA with G to 2D intensity ratio of bigger than 1, confirms the multilayer structure of the fabricated film.

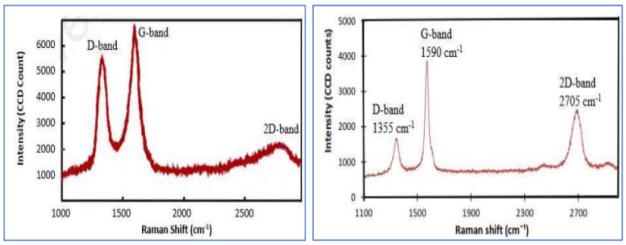


(a)



(b)

Figure 1. FESEM image of composited (a) graphene-PEO (b) graphene-PVA



(a) (b)

Figure 2. Raman spectrum of (a) graphene-PEO (b) graphene-PVA

3. RESULTS AND DISCUSSION

For saturable absorber characterization, a laser cavity measurement has been setup in which the fabricated graphene film was integrated in the cavity. The schematic of this setup is shown in Figure 3 with 2.8 meters of Erbium Doped Fiber (EDF) as the gain medium.

For monitoring, 10% of output light was extracted using 90/10 optical coupler. The InGaAs photodetector coupled with oscilloscope (OSC) used to measure the pulse repetition rate (RR) and pulse width while radio frequency spectrum analyser (RFSA) measurement used to measure the signal to noise ratio. Further calculation of pulse energy can be carried out from these data. The fabricated graphene-PVA /graphene-PEO are placed after the 90% output coupler path and connected back to wavelength division multiplexer (WDM) to complete the ring cavity laser configuration. We have prepared a 4 mm^2 square graphene-PVA/graphene-PEO film and sandwiched it in between two FC/PC fiber connectors.

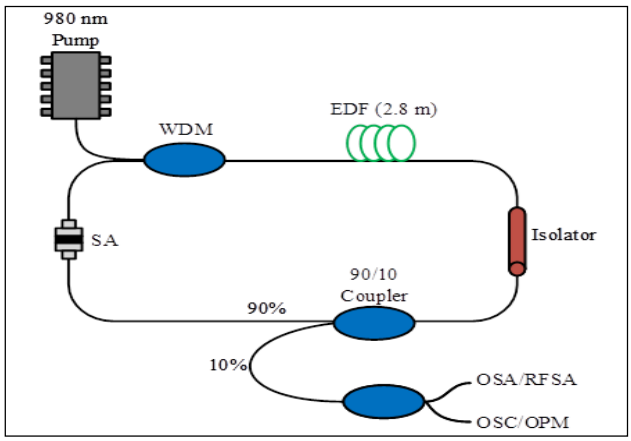
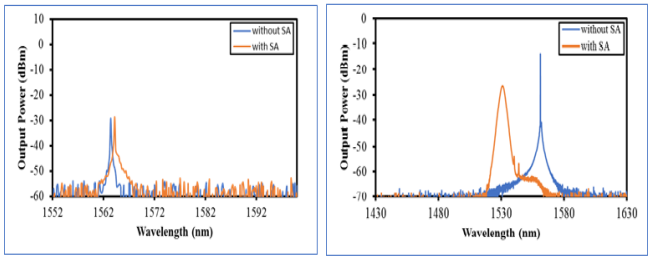


Figure 3. Schematic of experimental setup

The optical spectrum analyzer (OSA) traces with and without graphene-PVA and graphene-PEO as the SA are shown in Figure 4. Note that the recorded spectrums are for respective threshold input pump power. For graphene-PEO SA, threshold pump power of 35 mW will shift the central wavelength from 1563.4 nm to 1564.2 nm as shown in Figure 4(a). Based on our measurement, the maximum permissible input pump power is 55 mW which generates maximum peak power of 0.53 mW. In Figure 4(b), the laser cavity provided a continuous wave with a central wavelength of 1561.1 nm for the graphene-PVA SA. Threshold pump power of 39.2 mW will shift the central

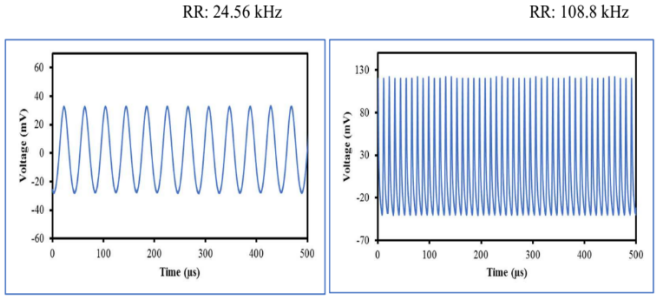
wavelength to 1531.1 nm. It is to be noted that the maximum permissible pump power is 236.3 mW which generates a peak power of 0.8 mW.



(a) (b)

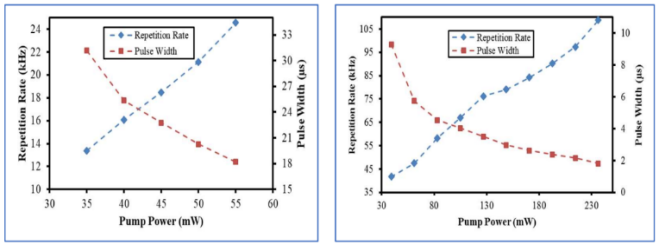
Figure 4. OSA traces for (a)graphene-PEO (b)graphene-PVA

Oscilloscope (OSC) traces for graphene-PEO and graphene-PVA are shown in Figure 5. Note that Q-switched pulses have been recorded with maximum repetition rate (RR) of 24.56 kHz and 108.80 kHz for graphene-PEO and graphene-PVA, respectively. These findings vindicated the shortest pulse width of 1.85 µs for graphene-PVA and 18.1 µs for graphene-PEO. Detail observation can be made by referring to Figure 6 which exhibits the pulse width and repetition rate variation with applied pump power. Meanwhile, calculated pulse energy and peak output power for maximum pump power are summarized in Table 1.



(a) (b)

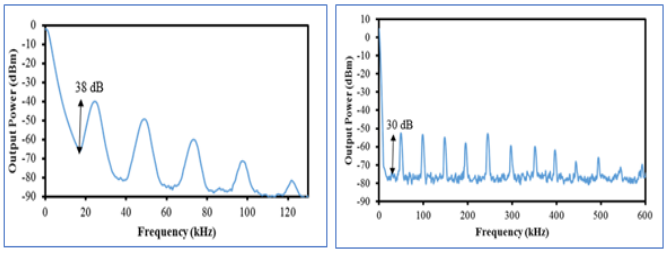
Figure 5. OSC traces for maximum applied pump power of (a) graphene-PEO (b) graphene-PVA



(a) (b)

Figure 6. Pulse width and repetition rate variation with pump power for (a) graphene-PEO (b) graphene-PVA

Radio Frequency Spectrum Analyzer (RFSA) measurement for stability assessment is graphically shown in Figure 7. The recorded signal to noise ratio (SNR) of graphene-PVA and graphene-PEO is 30 dB and 38 dB, respectively. Ultimately, the stability for both characterized SAs have been confirmed with these recorded values which agree with the previous published work on stability condition [21-23].



(a) (b)

Figure 7. RFSA measurement for (a) graphene-PEO (b) graphene-PVA

Table 1 summarizes comparisons of measured parameters between graphene-PEO and graphene-PVA for 12.04 wt.% graphene to solvent weight ratio. Based on these comparisons and presented figures, it is confirmed that the Q-switching performance of graphene-PVA SA is better as compared to graphene-PEO at 1.5 µm region. Although the calculated pulse energy of graphene-PEO is higher, other important parameters particularly the repetition rate, pulse width and maximum peak power are dominated by graphene-PVA. Most importantly, we have confirmed the ability of graphene-PVA based SA to withstand higher pump power, which is significant and agrees with previous reported work [5, 10, 23-25].

Table 1. Performance comparison of graphene-PEO and graphene-PVA

Parameters	Saturable absorber	
	Graphene-PEO (12.04 wt.%)	Graphene-PVA (12.04 wt.%)
Range of pump power (mW)	35-55	39.2-236.3
Threshold pump power(mW)	35	39.2
Central wavelength (nm)	1564.2	1531.1
Repetition rate (kHz)	13.38-24.56	41.70-108.80
Shortest pulse width (µs)	18.10	1.85
Pulse energy (maximum) (nJ)	10.18	0.16
Peak power (maximum) (mW)	0.53	0.80
Signal to Noise ratio (dB)	38	30

4. CONCLUSION

In this paper, performances of PVA and PEO as a host polymer for graphene based SA have been compared and demonstrated. A single concentration of 12.04 wt.% of graphene-PVA and graphene-PEO multi-layered films have been fabricated using a low-cost electrochemical exfoliation method. By applying 2.8 meters of EDF gain medium, Q-switched pulse train have been generated at 1.5 µm region for both type of SAs. Based on our temporal measurement, the graphene-PVA is better than graphene-PEO which shows that the former produced shorter pulse width of 1.85 µs as compared to 18.10 µs for the latter. For stability measurement, both SAs produced stable pulses with SNR >30 dB. In power related measurement, the maximum peak power is 0.80 mW and 0.53 mW for graphene-PVA and graphene-PEO, respectively. Although the calculated pulse energy of graphene-PEO is higher, the

ability of graphene-PVA to withstand higher pump power which has been observed in this work, together with superior temporal and power related parameters, make PVA a preferred binding host polymer as compared to PEO. Indeed, we have successfully compared for the first time, performance of graphene-based SA with PVA or PEO as the binding material. Additionally, the significant results reported here will surely assist future related work of SA fabrication and characterization.

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