

**CHEMICAL SYNTHESIS AND CHARACTERIZATION OF  
GRAPHENE OXIDE FOR USE AS SATURABLE  
ABSORBER AND BROADBAND POLARIZER**

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**THESIS SUBMITTED IN FULFILMENT OF THE  
REQUIREMENTS FOR THE DEGREE OF DOCTOR OF  
PHILOSOPHY**

**FACULTY OF SCIENCE  
UNIVERSITY OF MALAYA  
KUALA LUMPUR**

**2015**

**UNIVERSITY OF MALAYA**  
**ORIGINAL LITERARY WORK DECLARATION**

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Name of Degree: DOCTOR OF PHILOSOPHY

Title of Project Paper/Research Report/Dissertation/Thesis (“this Work”):

CHEMICAL SYNTHESIS AND CHARACTERIZATION OF GRAPHENE OXIDE  
FOR USE AS SATURABLE ABSORBER AND BROADBAND POLRIZER

Field of Study: LASER & PHOTONICS

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## ABSTRACT

Chemically derived graphene oxide (GO) is an atomically thin sheet of graphite that has traditionally served as a precursor for graphene. The tunability of the ratio of its  $sp^2$  and  $sp^3$  hybridized carbon atoms is a great way to tune its bandgap for different applications. Manipulation of the size, shape and relative fraction of the  $sp^2$ -hybridized domains of GO enables the tailoring of its optoelectronics properties.

This thesis presents works undertaken on the chemical synthesis, characterization and photonics applications of GO. The GO is synthesized using simplified Hummers' method. The emphasis of the synthesis method is on the experimental time, whereby the long experimental time spent during the oxidation process, typically on mixing the reactants and cooling or heating the reactants have been reduced from 3 – 5 hours to less than 5 minutes. The whole process was carried out without any temperature control, neither increasing nor decreasing the temperature. The mixture was stirred at room temperature for only 3 days and a high degree of oxidation is achieved.

Characterization of the area, size, morphology and physical properties of the GO is done using Atomic Force Microscopy (AFM), Field Emission Scanning Electron Microscopy (FESEM), X-ray Diffractometry (XRD), Raman spectroscopy, Fourier Transform Infrared (FTIR) spectroscopy and Thermogravimetric Analysis (TGA). For the nonlinear optical properties of the GO, a set of wide dynamic range power dependent transmission and absorption measurement apparatus and technique are constructed and developed, enabling the determination of some important parameters such as saturation intensity, modulation depth and non-saturable loss.

Based on the characterizations, the GO is used as a saturable absorber (SA). The GO was transferred onto a fibre ferrule using 'dip-coating' and thin film transfer method.

Reliable  $Q$ -switched fibre lasers were successfully demonstrated. Pulse duration as short as  $\sim 4.0 \mu\text{s}$  and pulse repetition rate of  $\sim 60.0 \text{ kHz}$  had been achieved. Comparison between GO with reduced graphene oxide (rGO) as a  $Q$ -switching device in the same laser resonator was carried out and studied.

Finally, to harness the wave-guiding and anisotropic dielectric properties of GO, a broadband waveguide polarizer is realized by coating GO on a polymer optical waveguide. The polarization state of the polarizer output was measured in free space using a polarimeter. The extinction ratio of the GO based polarizer is found to be approximately 40 dB at 1590 nm. This is the highest extinction ratio ever reported for graphene-based waveguide polarizer to date.

## ABSTRAK

Graphene oksida ( GO ) yang diperolehi secara kimia adalah lembaran nipis atom grafit dan digunakan sebagai pelopor untuk graphene mengikut tradisi. Kebolehtalaan nisbah pecahan  $sp^2$  dan  $sp^3$  adalah cara yang bagus untuk menala jurang jalur untuk kegunaan yang berlainan. Manipulasi saiz, bentuk dan pecahan relatif domain  $sp^2$  terhibrid GO membolehkan penyesuaian sifat-sifat optoelektroniknya .

Tesis ini menyampaikan kerja-kerja yang dilaksanakan pada sintesis kimia , pencirian dan kegunaan fotonik GO . GO disintesis dengan menggunakan kaedah Hummers yang dipermudahkan. Penekanan cara sintesis ini adalah pada masa eksperimen, di mana waktu eksperimen panjang yang dihabiskan semasa proses pengoksidaan, biasanya pada mencampurkan bahan-bahan tindak balas dan menyejukan atau memanaskan bahan-bahan tindak balas telah dikurangkan dari 3 - 5 jam ke kurang dari 5 minit . Seluruh proses ini dilakukan tanpa kawalan suhu , tidak meningkatkan atau menurunkan suhu . Campuran itu dikacau pada suhu bilik selama hanya 3 hari dan takat pengoksidaan tinggi dicapai .

Pencirian keluasan, saiz, morfologi dan sifat-sifat fizik GO dilakukan dengan menggunakan Mikroskopi Daya Atom, Mikroskopi Elektron Pengimbas Pancaran Medan, Belauan sinar-X, spektroskopi Raman, spektroskopi Infra-Merah Ubahan Fourier dan Analisis Termogravimetri. Untuk sifat-sifat optik tak linear GO, satu set alat dan teknik ukuran penghantaran dan penyerapan yang bergantung kepada kuasa yang berjulat dinamik meluas dibina dan diperkembangkan, membolehkan penentuan beberapa parameter yang penting seperti keamatan tertepu, kedalaman modulasi dan kehilangan tak linear.

Berdasarkan pencirian-pencirian tersebut, GO digunakan sebagai penyerap boleh tepu. GO dipindahkan ke atas ferrule serabut secara saluran celup dan pemindahan film nipis. Laser serabut optik  $Q$  tersuis yang boleh diyakini telah berjaya ditunjukkan. Tempoh denyutan sesingkat  $\sim 4.0 \mu\text{s}$  and kadar ulangan denyutan  $\sim 60.0 \text{ kHz}$  telah dicapai. Bandingan antara GO dengan GO yang dikurangkan sebagai suis  $Q$  dalam rongga resonans yang sama telah dijalankan dan dikaji.

Akhirnya, untuk memanfaatkan sifat-sifat memandu gelombang dan dielektrik anisotropik GO, sebuah pengutub pemandu gelombang jalur lebar direalisasikan dengan menyalurkan GO pada suatu pemandu gelombang optik polimer. Keadaan pengutuban output pengutub diukur pada ruang bebas dengan menggunakan polarimeter. Nisbah kepupusan pengutub berdasarkan GO didapati adalah lebih kurang 40 dB pada 1590 nm. Ini adalah nisbah kepupusan paling tinggi yang dilaporkan untuk pengutub pemandu gelombang berdasarkan graphene setakat ini.

## ACKNOWLEDGEMENTS

The Living and Almighty God whom I believe in deserves the highest praise and honour for sustaining me through the 3 years of unusual experience of research and study. My family, typically my parents, deserve a place only second to God. Their love, support and understanding are a unfailing source of motivation and encouragement.

Distinguished Professor Datuk Dr Harith Ahmad, my supervisor, deserves special thanks for his patience and valuable advice to me. Professor Datuk Dr Harith brought me into graphene research, opened my eyes to the endless opportunities of graphene photonics and optoelectronics, and convinced me that graphene research is worth doing.

A big ‘thank you’ is conveyed to my co-supervisor, Professor Dr Sulaiman Wadi Harun, for supporting me wholeheartedly in all laboratory work. I owe my debt to the University of Malaya, for granting me the University of Malaya Scholarship Scheme.

Dr Nay-Ming HUANG needs special mention here. He shared with me many of his quality work on graphene oxide, and graciously granted me the opportunity and permission to learn the synthesis method and use all the facilities at the Low Dimensional Material Research Centre.

To Emeritus Professor Richard De La Rue (University of Glasgow), Dr Wu-Yi CHONG, Dr Chang-Hong PUA, Dr Kok-Sing LIM, Mr Leonard Bayang, Mr Weng-Hong LIM, my heartfelt thanks is due for their selfless support, and the joy and friendship they offered to me while working together in the research laboratory.

Many other caring individuals could still be added to the list. For all those who helped me in one way or the other, God remembers and shall reward you richly Himself.

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## LIST OF SYMBOLS AND ABBREVIATIONS

AFM	:	Atomic Force Microscopy
ASE	:	Amplified Spontaneous Emission
Bi-EDFL	:	Bismuth-based Erbium-doped Fibre Laser
CNT	:	Carbon Nanotube
CW	:	Continuous wave
EDFA	:	Erbium-doped Fibre Amplifier
EDFL	:	Erbium-doped Fibre Laser
FESEM	:	Field Emission Scanning Electron Microscope
FTIR	:	Fourier Transform Infrared
FWHM	:	Full-Width at Half-Maximum
GO	:	Graphene Oxide
NMP	:	N-Methyl-2-Pyrrolidone
RFSA	:	Radio Frequency Spectrum Analyzer
rGO	:	Reduced Graphene Oxide
SA	:	Saturable Absorber
SEM	:	Scanning Electron Microscope
SESAM	:	Semiconductor Saturable Absorber Mirror
SWCNT	:	Single Wall Carbon Nanotube
TBF	:	Tunable Bandpass Filter
TE	:	Transverse Electric
TGA	:	Thermogravimetric Analysis
TM	:	Transverse Magnetic
VOA	:	Variable Optical Attenuator
XRD	:	X-ray Diffractometry

This Thesis Is Dedicated to

My Parents

Who Share My Joy and Tears, Providing Me With The Best in Education

Emeritus Professor Dr Kum-Sang LOW

My Physics Professor Who Opens My Eyes  
To The Endless Horizon of Science and Research

*Sifu* David Michael Peterson

My Martial Art Instructor Who Blasts My Mind  
In The *Wong Shun Leung Method* of *Ving Tsun*,  
Where I Realize That Fundamental Laws of Physics Can Be  
Simple, Direct and Efficient  
In Fighting

Shelian Stephen

Who Shares My Joy, My Grief of Life, Faith and Work