UNIVERSITI PUTRA MALAYSIA

STUDY OF THERMAL DIFFUSIVITY OF LIQUID REDUCED GRAPHENE OXIDE USING THERMAL WAVE RESONANT CAVITY TECHNIQUE

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By

ROSNO KINSU

Thesis Submitted to the School of Graduate Studies, Universiti Putra Malaysia, in Fulfilment of the Requirements for the Degree of Master of Science

December 2017

Abstract of thesis presented to the Senate of Universiti Putra Malaysia in fulfilment of the requirements for the degree of Master of Science.

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Chair : Nor Kamilah Sa'at, PhD
Faculty : Science

Graphene oxide (GO) exhibit excellent thermal properties where it has ability to diffuse heat efficiently. By reducing the GO to the reduced graphene oxide (rGO), the thermal diffusivity (TD) can be enhanced. The aim of study are to measure various standard liquid thermal diffusivities by employing a Thermal Wave Resonant Cavity (TWRC) technique, and secondly to study the thermal, optical property as well as morphology of rGO fabricated by ultra violet (UV) and Nd:YAG laser radiations at various exposure time. In order to achieve the first objective, the liquid thermal diffusivities (water, glycerol and ethylene glycol) were measured by using the TWRC technique and calculated via a normalisation procedure. For this, a control program in LabView programming language was written to automate the set-up in measuring the TD. For the second objective, it was achieved by reducing GO in water suspension with UV and Nd:YAG laser irradiation. The thermal, optical, morphological properties of it were investigated by TWRC technique, UV-Vis spectroscopy, X-Ray Photoelectron Spectroscopy (XPS), and a Field-emission Scanning Electron Microscopy (FeSEM).

Without the normalization process on the TWRC technique, TD of standard liquid water obtained by the cavity scan is 1.431×10^{-3} and 1.435×10^{-3} cm^2s^{-1} for phase and amplitude, respectively, and very close to literature (0.5%). This shows that the current TWRC set up is reliable enough and can be used to measure liquid TD. With the normalisation process, TD of water with for frequency scan of amplitude signal with respect to literature improves tremendously to 1.6% but with a little increase for that of phase signal, 3.5%. For cavity scan, TD of water for amplitude signal and for phase are 0.6% and 1.9%, respectively, with respect to literature which are very small indeed. Over all this agrees with the first hypothesis.

The TD measurement revealed that the rGO produced with UV radiation has higher value compared to that of with laser. The trend is apparent when the irradiation time increased. Accordingly, its TD decreased at higher rGO concentration. FeSEM images revealed that the UV radiation reduced graphene oxide (UVrGO) sample has more ripples, deeper wrinkles, and fluffier texture compared to that of laser reduced graphene oxide (LrGO). The coarser rGO surface suggests the higher rate of reduction. The UV-Vis peak of UVrGO has shifted further to red than that of LrGO, complimenting morphology results. The XPS survey analysis showed that atomic concentration of carbon increases whilst the concentration of carbon containing C-O, and C=O group decreases. Hence, verifying the reduction of GO to rGO. As a conclusion, exposing GO to UV or laser radiation will reduce it to rGO, consequently increasing its TD. The reduction rate was highly influenced by type of treatment and exposure time. It was found that UVrGO give higher reduction rate and higher increase in TD when the exposure time increased as compared to LrGO. These findings have given a promising future for rGO to be further studied as an alternative to conventional liquid conductors.

Abstark tesis ini diserahkan kepada Senat Universiti Putra Malaysia sebagai memenuhi keperluan untuk ijazah Master Sains

KAJIAN KEMERESAPAN TERMA BAGI GRAFIN OKSIDA DITURUN MENGGUNAKAN TEKNIK RONGGA RESONAN GELOMBANG TERMA.

Oleh

ROSNO KINSU

Disember 2017

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Grafin oksida (GO) menunjukkan sifat terma yang unggul, bahan ini boleh meresapkan haba secara efisien. Keresapan terma (TD) bahan ini boleh ditingkatkan dengan menurunkan GO kepada grafin oksida terturun rGO. Matlamat kajian ini adalah untuk mengukur TD beberapa cecair piawai menggunakan teknik rongga resonan gelombang terma (TWRC), dan keduanya adalah untuk mengkaji sifat optik serta morfologi rGO yang dihasilkan menggunakan sinaran ultra violet (UV) and laser Nd:YAG yang didedahkan pada masa penyinaran yang berbeza. Untuk mencapai matlamat pertama, TD (air, gliserol, dan etilena glikol) telah diukur menggunakan teknik TWRC dan dikira menggunakan prosedur penormalan. Bagi tujuan ini, satu program kawalan menggunakan bahasa pengaturcaraan Lab View telah dibangunkan untuk mengautomasikan peralatan untuk mengukur TD. Untuk objektif kedua, GO dalam larutan air diturunkan menggunakan sinaran UV dan ablasi laser Nd:YAG. Sifat terma, optik dan morofologi dikaji menggunakan teknik TWRC, spektroskopi UV-Vis, fotoelektron X-Ray (XPS), dan mikroskop elektron imbasan medan pancaran (FeSEM).

Tanpa proses penormalan ke atas teknik TWRC, TD cecair air piawai yang didapati dari imbasan rongga adalah 1.431×10^{-3} dan 1.435×10^{-3} cm^2s^{-1} untuk fasa dan amplitud, masing-masingnya, dan mendekati nilai kepada literatur (0.5%). Ini menunjukkan bahawa set up TWRC semasa boleh dipercayai dan boleh diguna untuk mengukur TD cecair. Tanpa proses penormalan, TD air melalui imbasan frekuensi dari signal amplitude merujuk kepada literature meningkat secara mendadak kepada 1.6% tetapi dengan kenaikan kecil sahaja untuk signal fasa, 3.5%. Untuk imbasan rongga, TD air untuk signal amplitude dan fasa adalah 0.6% dan 1.9%, masing-masingnya, dengan merujuk kepada literature yang mana sesungguhnya adalah terlalu kecil. Secara keseluruhan ini adalah bersetuju dengan hipotesis pertama.

Pengukuran TD menunjukkan GO yang didedahkan kepada sinar UV mempunyai nilai yang lebih tinggi berbanding dengan yang didedahkan kepada laser. Trend ini lebih jelas apabila masa penyinaran bertambah. Nilai TD didapati menurun apabila kepekatan larutan bertambah. Imej FeSEM sampel UVrGO menunjukkan lebih kedutan yang agak dalam dan mempunyai tekstur yang kelihatan lebih empuk berbanding LrGO. Permukaan rGO yang lebih kasar menunjukkan kadar penurunan yang lebih tinggi. Puncak UV-Vis bagi UVrGO menunjukkan anjakan yang lebih besar ke arah panjang gelombang yang lebih panjang berbanding (red shift) LrGO, mengukuhkan lagi analisis morfologi. Analisis XPS juga menunjukkan kepadatan atom karbon bertambah manakala kepadatan kumpulan mengandungi karbon C-O, dan C=O menurun. Keputusan analisis-analisis telah mengesahkan penurunan GO kepada rGO. Sebagai kesimpulan, GO dapat diturunkan kepada rGO dengan mendedahkannya kepada sinaran UV dan laser, seterusnya meningkatkan TD nya. Kadar penurunan dipengaruhi oleh jenis sinaran dan masa penyinaran. Didapati bahawa UVrGO memberikan kadar penurunan yang lebih tinggi serta peningkatan TD yang lebih tinggi apabila masa penyinaran bertambah berbanding dengan LrGO. Dapatan kajian ini memberikan ruang bagi rGO untuk kajian lebih lanjut pada masa hadapan sebagai alternatif untuk konduktor cecair konvensional.

ACKNOWLEDGEMENTS

I would like to express my sincere gratitude to Dr. Nor Kamilah Sa'at for her supervision, invaluable support, and guidance throughout the completion of this thesis. I also would like to extend my gratitude to my previous retired supervisor Prof. Dr. Azmi Zakaria for his patience and constant support for my study at all time. This research will never been completed without his persistence guidance and supervision. His expertise, skills, and perseverance had inspired me for further exploring the exciting world of physics.

My sincere appreciation goes to my supervision committee members, Assoc. Prof. Dr. Zulkifly Abbas and also to Dr. Raba'ah Syahidah Azis for their suggestions, comments and assistance.

Appreciation is also given to all staffs of Physics Department of Faculty of Science, Institute of BioScience UPM, Pusat Pengajian Fizik Gunaan UKM for their assistance. I would also express my deepest gratitude to my friends and colleagues, Dahiru Umaru, Mohd Abdul Azeez Nareeman, Tasiu Zangina, Ammar Abd Ali Najm, Nor Hasanah Isa, and Noraznirashaila Sakeri, amongst other for their cooperation, encouragement and assistance to complete this study.

There can be no adequate acknowledgement to my wife and children, parents, and family members for their encouragement, support and patience that make all this tasks possible.

I am grateful to the Ministry of Education Malaysia for granting me a study leave.

I certify that a Thesis Examination Committee has met on 28 December 2017 to conduct the final examination of Rosno bin Kinsu on his thesis entitled "Study of Thermal Diffusivity of Liquid Reduced Graphene Oxide using Thermal Wave Resonant Cavity Technique" in accordance with the Universities and University Colleges Act 1971 and the Constitution of the Universiti Putra Malaysia [P.U.(A) 106] 15 March 1998. The Committee recommends that the student be awarded the Master of Science.

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This thesis was submitted to the Senate of Universiti Putra Malaysia and has been accepted as fulfilment of the requirement for the degree of degree of Master of Science. The members of the Supervisory Committee were as follows:

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LIST OF ABBREVIATIONS

FWHM	Full width of half maximum
GO	Graphene oxide
LabVIEW	Laboratory Virtual Instrument Engineering Workbench
LrGO	Laser reduced graphene oxide
OD	Optical density
PVDF	Polyvinylidene difluoride
PE	Pyroelectric
PPE	Photopyroelectric
PT	Photothermal
R	Optical reflectivity
rGO	Reduced graphene oxide
TD	Thermal diffusivity
TW	Thermal wave
TWRC	Thermal wave resonant cavity
UV	Ultra violet
UVrGO	Ultra violet reduced grapheme oxide
VI	Virtual instrument

LIST OF SYMBOLS

α	Thermal diffusivity
C	Specific heat
ε	Thermal effusivity
f	Modulation frequency
m	Slope or gradient
L / l	Thickness
λ	Wavelength
ρ	Density
K	Thermal conductivity
μ	Thermal diffusion length
ω	Angular modulation frequency
V	voltage
Ø	Phase

CHAPTER 1

INTRODUCTION

1.1 Introduction

Graphene has attracted so much attention due to its superior characteristics of thermal and electrical properties (Sur, 2012) that promises a better solution for industries that will enhance the recent thermal management technology (Zhang et. al., 2015) to a higher standard. Graphene is a very promising candidate as alternative to conventional conductors (Feng et.al., 2016). Reduced graphene oxide (rGO) is a material which has a graphene like properties. rGO is produced by removing the oxygen content in graphene oxide (GO), by exposing GO to radiation, using chemical method, or heat treatment. One of the parameter used to characterise the thermal properties of a material is the thermal diffusivity (TD).

TD is an important thermophysical parameter used in the design of heat transporting systems, which can also be used in identification and diagnostics of materials of practical interest (Touloukian et. al., 1974) .

There are many techniques employable in measuring TD. Among them, photothermal (PT) techniques have been proven very useful in the measurement of thermal diffusivities (Mandelis and Lymer, 1985) because of its simplicity and reliability.

Measuring the TD manually is quite a tedious task, it is time consuming process required high skill in handling the instruments and could lead to errors in measurement. For that reason, automation is required to speed up the processes and reducing parallax errors.

1.2 Photopyroelectric detection

Pyroelectricity is the ability of a material to generate momentary voltage when heated or cooled (Wan and Bowen, 2017). This property is due to spontaneous polarization, P_s, in certain anisotropic solids (Lang, 2005) .

The change in temperature, $\frac{dT}{dt}$ causes the changes in spontaneous polarisation thus changing the amount of bound charges, the unbalanced distribution of charges create potential difference which lead to redistribution of free charges as illustrated in

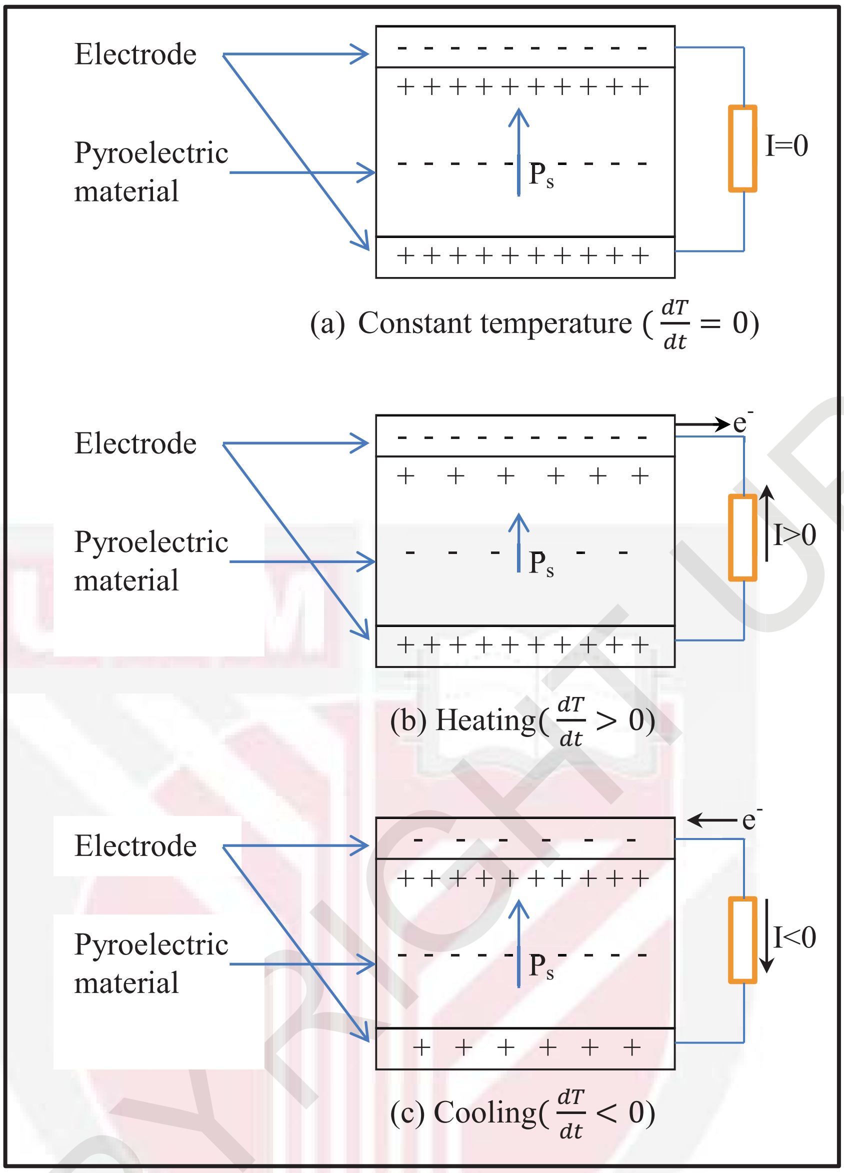

Figure 0.1: (a) At constant temperature, (b) When the temperature is increased, and (c) When the temperature is decreased.

Figure 0.1. Movements of charges result in current flow. Therefore this effect is present only during the temperature change period.

This unique property is being put to good use by using materials such as polyvinylidenefluoride (PVDF) as a transducer in changing thermal signal into electrical signal. In photopyroelectric (PPE) technique this transducer is placed in the pyroelectric (PE) cell in front or at the back of the sample, to sense the thermal signal carried in the thermal wave (TW).

In case of "thermal wave resonant cavity" (TWRC) technique, the TW generated by the TW generator will propagate through the liquid sample and reaches the boundaries of sample-PVDF, a fraction of the wave will be reflected and a portion are transmitted into the PE transducer. The transmitted TW is converted into electrical signal in form of phase and amplitude that can be used to determine the TD of the sample. The value of the output signal is depending in the amount of transmitted TW and the amount of transmitted TW is depending on the thermal properties of the sample material.

1.3 Thermal diffusivity

The heat is the energy flow due to different in temperature, in real situation it occurs in form of conduction, convection and radiation. For experimental purpose only one will be considered, in this case is the conduction. The specific heat capacity, C, thermal conductivity, K, and thermal diffusivity (TD), α, are among the term normally used characterise thermal property of a material.

For isotropic material (Berinskii, 2013) like graphene, only C is needed in solving related problem. K is contributed by only thermal conductivity of component in material and it is able to deal with steady state problem when temperature does not vary over time, and α depends on both thermal conductivity and TD of a material, therefore will give a comprehensive characterisation.

When heat, Q is supplied to a mass, m and contribute to temperature, T rise can be express as

$$dQ = mCdT \qquad 1.1$$

where C is the amount of heat required to change the temperature of a unit mass in a unit of temperature. The specific heat capacity, C measures the ability of a material to store internal energy.

The heat flow, $\vec{Q}$ between two media due to the existence of a temperature gradient, $\vec{\nabla}T$ in a homogeneous isotropic material is given by Fourier law.

$$\vec{Q} = -K\vec{\nabla}T \qquad 1.2$$

where K is the heat flows in a unit time through a unit of area of layer of material of unit thickness with temperature difference between its faces, or the ability of material to transport the heat from one point to another.

In the case of temperature dependent, the thermal properties of homogeneous isotropic material whereas there is no heat generated inside it is given by,

$$\nabla^2 T - \frac{1}{\alpha}\frac{dT}{dt} = 0\ . \qquad 1.3$$

The TD, α is given by Laplace equation. It measures the change in temperature produced in a unit volume of material by the amount of heat that flows in unit time through a unit area of layer material of unit thickness with temperature difference between its faces, or the speed of heat propagation during the change of temperature per unit time.

The TD, thermal conductivity, and the specific heat capacity are interrelated to each other. The TD is in direct proportion to thermal conductivity, K but inversely proportional to the heat capacity, C and density, ρ of a material. Mathematically,

$$\alpha = \frac{K}{\rho C} \,. \qquad 1.4$$

It measures the rate of transfer of heat of a material from the hot side to the cold side. The unit of TD is centimetre square per second, $cm^2\ s^{-1}$.

1.4 Research problem

The accuracy in obtaining the liquid TD of a few standard liquids by photothermal method is a challenging effort in order to claim the importance of this method. Therefore, the robustness in data processing and a certain way of calculating the TD rather than that of the conventional one is needed. Secondly, a reduced graphene oxide liquid is known to have good heat conductivity. Therefore the easy production of this liquid and the TD study of it at various concentrations are important for heat conductivity application in thermal cooling application.

1.5 Objectives

The objectives for this research are;

1. to study various standard liquid thermal diffusivities by employing a TWRC technique calculated by normalisation process,

2. to study the thermal, optical properties as well as morphology of reduced graphene oxide (rGO) fabricated by ultra violet (UV) and Nd:YAG laser irradiation at various exposure time.

1.6 Hypothesis

i) The normalisation process from the TWRC technique would increase the accuracy of the liquid standard sample TD.

ii) The TD of rGO would increase as both UV and laser irradiation time increase.

1.7 Scope and limitation

The scope and limitation of this work are:

i. Only LabView and C programming language will be used to develop the interfacing program.

ii. Only light irradiation from laser and UV sources will be applied in this study.

iii. The study will be limited to commercialized liquid GO.

iv. The PVDF sensor used in PE technique is limited to only single type.

1.8 Thesis outline

This thesis focuses on the measurement of various standard liquid thermal diffusivities by employing a TWRC technique calculated by normalisation process and study the thermal, optical property as well as morphology of rGO fabricated by ultra violet (UV) and neodymium-doped yttrium aluminium garnet (Nd:YAG) laser irradiation at various exposure times. The thesis is organized into six chapters.

Chapter 1 gives a general introduction on photopyroelectric detection and TD. It also covers research problem, objectives, hypothesis, scope and limitation. Chapter 2 provides the review of photopyroelectric technique, graphene and graphene oxide. Theory of photopyroelectric effect, thermal wave, graphene oxide and reduced-graphene oxide is covered in Chapter 3. Chapter 4 describes system automation, the materials used, the techniques and procedures, and finally the characterization of the samples. Chapter 5 reports and discusses the results of rGO characterizations. Finally, Chapter 6 concludes the study and suggests the area to be investigated for future studies.

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