NATURAL REDUCTION OF GRAPHENE OXIDE TO GRAPHENE USING KAFFIR LIME LEAVES EXRACT

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Thesis submitted in partial fulfilment of the requirements
for the award of the degree of
Bachelor of Chemical Engineering (Biotechnology)

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JULY 2014

ABSTRACT

Since the reduction of graphene from graphene oxide was early investigate for successful in chemical reduction method using hydrazine and dextran, thermal reduction and also using vitamin C. In this proposal, natural reduction of graphene from graphene oxide is presented by natural reduction for performing higher reduction rate. Graphene oxide was prepared using the method form a part of Hummers Method. Graphite will oxidize to graphene oxide and synthesis to graphene oxide. Then graphene oxide will be reduce for using natural reduction by using kaffir lime leaves extract. Optimization of the reductant concentration and minimum reduction time to produce highest reduction rate was done in this research by the parameter of retention time and kaffir lime leaves extract concentration. The electrochemical properties and the functionalized carbon nanosheets analysis of graphene are examined by the Scanning Electron Microscopy (SEM).

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CVD	Chemical Vapour Deposition
UV	Ultraviolet
SEM	Scanning Electron Microscopy
UV-Vis	Ultraviolet-Visible Spectroscopy
UV/Vis	Ultraviolet-Visible Spectrometer

1 INTRODUCTION

1.1 Background of Study

Graphene has provide huge interest for its excellent mechanical, electrical, thermal and optical properties. Songfeng Pei et al was found that graphene can be synthesis by using micro-mechanical exfoliation of highly ordered pyrolytic graphite, epitaxial growth, chemical vapor deposition, hybridizing polypyrrole (PPY) nanotubes with chemically reduced graphene sheets (Jianhua Liu, 2011), and the reduction of graphene oxide (GO). M.J Fernández-Merino investigate that graphene from graphene oxide reduction using natural reduction method by kaffir lime leaves can be recently considered as a best choice to substitute chemical reduction for example hydrazine. Using kaffir lime leaves for reduction which contains high concentration of chlorophyll as reducing agent and it can be remove easily during separation process without the need of modification. The advantages of natural reduction are the material is ready availability and the potential for commercial use is high due to the economic price.

There are several sources of chlorophyll suitable for reduction of graphene oxide to graphene such as leaves from tree. The reducing method by ethanolic extract of M. oleifera was done by researcher Yen and Chen. Using vegetables for natural reduction are not suitable because it can be consume as food which is more important compare to using food to produce second product.

1.2 Objectives

This work aims to prepare a reduction material from kaffir lime leaves as natural reduction.

a. To identify another cheaper reduction material from previous reduction to replace with chemical reduction method.
b. To identify an easier step of reduction process.
c. To optimize the reduction process based on concentration of reductant and time of reduction to achieved maximum graphene.
d. To study the functionalized carbon nanosheets of graphene using Scanning

Electron Microscopy (SEM).

1.3 Scope of this research

From the beginning, graphene oxides will be synthesized from graphite powder to graphite oxide, then synthesis to commercial graphene oxide. Besides that, the scope of this research is to prepare reducing agent from natural resource for graphene oxide reduction. The next scope is to reduce the graphene oxide after the graphene oxide synthesis to graphene by natural reduction using kaffir lime leaves extract. Further purification of graphene via ultrasonic and washing with deionise water and filtration will be carried out as the separation process. Then, another scope was continued to optimization of the retention time and the concentration yield of the kaffir lime leaves extract. In addition, a qualitative measurement was done by using Scanning Electron Microscopy (SEM) to study the functionalized carbon nanosheets of graphene.

1.4 Main contribution of this work

Current reduction of graphene from graphene oxide can be done by micro-mechanical exfoliation which is high ordered pyrolytic graphite by Geim AK et al 2007,chemical vapour deposition (CVD) and epitaxial growth (Berger C et al, 2006). Z.J. Fang, 2010 was investigating that using chemical reagent in the reduction process are very critical for graphene oxide reduction. Therefore, R. S. Pantelic, 2010 was proven that had disadvantages on using chemical reagent because natural response of graphene to detect analyte may be buried by the signals caused by impurities. For example, reduction using hydrazine in large-scare reduction is not desirable because it is high toxicity (M.J Fernandez-Merino et al, 2010). Kaffir Lime leaves was a material that is cheap abundant in Malaysia, easily to in Malaysia Resource and it is natural reduction that use organic chemical which can remove easily by evaporation. High chlorophyll concentration can be extract from kaffir lime leaves has characteristic of reducing agent in present of UV light. It is considered as non-toxic material that extract from plant, while it can converting UV light into chemical energy for reduction process of graphene oxide in room temperature.

1.4.1 Application of Commercial Synthesis of Graphene in Industry

Graphene from the process of reduction graphene oxide has been fascinated as a great interest for the removal and detection of environmental pollutants because it had special physicochemical properties. For the application of environment protection, adsorption of heavy metal ions can be done in a high selectivity and high efficiency, and hence reduces them become recycling metal due to it high adsorption capacity. Besides that, heavy metal ions, organic and toxic gases pollutants in environment were reported to detect from some graphene-based sensors with great limit of detection. The reduction process to graphene from graphene oxide has continued over some years.

1.4.2 Potential for Future

There are several sources of chlorophyll suitable for reduction of graphene oxide to graphene such as leaves from tree. The reducing method by ethanolic extract of M. Oleifera was done by researcher Yen and Chen. Using vegetables for natural reduction are not suitable because it can be consume as food which is more important compare to using food to produce second product. This open a new avenue of natural reduction of graphene oxide by using leaves from tree, which is kaffir lime leaves and it can be used in this work. Most of the kaffir lime leaves can be found easily in Malaysia, Thailand, Vietnam and Indonesia. It producing more than 800 tons of kaffir lime leaves annually and this project aims to utilise these leaves for the reduction of graphene oxide. The kaffir lime leaves have characteristic of aroma that identified as (−)-(S)-citronellal, which is present in the leaf oil over 80%, small components included citronellol (10%), nerol and lomonene. The small amount of citronellol can also be reducing agent which is acidic compound as reducing agent to contribute in the reduction process of graphene oxide.

Natural reduction of graphene oxide to graphene using kaffir lime leaves which can be consider as the most easier and most cheaper process compare to using thermal reduction and chemical reduction. Preparation of chlorophyll extract is very simple which are using commercial blender that blend with kaffir lime leaves with sufficient amount to produce a simplest and cheapest chlorophyll extract. Then the reduction method can be continued by adding in graphene oxide. It can be done by large scale reduction by using the non-toxicity kaffir lime leaves extract as reductant. Therefore, these researches are providing a simplest, cheapest and commercial for large scare industry to prepare commercial graphene powder from cheap graphite powder.

1.5 Organisation of this thesis

The structure of the reminder of the thesis is outlined as follow:

Chapter 2 provides general study background for the different type of reduction and the reduction mechanism. In details of graphene oxide reduction mechanism, disadvantages of the previous graphene oxide reduction were stated in this chapter. The information of qualitative test in this research which is electrochemical analysis of the graphene by natural reduction was also included in this chapter. In addition, the method chosen for graphene oxide reduction included purpose of choosing graphene oxide reduction method using kaffir lime leaves extract, the physical and chemical properties of kaffir lime leaves extract, an briefly introduction of chlorophyll reduction and the advantages of choosing kaffir lime leaves extract for graphene oxide reduction.

Chapter 3 gives a method of synthesis graphene oxide from graphite powder using a part of Hummers Method. Full methodology of preparation a reductant which is kaffir lime leaves extract and the separation method was also provided in this thesis. The reduction process of graphene oxide to graphene and optimization of the reduction by retention time and concentration was also provided. Besides that, a qualitative analysis was done in this research which is Scanning Electron Microscopy (SEM) to study the functionalized carbon nanosheets of graphene.

Chapter 4 is devoted to the result and discussion of the graphene oxide synthesis, reduction of graphene oxide, optimization of concentration and reduction time to achieved maximum graphene. The result of qualitative analysis to determine the graphene reduce by kaffir lime leaves using Scanning Electron Microscopy (SEM) was also will be discuss at this chapter.

2 LITERATURE REVIEW

2.1 Overview and Introduction

2.1.1 Type of Reduction

Reduction is chemical reactions that consist of gaining electrons. There are two type of reduction process such as acid-base reaction and chemical reaction. For acid-base reaction, the reaction was refer to the electron transfer between hydrogen atom and hydroxide molecule. Chemical reaction is the electron transferring process from an atom to another atom (Fundamental of redox reaction: http://www.shodor.org, 2014)

2.1.2 Reduction Mechanism

In general, reduction process is come together with another process call oxidation. Reduction process cannot occur without present of oxidation. This oxidation-reduction process that concern with election transfer between species was name as redox reaction fundamentally (Fundamental of redox reaction: http://www.shodor.org, 2014)

Oxidation process and reduction process was refer to loss of electron and gain of electron respectively. The example of oxidation process in chemical reaction can be show on the loss of election of copper ion.

$$Cu\ (s) \text{ ----> } Cu^{2+} + 2\ e^-$$

This is the half-reaction from the redox reaction which is the copper is losing of electron as known as oxidation reaction. The symbol "e^-" represents a free electron which is negatively charge was go out from copper atom to reduce others species.

$$2\ Ag^+\ (aq) + 2\ e^- \text{ ------> } 2\ Ag\ (s)$$

For the silver ions which are positively charge are being reduce by the electron to form a solid silver atom. The accepting electron of silver ions in aqueous solution from the oxidation process was known as reduction process.

$$Cu(s) \longrightarrow Cu^{2+}(aq) + 2e^-$$
$$2Ag^+(aq) + 2e^- \longrightarrow 2Ag(s)$$

$$Cu(s) + 2Ag^+(aq) + 2e^- \longrightarrow Cu^{2+}(aq) + 2Ag(s) + 2e^-$$

or

$$Cu(s) + 2Ag^+(aq) \longrightarrow Cu^{2+}(aq) + 2Ag(s)$$

From the equation above, this is the combination of oxidation and reduction reaction from copper atom and silver ions. Here, the chemical reagents that affect another substance to oxidise are call oxidizing agent while the chemical that affect another substance to reduce are call reducing agent. The copper release 2 electrons to make silver ion reduce to silver atom, therefore, the copper atom is reducing agent. The silver ion is accepting electron from copper atom, therefore the silver ion is call oxidising agent.

2.1.3 Graphene Oxide Reduction Mechanism

The conductivity of monolayer graphene mainly relies on carrier transport within the carbon plane, for instant, functional groups bind to the plane was affecting the factor on its conductivity, while functional groups binded to the edge have minimal affect. Basically the reduction process of graphene oxide is mainly aimed at eliminating epoxy and hydroxyl groups on the plane. For carboxyl, carbonyl and ester groups, its present at the edges or defective areas only have a minimal effect on the conductivity of a graphene sheet (Pei S et al, 2011). Reducing graphene oxide using hydrazine, carbonyl groups binded to the graphene oxide are preserved after reduction was investigated by proof Li et al (Li D et al, 2008).

For the reduction process on graphene oxide to graphene, it can be refer to the method of deoxygenation as common removal of oxygen containing groups from the graphene. Chemical reduction also can remove the functional groups in graphene oxide since the highest carbon-oxygen ratio was reported more than 15 (Pei S et al, 2010).

For thermal reduction, the hydroxyl groups can be fully eliminated at temperature of 700-1200^{0}C in vacuum, while carbonyl groups are expected to be slowly reduced at 100-150^{0}C. the carbonyl groups are more stable, therefore it can only be removed at temperature higher than 1730^{0}C (Pei S et al, 2010).

For chemically reduction using hydrazine, the mechanism of this reduction was firstly proposed by Stankovich et al, 2007. The reduction of graphene oxide is begin from the ring-opening of epoxy groups by hydrazine to form hydrazine alcohols, and the first derivative produce by the epoxide opening reacts continue with the formation of an aminoaziridine moiety which is undergoes thermal elimination of di-imide to form a double bond, the result was shown in the re-establishment of the conjugated graphene network (Muller RK et al, 1970; Lathi PM et al, 1983).

2.2 Previous Work on Graphene Oxide Reduction

There are three ways can be synthesize graphene with excellent properties and also in best structure. One of the characteristics of graphene is it can be produce by very cheap graphite as raw material by commercial chemical method with very high yield production (Novoselov KS et al, 2004). Besides that, graphene is extremely high hydrophilic and it can be form a stable aqueous colloids to facilitate the binding of macroscopic structures by easy and inexpensive solution procedure (Geim AK, 2009). Therefore, synthesis of graphene from graphite is very important to the large scare used of graphene (Songfei Pei, 2011). Geim and Novoselov et al. reported a method to synthesis an individual graphene sheet has started with grand scientific activity (Geim AK, 2007). The chemical reduction of graphene oxide is a promising route towards the large scale production of graphene for commercial applications. The current state-of-the-art in graphene oxide reduction, consisting of more than 50 types of reducing agent, will be reviewed from a synthetic chemistry point of view (Chun Kiang Chua, 2014).

2.2.1 Previous Graphene Oxide Reduction Methodology

One of the methods for graphene synthesis based nanosheets was using chemical reduction of exfoliated graphite oxide that investigated by Sasha Stankovich et al, 2007. The researcher used hydrazine hydrate for the reduction process of graphene oxide to graphene. They used graphene oxide add with water to produce yielding inhomogeneous yellow-brown dispersion. After that the mixture will then sonicated by using ultrasonicator bath cleaner until the solution become clear. The hydrazine hydrate will add and heated in oil bath then cooled. The black solid will be produce which is reduce graphene-oxide will then isolated by filtration with washing with water and methanol. When the sludge is dry, the solid product cake is the graphene.

Another method of graphene reduction from graphene oxide was reported by Wufeng Chen et al which are preparation of graphene by using low temperature thermal reduction at atmosphere pressure. The researcher was reported that graphene oxide can be reduced at low temperature which is below 150^0C under the protection of nitrogen gas (Wufeng Chen, 2009). Firstly they add in water to the graphene oxide then sonicated using ultrasonicator until transparent yellow-brown suspension of graphene oxide obtained. Then purge with nitrogen gas then heated. After that the solution was filtered and washes by anhydrous ethanol then water, and freeze-dried to obtained graphene as black powder.

Besides that, Young-Kwan Kim et al,2011 was discovered that the reduction of graphene from graphene oxide can be prepared by using dextran as a multifunctional reducing agent. The researcher was found that the method was begin by the dilution of graphene oxide with water then filtered through mixed cellulose ester membrane then dried. After that it will dissolved in acetone then annealed under argon atmosphere for the removal of dextran.

In additions, Yan Fen et al,2013 was also investigate a new green reduction method using starch based material to reduce graphene oxide such as soluble starch, corn powder, potato and also sweet potato. The graphene was prepared by starch solution in high temperature which is 80°C for 3 hours .

Reduced graphene oxide sheets covalently functionalized with n-type tetrachloroperylene diimide (PDI) and oligomeric PDI have been prepared by a two-step approach. The first step is using graphene oxide (GO) and modified by an amidation reaction with 3-aminopropyldimethy-lethoxylsilane or3-aminopropyldiethoxylmethyl-silane with thermally reduce method. After that, it react with silanol modified PDI by dehydration condensation to obtain PDI and oligomeric PDI modified rGO (rGO–PDI and rGO–(PDI)$_n$). This method are discovered by ZhongJie Ren et al, 2012.

In Kyu Moon et al, 2010 was reported that reducing method using hydriodic acid with acetic acid (HI–AcoH) of a solution-phased RG-o powder and vapour-phased RG-o (VRG-o) paper and thin film. The reducing method provided highly qualified RG-os by mass production, resulting in highly conducting RG-oHI – AcoH. In adition, VRG-oHI – AcoH paper and thin films were prepared at low temperatures (40 °C) and were found to be applicable to flexible devices. . This one-pot method is proven that this research was on highly conducting graphene platelets.

2.2.2 Disadvantages of Previous Graphene Oxide Reduction

Fang et al, 2010 was investigating that using chemical reagent in the reduction process are very critical for graphene oxide reduction. Therefore, R. S. Pantelic et al, 2010 was proven that had disadvantages on using chemical reagent because natural response of graphene to detect analyte may be buried by the signals caused by impurities. For example, reduction using hydrazine in large-scare reduction is not desirable because it is high toxicity (M.J Fernandez-Merino, 2010). Due to the disadvantages of using hydrazine hydrate for graphene oxide reduction, M.J Fernández-Merino et al, 2010, was discovered that vitamin C have potential to substitute hydrazine in the reduction process. The disadvantage of thermal reduction method is it only can stable almost 2 month at room temperature (Wufeng Chen et al, 2009), while the disadvantages of the reduction using dextran is because the hard removal of dextran and it is higher cost compared with others methods (Young-Kwan Kim et al, 2011).

2.3 Analysis Method for Graphene

2.3.1 Electrochemical Analysis

Electrochemical studies are needed to test the biofuntionalization of carbon nanosheet surface. In this work, the functionalized carbon nanosheet must go through the surface characterization procedure using Scanning Electron Microscopy (SEM).

2.4 *Method Chosen for Graphene Oxide Reduction*

2.4.1 *Purpose of Choosing Graphene Oxide Reduction Method Using Kaffir Lime Leaves Extract*

Kaffir Lime leaves was a material that is cheap abundant in Malaysia, easily to found in Malaysia Resource and it is natural reduction that use organic chemical which can remove easily by evaporation. High chlorophyll concentration can be extract from kaffir lime leaves has characteristic of reducing agent in present of UV light. It is considered as non-toxic material that extract from plant, while it can converting UV light into chemical energy for reduction process of graphene oxide in room temperature.

2.4.2 *Physical and Chemical Properties of Kaffir Lime Leaves Extract*

The kaffir lime leaves are dark green colour with a glossy sheen. It's come together in two part which is top and bottom. The top leaflet is lightly pointed at its tip and is attached to another leaflet beneath that is broader on its upper edge. In addition, the size of the kaffir lime leaves is about several inches long (Bai Ma-gkood et al retrieved frommvcitrus.org.au).

Kaffir lime leaves extract was major in contain of chlorophyll which is green to black paste in physical state. Besides that, chlorophyll in kaffir lime leaves extract was insoluble in water but it is soluble in organic solvent including ether, benzene and white oil. In addition, chlorophyll in kaffir lime leaves extract was stable under ordinary conditions (Physical and Chemical Properties retrieved from http://chemicalland21.com). The kaffir lime leaves extract should be store at chiller but not freezer and room temperature to keep the chlorophyll fresh and not affect the result in this research.

2.4.3 *Introduction of Chlorophyll Reduction*

Natural reduction of graphene oxide to graphene using kaffir lime leaves which can be consider as the most easier and most cheaper process compare to using thermal reduction and chemical reduction. Preparation of chlorophyll extract is very simple which are using ethanol as the extraction chemical to extract. Reduction of graphene oxide to graphene can be done in room temperature by mixing the chlorophyll extract with the graphene oxide. After that, separate by using separation funnel. This method was investigated by Chloro Handout, 2002. Then the reduction method can be continued by adding in graphene oxide. The separation method of the natural reduction is magnetic separation by mixing with ideal Fe3O4 that prepare by Fe(III) and Fe(II) solution then separate it using magnetic field in room temperature. Graphene that reduce from graphene oxide are then bind with the ideal magnet can de separate by using ultrasonic after done the washing procedure. Then the pure graphene are being separated by another magnetic separation method to separate the ideal magnet. The work aims to prepare a cheaper and renewable separation method from ideal magnetic separation material that is easy recover apart from providing an very efficient conversion of kaffir lime leaves reduction (Zare et al, 1995).

2.4.4 Advantages Graphene Oxide Reduction Using Kaffir Lime Leaves Extract

Using kaffir lime leaves for reduction which contains high concentration of chlorophyll as reducing agent and it can be remove easily during separation process without the need of modification. The advantages of natural reduction are the material is ready availability and the potential for commercial use is high due to the economic price.

This open a new avenue of natural reduction of graphene oxide by using leaves from tree, which is kaffir lime leaves and it can be used in this work. Most of the kaffir lime leaves can be found easily in Malaysia, Thailand, Vietnam and Indonesia. It producing more than 800 tons of kaffir lime leaves annually and this project aims to utilise these leaves for the reduction of graphene oxide. The kaffir lime leaves have characteristic of aroma that identified as (–)-(S)-citronellal, which is present in the leaf oil over 80%, small components included citronellol (10%), nerol and lomonene. The small amount of citronellol can also be reducing agent that support normal chlorophyll reduction which is acidic compound as reducing agent to contribute in the reduction process of graphene oxide.

Natural reduction of graphene oxide to graphene using kaffir lime leaves which can be consider as the most easier and most cheaper process compare to using thermal reduction and chemical reduction. Preparation of chlorophyll extract is very simple which are using ethanol as the extraction chemical to extract.

2.5 Summary

This chapter was providing knowledge for the different type of reduction and the reduction mechanism. In details of graphene oxide reduction mechanism and the disadvantages of the previous graphene oxide reduction were stated in this chapter. The information of qualitative test in this research which are electrochemical analysis of the graphene by natural reduction was also included in this chapter. In addition, the method chosen for graphene oxide reduction included purpose of choosing graphene oxide reduction method using kaffir lime leaves extract, the physical and chemical properties of kaffir lime leaves extract, an briefly introduction of chlorophyll reduction and the advantages of choosing kaffir lime leaves extract for graphene oxide reduction.

3 METHODOLOGY

3.1 Chemical, Reagent and Instrument

Chemical were obtained mostly from Sigma Aldrich (potassium persulfate, phosphorus pentoxide, potassium permanganate, graphite powder and concentration sulphuric acid). Kaffir lime leaves was obtained from the kaffir lime tree planted on my own. Some of the common chemicals such as sodium chloride were obtained from UMP FKKSA chemical warehouse.

3.2 Synthesis of Graphene Oxide

A solution concentrated sulphuric acid (H_2SO_4), $K_2S_2O_8$ and P_2O_5 was prepared and heat at temperature up to 80°C. 20 grams of graphite powder was then added into the solution with continuous stirred about 30 minutes. The solution was then cool at room temperature for 6 hours. After that, deionize water (DI water) was added to filter using vacuum filter and wash the filtrate until it becomes neutral pH. Then the filtrate was dried overnight at room temperature inside vacuum desiccator. The next day, 20 grams of dried graphite powder was poured into a solution of 0°C concentrated H_2SO_4 by using a 2 litre conical flask. 60 grams of $KMnO_4$ was then added slowly into the solution with stirring and maintained at temperature below 20°C. Then, the mixture was heated up to 35°C for 2 hours using oil bath. Next, 920 mL of DI water was added into the mixture. The temperature was maintained at 98°C for 15 minutes. Another 2.8 litre of DI water and 30% H_2O_2 was then added into the mixture. The mixture was then filter using vacuum filter with 5 litre of 1:10 concentrated HCl. The cake was dried overnight at room temperature using a vacuum desiccator.

3.3 Natural Reduction of Graphene Oxide to Graphene

3.3.1 Preparation of conventional natural reduction material

Preparation of kaffir lime extract was using kaffir lime leaves and blended with water by commercial blender. After that, the blended solution was filtered using vacuum pump. The kaffir lime leaves extract was then store in refrigerator at temperature 3-8°C. The yield of the kaffir lime leaves was measured by UV-vis Spectrometer.

3.3.2 Reduction of graphene oxide to produce conventional graphene

The reduction was done by dispersing small amount of GO powder from GO synthesis into DI water. Next, the mixture was ultrasonicated for 30 minute to remove unexfoliated graphite oxide. After that, the solution was then centrifuged under 500rpm for 10 minutes to remove the impurity materials. The supernatant was located at the top layer was the graphene oxide. Then the supernatant graphene oxide was poured into a beaker and the pH was adjusted for the solution using 5M of KOH. For the next step, the kaffir lime leaves extract was place under UV light supply and stirred. Then final product of reduction is the mixture of graphene and the kaffir lime leaves extract which need vacuum separation process to separate the graphene from solution.

3.4 Separation of Graphene from Solution

3.4.1 Separation of the Graphene from mixture solution

Separation process was done by vacuum filtration. The mixture solution was pour to the vacuum filter to separate the chlorophyll and the graphene powder. The graphene was then wash with 2 litre of DI water.

3.5 Optimization of Reduction Process

3.5.1 Optimization of Retention Time and Concentration

In this experiment, the effects of the kaffir lime leaves extract concentration on reduction process were studied with time constant and also monitored with the kaffir lime leaves extract concentration constant. This is important in order to know the optimal amount of reducing agent required with minimal time for the reduction completion. Several parameters were set for the optimizing process shown in Table 3.1.

Table 3.1: Optimization Parameters of Different Reductant Concentration and Reduction Time

Reduction at Different Concentration of Kaffir Lime Leaves Extract (g/L)	5
	10
	15
	20
	25
Retention Time for Completed Reduction for each Different Reductant Concentration (hour)	0
	0.5
	1.0
	1.5
	2.0
	2.5
	3.0
	3.5
	4.0
	4.5

3.6 Identify Reduction Rate by UV-Vis Absorption Spectroscopy

Ultraviolet–visible spectroscopy (UV-Vis) and ultraviolet-visible spectrophotometry (UV/Vis) was refers to the absorption spectroscopy and reflectance spectroscopy respectively in the ultraviolet-visible spectral region. The visible range of the absorption and reflectance will affects the perceived colour of the chemicals that involved. Based on qualitatively applications, ultraviolet radiation was useful to detect chromopholic groups in the sample. For this experiment, UV-vis Spectroscopy was used as graphene oxide and give brownish colour while reduced graphene oxide (Graphene) will give black colour. However, this technique must be supplemented with other physical or chemical evidence. This is due to this spectrometer just give the idea to identify the absorbing groups by comparing the spectrum of an analyte with those of simple molecules containing different chromophoric groups.

In this experiment, the reduction rate was measured by using UV-vis absorption spectrometer through the process of graphene oxide reduction. Theoretically, graphene oxide peak was located at 231nm but gradually red shift to nearly 270nm as deoxygenation take place and electronic conjugation is restored. Powder form of graphene oxide and reduced graphene oxide was dispersed in deionized water with water bath sonicator for 30 minutes until homogenized solution was form then characterized by using Ultra Violet visible spectroscopy (UV-Vis) the wave length of 200-350nm.

3.7 Qualitative Measurements of Reduce Graphene

Electrochemical studies are needed to test the biofuntionalization of carbon nanosheet surface. In this work, the functionalized carbon nanosheet must go through the surface characterization procedure using Scanning Electron Microscopy (SEM). This measurement was done by Central Lab of University Malaysia Pahang.

4 RESULT

4.1 Overview

This chapter only was discussed the result of the research experiment which is synthesis of graphene oxide from graphite powder using commercial method, reduction of graphene oxide to graphene using natural reduction, optimization of reduction process with parameter of reduction time and concentration of reductant to achieved completed reduction and the shape of the graphene after natural reductions.

4.2 Result and Discussion

4.2.1 Pre-oxidation of Graphite during Synthesis of Graphene

Due to first step to synthesis graphene oxide when a solution of sulphuric acid, phosphorus pentoxide and potassium persulfate was mixed with the graphite powder, graphite was not change in dark blue colour as the propose method. These shows that the pre-oxidise graphite powder was not affect the quality of graphite oxide as a step to speed up the oxidation of graphite.

4.2.2 Synthesis of Graphene Oxide from Graphite Powder

After pre-oxidation of graphite powder, the solution was then cool at room temperature and filter using vacuum filter with washing deionize water until it becomes neutral pH then dried overnight inside vacuum desiccator. When concentrated H_2SO_4 and $KMnO_4$ was added slowly into the solution and heated up to 35°C, brownish grey paste was appeared. This proved that the oxidation of graphite to graphite oxide was done. After 30% H_2O_2 was added into the mixture, the colour of the mixture was change to bright yellow and this shows that the mixture was completely synthesis to graphene oxide from graphite oxide.

4.2.3 Reduction of Graphene Oxide to Graphene

During the reduction process, the mixture of graphene oxide and the kaffir lime leaves extract was place under UV light and stir. The solution was remained green