

UNIVERSITI TEKNOLOGI MARA

**PARAMETER OPTIMIZATION OF CHEMICAL
REDUCTION OF GRAPHENE OXIDE USING DESIGN
EXPERT®**

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ABSTRACT

Graphene or also known as reduced graphene oxide exhibit variety of fascinating properties. Due to many possibilities to be utilized in wide field of application, the researches in fabrication had been made. Chemical reduction of graphene oxide is one of the methods known in synthesizing graphene. Graphene oxide was synthesized by the oxidation of graphite by following modified Hummer's method. In the reduction of graphene oxide, L-ascorbic was used as the reductant followed with addition of ammonia. This research was done optimize the reduction parameters (concentration of ascorbic acid, temperature and time of reduction reaction) to maximize the properties of reduced graphene oxide (rGO) using central composite design (CCD) from Design Expert® and to investigate the effect of chemical reduction of graphene oxide (GO) parameters towards the final properties of reduced graphene oxide (rGO) in term of phase composition, crystallinity, particle size and conductivity. The properties of rGO was characterized by undergoing different analysis which are crystalline structure analysis by XRD diffractometer, zeta potential and particle size by Nano-ZS Zetasizer and finally the conductivity analysis by calculation of electrical conductivity of rGO. The optimum conditions of chemical reduction of GO are 17.612 mg/mL for the concentration of ascorbic acid, duration of 197.817 minutes at temperature 95 °C which predicted to result zeta potential of -16.70 mV in deionized water, has conductivity of 2.2×10^{-2} S/m, particle size of 121.63 nm with interplanar spacing of 0.36 nm.

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CHAPTER ONE

INTRODUCTION

1.1 Research Background

Graphene or reduced graphene oxide (rGO) is a single-layer of carbon atom with sp² hybridization tightly bonded with hexagonal-like structure. Graphene is known to be the lightest and thinnest substances known to man (Brownson & Banks, 2014). According to Geim, (2014), many researches were done to synthesis this allotrope of carbon because of its variety of fascinating properties. According to Kim, et al. (2016), graphene exhibit transcendent thermal, mechanical and electrical properties as well as optical properties as it has transparency about 98% which is almost transparent. Due to its captivating properties, graphene has high potential to be applied in wide range of fields. According to the research made, graphene is known to be the best electrical conductor (high conductivity $\sim 10^4 \Omega^{-1} \text{ cm}^{-1}$) and the best heat conductor (thermal conductivity = $5000 \text{ W m}^{-1} \text{ K}^{-1}$) at ambient temperature and thus has high potential to be used as supercapacitors, composite compounds, sensors, electrodes and many other fields of applications (Khosroshahi, et al. 2018).

Geim, et al. firstly produced Graphene accidentally using a scotch tape in 2004 when Geim and Novoselov are trying to clean the graphite with scotch tape. Ever since graphene was introduced for its existence to the world, there are many methods has been used to synthesize graphene from graphite. Some of the methods known are mechanical exfoliation of graphite, epitaxial growth, chemical vapor deposition and finally, chemical reduction of graphene oxide (GO). Different methods will produce different properties of graphene. From all the methods mentioned, Hou, et al. (2017) state that chemical reduction of graphene oxide (GO) is considered as advantageous since this methods known to be the fastest way to fabricate graphene as well as its cost effectiveness and bulk-scale productivity. This method involve two steps where graphene oxide is first produced before it chemically reduced to graphene. The first step involved the oxidation of graphite powder to form graphite oxide and continued with the second step where the graphite oxide is exfoliated to graphene oxide by ultrasonication. Several approach was done to prepare the graphene oxide such as Brodie method, Staudenmaier method,