THE STRUCTURAL PROPERTIES OF GRAPHENE OXIDE AND REDUCED GRAPHENE OXIDE GROWN ON STAINLESS STEEL SUBSTRATE

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ABSTRACT

In this paper, the structural properties of graphene oxide (GO) and reduced graphene oxide (rGO) based on custom-made, sodium 1,4-bis (neopentyloxy)-3-(neopentyloxycarbonyl)-1,4-dioxobutane-2-sulphonate (TC14) surfactant were investigated. GO was synthesized by electrochemical exfoliation and chemical reduction process utilizing hydrazine hydrate was further done to produce rGO. The obtained GO and rGO solution were then transferred to stainless steel substrate by spraying deposition method. Based on several characterizations, the fabricated film has potential to be used as electrode for water treatment application.

Key words: Graphene Oxide, Reduced Graphene Oxide, Stainless Steel, Electrochemical, Spraying.

ABSTRAK

Dalam artikel ini diteliti sifat struktural dari oksida grafin (GO) dan oksida grafin yang direduksi (rGO) menggunakan surfaktan sodium 1,4-bis (neopentyloxy)-3-(neopentyloxycarbonyl)-1,4-dioxobutane-2-sulphonate (TC14) yang dibuat khusus. GO disintesis menggunakan eksfoliasi elektrokimia dan proses reduksi kimiawi menggunakan hidrazin hidrat untuk menghasilkan rGO. Larutan GO dan rGO yang dihasilkan kemudian ditransfer ke substrat *stainless steel* degan metode deposisi penyemprotan. Berdasarkan beberapa karakterisasi, film yang difabrikasi memiliki potensi untuk digunakan sebagai elektroda untuk aplikasi pengolahan air.

Kata kunci: Oksida grafin, oksida grafin direduksi, *stainless steel*, elektrokimia, penyemprotan

INTRODUCTION

Graphene has attracted many attentions since its invention in 2004 due to its good mechanical, optical (high transparency), and electrical properties (high conductivity) [Zhang, *et. al.*, 2017; Metke, *et. al.*, 2016; Cooper, *et. al.*, 2013; Hayes, *et. al.*, 2014; Marinho, *et. al.*, 2012]. Several applications which used graphene are energy storage and conversion [Brownson, *et. al.*, 2011; Fan, *et. al.*, 2015; Liao, *et. al.*, 2010], communication [Mueller, *et. al.*, 2010], solar cells [Li, *et. al.*, 2015], and capacitive deionization in the water treatment [Shi, *et. al.*, 2016]. Graphene oxide (GO) as one of graphene derivation also interesting to be developed due to the easier transfer process to the desired substrate. The synthesis of GO can be done by electrochemical exfoliation (EE) utilizing surfactant to assist the GO dispersion [Suriani, *et. al.*, 2016]. The EE is known as simple, green, and low-cost method as compared to Hummers method in the GO synthesis. The oxygen functional groups, including epoxy, hydroxyl, and carboxyl groups resulted during oxidation process makes the thick layer of synthesized GO [Pei and Cheng, 2011]. Chemical reduction process was further done in order to remove the oxygen functional groups and produce reduced GO (rGO). Hydrazine hydrate was chosen as reducing agent due to its effectiveness to produce thin rGO. The GO and rGO can also be utilized as metal protection from oxidation and corrosion [Raman and Tiwari, 2014].

There are many transfer process method to deposit GO and rGO solution to desired substrate, such as dip coating, spin coating, layer by layer self-assembling, direct apply and curing, electrophoretic deposition, and spray coating. Spray coating method have been widely used to fabricate thin film and applied for industrial coating [Tong, *et. al.*, 2013]. In this work, the GO synthesis was done by EE method utilizing custom-made surfactant, sodium 1,4-bis (neopentyloxy)-3-(neopentyloxycarbonyl)-1,4-dioxobutane-2-sulphonate, known as TC14 in the deionize (DI) water as a solvent. The GO and rGO solution was then transferred to stainless steel (SS) substrate using spraying deposition method. The utilization of SS as substrate was due to its properties which were thermally and chemically stable as compared to copper and nickel.

The fabricated GO and rGO film on SS substrate were characterized using field emission scanning electron microscopy (FESEM) and micro-Raman spectroscopy in order to investigate the morphology and crystallinity of the samples, respectively. Based on the characterizations, the fabricated films were potential to be applied as electrode for water treatment application and avoid the corrosion of SS substrate.

MATERIALS AND METHODS

The 0.1 M of electrolyte solution was prepared by dissolving TC14 surfactant into DI water. Two graphite rods were immersed into the prepared electrolyte and connected to 7V. The oxidation and exfoliation process was done for 24 hours and yielded GO solution. The amount of 0.5 ml of hydrazine hydrate was dropped to the obtained GO solution then maintained at ~95℃ under constant stirring to produce rGO. The GO and rGO solution were then sprayed onto the cleaned SS substrate with the dimension of 2×2 cm^2. Prior to spraying, the SS substrate was preheated at 120℃ for 5 minutes. The sample was then annealed in the furnace at 400 ℃ for 1 hour under argon ambient.

RESULTS AND DISCUSSIONS

The morphology of the fabricated film was characterized by FESEM and the results are presented in Fig. 1. Based on Fig. 1 (a) and (b), it is clearly seen that the stacked and agglomerated GO was spread almost in the entire SS substrate. The agglomerated GO was caused by the oxygen functional groups that exist in the sample which makes the film looks thick. After the reduction process, the stacked and agglomerated GO becomes thinner and forms layer sheets. The thin layer of rGO was believed due to the effectiveness of hydrazine hydrate in the oxygen functional groups removal during reduction process. The fold up layer on the edge plane of the film shows that the oxidation process was successfully performed during EE. The rough and porous morphology of GO and rGO sheets were believed to give an advantage for better adsorption process in the water treatment application. The heavy metal ions on the polluted water can be trapped in those structure thus can help the water cleaning process. In addition, the covered GO and rGO film on the SS substrate gives longer term *corrosion protection* to the SS *substrate* which is beneficial for water treatment application.

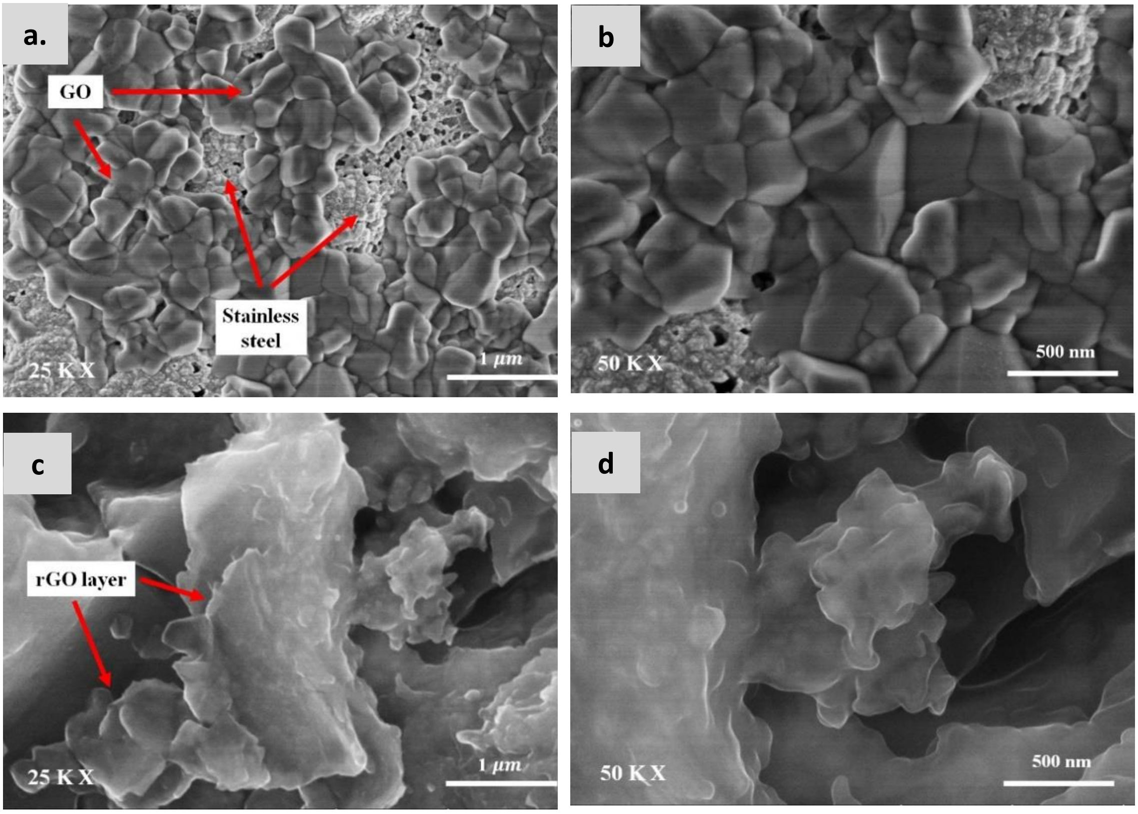

Fig. 1. FESEM images of; (a)-(b) GO and (c)-(d) rGO film grown on stainless steel substrate.

Micro-Raman spectra of GO and rGO films are presented in Fig. 2. Based on the spectra, the rGO film exhibits three peaks in the range of 500-3000 cm^{-1}. The D-band which indicates the disorder and defect level was observed at 1359 cm^{-1}. The high intensity of D-band (I_D) on rGO sample suggests the successful oxidation process during EE. This was also confirmed by the shifted G-band at 1584 cm^{-1} from graphite peak (1581.72 cm^{-1}). The crystallinity of the sample can be determined by I_D/I_G value, and its value was found to be 0.94 for rGO sample. The high value of I_D/I_G suggests that the fabricated film has a good crystallinity. The rGO sample also present the 2D-band at 2707 cm^{-1}. The sharp and high intensity of 2D-band attributes to the thin and few layers of rGO film which was believed due to the reduction process.

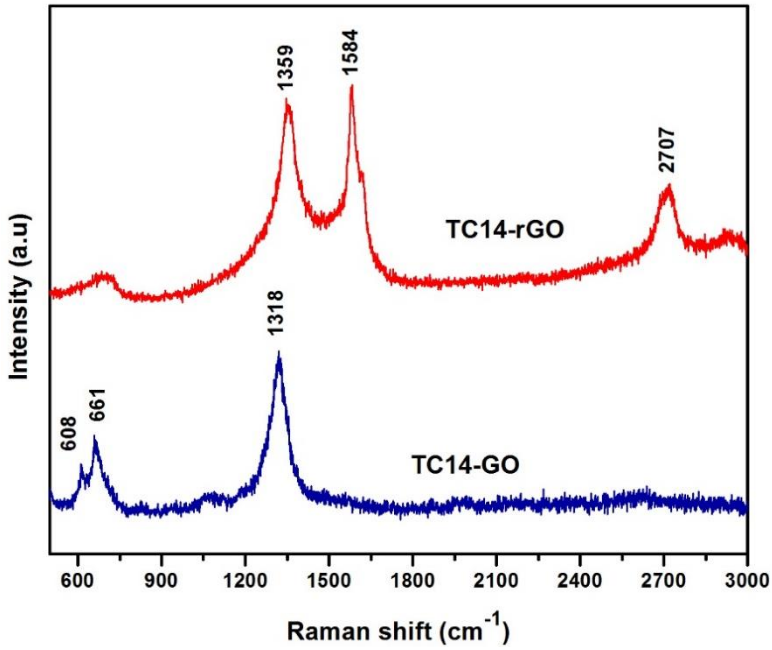

Fig. 2. Micro-Raman spectra of GO and rGO film grown on stainless steel substrate.

CONCLUSION

In this work, we have successfully fabricated GO and rGO film based on GO and rGO solution produced by electrochemical exfoliation and chemical reduction process, respectively on the stainless steel substrate. Good crystallinity and morphology of the fabricated films gives a potential possibility to be applied as electrode for water treatment application. In addition, GO and rGO have a play role in the anti-corrosion for SS substrate.

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