rGO based photo-anode in dye-sensitized solar cells (DSSC) and its photovoltaic characteristics

Waode Sukmawati Arsyad¹, Aricia Pristianti¹, La Agusu¹, and Rahmat Hidayat²

¹ Physics Department, Faculty of Mathematics and Natural Sciences, Halu Oleo University, Jl. Prof. HEA. Mokodompit No. 1 Kendari 93231, South East Sulawesi, Indonesia
² Physics of Magnetism and Photonics, Physics Department, Faculty of Mathematics and Natural Sciences, Bandung Institute of Technology, Jl. Ganesha 10 Bandung 40132, West Java, Indonesia

*Corresponding author: wdsukmawati@gmail.com

Abstract. On the attempt to increase transport properties of the photo-anode in DSSC, we synthesized rGO powder from graphite bar (commercially available) using modified Hummer’s method. The SEM-EDS results had confirmed the attachment of the rGO layer to the FTO substrate. For DSSC cells, we made two cells configuration, the first one was stacking layers of rGO and TiO₂ resulting configuration of TiO₂/rGO/TiO₂ (A2) and rGO/TiO₂/rGO (A3), where rGO was deposited by spin coating and TiO₂ was deposited by a screen printing technique. The second one, the rGO powder was mixed with TiO₂ paste with several ratios in weight, namely TiO₂:rGO 40:1 (B1), 40:2 (B2), and 40:8 (B3) and then deposited on the FTO substrate by screen printing. The reference cell was assigned as A1 (TiO₂ only). From the conductivity measurement using the four-probe method, the utilization of rGO layer increased the conductivity of photo-anode layer, namely (1.37, 2.9 and 6.3)x10⁻² Ω⁻¹ cm⁻¹ for A1 to A3 and (1.5, 2.5, and 3.7)x10⁻² Ω⁻¹ cm⁻¹ for B1 to B3. From the photovoltaic measurement, we found that the efficiency of the DSSC cell firstly increased with the insertion of rGO layer, from 1.8% (A1) to 4.59% (A2), and decreased to 3.22 %, as the conductivity increased in A3. While for the composite of TiO₂:rGO, the efficiency of the cell reduced with the increased amount of rGO, from 3.45% (B1) to 2.9% and 1.9% for B2 and B3. We found that the reduction of photovoltaic performance was affected by two main factors, specifically, direct contact between rGO and redox species in the electrolyte, which induced recombination process, and conductivity of the photo-anode layer. To fully achieve the advantage of rGO utilization in photo-anode, once must be considered was the use of protection layer on top of the rGO layer to avoid direct contact between the rGO/electrolyte interface.

Keywords: rGO, DSSC, photo-anode, conductivity, photovoltaic, efficiency
1. Introduction

Since the first time reported [1] until now, there have been numerous efforts to improve the performance of the dye-sensitized solar cell (DSSC) through optimization of all its constituent components [2, 3, 4, 5, 6]. Among other components such as a dye, redox electrolyte and counter electrode whose only has one function, photo-anode serve a dual function in DSSC, specifically as the dye adsorption site and medium for charge transfer and transport. Photo-anode of a DSSC consist of a dye-sensitized semiconducting layer on top of a conducting substrate. To be effective, photo-anode must fulfill some characteristics specifically, high surface area for dye loading, high transparency, high electron mobility, low recombination rate, and contains a hydroxyl group or defect states for dye attachment [7, 8, 9]. FTO is the most frequently used conducting substrate owing to its resistance that does not significantly change, even after heating up to 700°C [10], while TiO$_2$ is the most utilized n-type metal oxide semiconductor in DSSC, due to its high chemical stability [11, 12], high dielectric constant and fewer defect states, which leads to less recombination [13, 14]. Despite TiO$_2$ nano-porous layer ability to provide large surface area for dye loading and light absorption [7, 15], but it also acts as the source of recombination site at TiO$_2$/electrolyte and FTO/electrolyte interfaces [7, 16]. The presence of the recombination process will induce photocurrent reduction and eventually affect the overall conversion efficiency of the DSSC cells. The major challenge for the DSSC performance improvement is to improve the charge transfer and transport in the TiO$_2$ layer since efficient charge transfer and transport will be able to compete with the recombination process. One way to accomplish efficient charge transfer and transport are by introducing charge carriers materials into photo-anode. This can be done through doping of semiconducting layer with Mg [17], Sb [18], Zr [18], N [19], S [20] and Cr [21], or through modification of photo-anode configuration, with the utilization of blocking layer [5, 22], scattering layer [23, 24] or incorporation efficient charge transport material such as carbon nanotube [25] and graphene [26, 27, 28].

Graphene has gained many attentions due to its excellent properties including optical transparency, high stability, low cost, and non-toxicity. Moreover, its electrical conductivities in two dimension can reach more than 2x10$^5$ cm$^2$V$^{-1}$s$^{-1}$ [26, 29], and can easily anchor with TiO$_2$ through intermolecular forces [30] and thus provide a fast channel for electron transportation [31]. Graphene or its partly reduce part forming graphene-like sheets or reduced Graphene Oxide (rGO) can be used both in photo-anode and counter electrode part of DSSC. In photo-anode, rGO is used as blocking layer (BL) [32], mixed with TiO$_2$ particles to form composites or scaffold layer [33, 34] and stacked layer by layer with TiO$_2$ [35]. Although all the previously reported studies on the incorporation of graphene particularly rGO in DSSC stated that there are photovoltaic (PV) performance improvement with the utilization of rGO, some studies also reported the reduction of PV performance as the escalation amount of rGO content in the photo-anode [36]. The PV performance reduction, in this case, was attributed to the low light harvesting by the dye molecules due to the absorbing properties of rGO itself.

Herein, we investigated the possibility that there may be another mechanism responsible for PV performance reduction in DSSC utilized rGO. For that purpose, we fabricated two different structure of photo-anode. The first one is by stacking rGO and TiO$_2$ layer-by-layer forming configuration of TiO$_2$/rGO/TiO$_2$ and rGO/TiO$_2$/rGO, and the second is by mixing rGO and TiO$_2$ with different weight ratio to form composite or scaffold layer deposited on top FTO conductive substrate, where the photovoltaic parameters were analyzed from the current-voltage (I-V) measurement.
2. Experimental

2.1. Reduced Graphene oxide (rGO) preparation

2.1.1. Graphene oxide (GO) preparation. GO preparation was following the same method that already reported elsewhere based on modified hummers method re-described in [37, 38]. The graphite bar was crashed until smooth and strained using 250 mesh filter to form homogeneous graphite powder. 2 gr of graphite powder was mixed 98 ml of 98% Sulphuric Acid (H₂SO₄) and vigorously stirred for four hours in the ice bath environment at 0°C temperature. During the stirring process, 8 gr of Potassium Permangenate (KMnO₄) and 4 gr of Sodium Nitrate (NaNO₃) was added very slowly into the mixture until the color of the solution turned into blackish green. The temperature then slowly raised to 35°C for 24 hours. The color of the suspension slowly turned to light brown, and the suspension became more viscous. 200 ml of distilled water slowly added into the suspension while stirred for one hour, where the color changed into dark brown and became golden yellow with the addition of 15 ml of Hydrogen Peroxide (H₂O₂). The suspension was then precipitated for 24 hours to separate the liquid phase and solid phase. The solid phase was washed with 10 ml Hydrochloric Acid (HCl) 37% and distilled water several times until the pH neutralized, dried in the oven at 110°C for 12 hours. 40 mg of the resulting graphite oxide was then diluted into 40 ml of distilled water, stirred overnight and sonicated with the frequency of 53 KHz for 120 minutes.

2.1.2. Reduction of Graphene Oxide using Zinc particles and thermal process. 10 ml HCl 37% added into the pre-prepared GO solution, to form an acidic environment. 1.6 gr of Zinc powder then added into the GO solution and let for 16 hours. The mixture then stirred for one hour and added 10 ml HCl 37% again to remove the Zn particles that did not react in the solution, washed with distilled water until the pH of the solution was neutralized and dried in the oven at 110°C for 12 hours. The resulted rGO powder then subsequently dried in the microwave with 800 Watt power for two minutes with five seconds delay at every 30 seconds. The utilization of microwave in the drying process to produce homogeneous heat in relative very short time for speeding up the reduction time [38].

2.2. DSSC preparation

The materials and preparation methods for fabricating DSSCs here were basically the same as reported in [1], where all materials were used as received. The complete DSSC samples were made by the same fabrication method as reported in our previous work [39, 40] for preparing the A1 reference cell. First, the conductive substrate (FTO, surface resistivity = 15Ω/ per square) was cleaned with teepol for removing dirt, rinsed with ethanol (technical) and acetone (technical), and ultra-sonicated for 15 minutes in the mixture 1:1 (in volume) of Ethanol (Merck) and Isopropanol (Merck), and dried with air gun. One layer of transparent TiO₂ paste (18-nrt from Dye-Sol) was deposed by screen-printer (0.5 cm x 0.5 cm) followed by the heating process on the hot-plate for five minutes in 80°C. The procedures were repeated two times for resulting thicker transparent TiO₂ layer. One layer of reflector TiO₂ paste (Wer-02 from Dye-Sol) was deposited using the same size of screen printer on top of former TiO₂ layers, followed with heating process on the hot-plate where the temperature gradually increased from 80°C with the step of 20°C / 6 minutes until it reached the final temperature of 500°C, where it was maintained for 30 minutes. The resulting TiO₂ electrode then immersed in 0.7mM of cis-bis(isothio-cyanato)bis(2,2'-bipyridyl-4,4'-dicarboxylato) ruthenium(II)-bis-tetrabutylammonium (N719 dye, Solaronix) for 24 hours. The photo-anode and the counter electrode (1mm drilled FTO layered with Pt from Solaronix) were assembled and separated by a separator (Surlyn® of 50 µm thickness), the electrolyte solution (Mosalyte TDE-250 from solaronix) then injected through the hole of the counter electrode using a vacuum pump.

In this research, we made two cells configuration. In the first one, the rGO was made layer by layer with the TiO₂ layer resulting configuration of rGO/TiO₂/rGO (A2) and TiO₂/rGO/TiO₂ (A3). The rGO solution was dropped on top of FTO substrate, and let for five seconds before spin coated at 2000 rpm
for 30 seconds, and subsequently heated on the hot plate at 250°C for two minutes. TiO$_2$ layer was deposited by a screen printing technique. In the second one, the rGO powder was mixed with TiO$_2$ paste with several ratios in weight, specifically TiO$_2$:rGO 40:1 (B1), 40:2 (B2), and 40:8 (B3) and then deposited on the FTO substrate by screen printing, following by annealing. In all samples, the procedures for preparing TiO$_2$ layers following the same steps for preparing the reference cell (A1).

2.3. Characterization
The resistance of the rGO layer was measured by using Four Probe Methode (FPM) at the bias voltage of 9 Volt. Morphology of rGO and stacking layer of rGO-TiO$_2$ was scanned using SEM. The fabricated DSSCs were characterized by the current density-voltage (J–V) measurements using a solar simulator (Newport, Model no. 11000) with the average light power density of 100mW/cm$^2$.

3. Result and Discussions
3.1. Morphology of rGO and stacked layer of rGO-TiO$_2$
Fig. 1 shows the SEM results of one layer rGO, TiO$_2$/rGO/ TiO$_2$, and rGO/TiO$_2$/rGO. Fig. 1(a) is the rGO layer that shows morphology like a stack of corn flakes. The distribution of the rGO flake is not homogeneously covered the substrate, which may cause a drawback when functioning as the blocking layer. Fig.1(b) shows the morphology of a stacked layer of TiO$_2$/rGO/ TiO$_2$, where TiO$_2$ was screen printed and the rGO was spin coated. The rGO is not evenly distributed on top of the TiO$_2$ layer and it seems that the spin coating process induces agglomeration of rGO particle forming bigger particles.

![Figure 1. SEM results of one layer rGO (a), (b) TiO$_2$/rGO/TiO$_2$, (c) surface of rGO/TiO$_2$/rGO, cross section of rGO/TiO$_2$/rGO (d), enhanced version of cross section of rGO/TiO$_2$/rGO using ImageJ (e), and (f) thickness and width estimation of rGO flake.](image)

Fig.1(c) shows the surface morphology of the rGO/TiO$_2$/rGO layer, the upper rGO layer agglomerates to form islands with a different size that scatter on top of the substrate. Fig.1(d) is the cross-section of the rGO/TiO$_2$/rGO layer. The interface between each layer can be seen, where the structure does form layer by layer, that was confirmed through the images contrast enhancement using ImageJ software in Fig.1(e). The SEM results confirmed that the size of the rGO particles that yielded is still too big, which may cause uneven surface, resulting in poor contact between the interfaces.
The thickness and width estimation of rGO flake is shown in Fig.1(f), where the thickness varies from (0.67 – 2.62) µm, while the widest rGO flake is 12.68 µm. This result is in good agreement with another study [41] who has also produced two-dimensional wrinkle sheets with the size of several micrometers. Fig.2 confirm the existence of rGO on the substrate through the EDS spectrum, with 60.98% of C atoms.

![EDS result for one layer rGO on top of a glass substrate](image1.png)

**Figure 2.** EDS result for one layer rGO on top of a glass substrate

### 3.2. Conductivity of rGO and rGO-TiO$_2$ composites layer

The conductivity of all the samples that were calculated from the V-I measurement using FPM is tabulated in Table 1. Overall, the utilization of rGO increases the conductivity of all samples compared to the reference cell (A1). The increasing of the conductivity in consecutive order are 111.6%, 359.8%, 9.5%, 82.5%, and 170.0% for A2, A3, B1, B2, and B3 compared to A1. For layer by layer structure (A2 and A3), A3 with TiO$_2$ in between two rGO layer shows 53.9% higher conductivity compared to A2 where rGO position is in between two TiO$_2$ layer. While for the composite of TiO$_2$-rGO (B1, B2, and B3), the conductivity increase with the amount of rGO added.

**Table 1.** Conductivity calculation of TiO$_2$, stacked layer of TiO$_2$ and rGO and composite of TiO$_2$ and rGO, measured with FPM at 9 Volt bias voltage.

| Cell  | V (Volt) | I ($10^{-4}$ A) | Thickness ($\times 10^{-2}$ cm) | $\rho$ ($\Omega \cdot \text{cm}$) | $\sigma$ ($\times 10^{-2} \Omega^{-1} \cdot \text{cm}^{-2}$) |
|-------|----------|----------------|-------------------------------|-------------------------------|--------------------------------------------------|
| A1    | 0.35     | 0.5            | 2.3                           | 73.1                          | 1.37                                             |
| A2    | 0.36     | 1.1            | 2.3                           | 34.14                         | 2.9                                              |
| A3    | 0.35     | 2.3            | 2.3                           | 15.87                         | 6.3                                              |
| B1    | 0.37     | 0.6            | 2.3                           | 64.66                         | 1.5                                              |
| B2    | 0.38     | 1.0            | 2.3                           | 39.61                         | 2.5                                              |
| B3    | 0.38     | 1.5            | 2.3                           | 25.33                         | 3.7                                              |

**Table 2.** Photovoltaic parameters of DSSC incorporated rGO in the photo-anode

| Cell  | Configuration / composition | $V_{OC}$ (Volt) | $J_{SC}$ (mA/cm$^2$) | FF (%) | $\eta$ (%) |
|-------|-----------------------------|-----------------|----------------------|--------|------------|
| A1    | TiO$_2$ only (ref. cell)    | 0.67            | 4.62                 | 59.0   | 1.80       |
| A2    | TiO$_2$-rGO/TiO$_2$         | 0.70            | 12.2                 | 53.7   | 4.59       |
| A3    | rGO-TiO$_2$-rGO             | 0.70            | 10.0                 | 48.7   | 3.22       |
| B1    | TiO$_2$:rGO 40:1           | 0.68            | 10.0                 | 51.0   | 3.45       |
| B2    | TiO$_2$:rGO 40:2           | 0.70            | 7.68                 | 55.0   | 2.90       |
| B3    | TiO$_2$:rGO 40:8           | 0.76            | 4.45                 | 56.0   | 1.90       |
3.3. DSSCs photovoltaic properties

Fig.3 and Fig.4 show the influence of the exertion of rGO in photo-anode to the photovoltaic properties of the resulted DSSC cells, and the parameters including photocurrent ($J_{sc}$), open circuit voltage ($V_{oc}$), fill factor (FF) and overall conversion efficiency ($\eta$), tabulated in Table 2. The performance of the A1, A2 and A3 cells is depicted by its J-V curves under A.M 1.5 condition (Fig.3). As can be seen that rGO utilization has a pronounced impact on the photocurrent value, in which A2 configuration resulted in 164% photocurrent enhancement over the A1. This may be explained by the fact that graphene can act as an electron bridge in the photo-anode, shuttling electrons to the current collector and lowering recombination in the device [42]. Incorporation graphene materials in photo-anode also increase dye absorption capability [27, 32, 43], increased the light harvesting and eventually the photocurrent.

In the A3 configuration, where TiO$_2$ is in between two rGO layer, the photocurrent decrease by 17.6%, although it has higher conductivity compared to A2. The decrease in the photocurrent may be due to the cell configuration, where the first rGO layer is in direct contact with the FTO and therefore act as a blocking layer (BL), providing a barrier between triiodide and FTO conductive substrate, but it would also provide an insulating barrier [7]. This insulating barrier will potentially retard electron diffusion from TiO$_2$ to FTO or lengthen electron path distance and opening up possibilities for recombination process to take place. Therefore, the rGO layer must be very thin so that the tunneling effect can be effective. Besides that, BL can absorb some light as much as 1.6% [7], that will reduce the light absorbed by the dye, which is in agreement with the result from Chen, et al [36], that reported the light utility of dyes to decrease by thick graphene coating on FTO. The existence of the second rGO layer, which is in direct contact with redox couple (I$_3^-$/I$^-$/I$^-$/I$_3^-$) also may contribute to the decrease in photocurrent. Graphene has excellent electrical conductivity, so there will be a large number of recombination between the interface of graphene and electrolyte that will reduce the photocurrent. Moreover, for a metallic or semi-metallic surface, the proximity of an ion induces a further effect associated with polarization of the surface that strongly affects the interfacial attraction/repulsion of the ions [44]. Graphene has an abundance of the aromatic ring with delocalized π-electrons [45], and from the computational simulation from Shi et.al. [46] revealing that there are strong anion-π interactions on the graphene flake that associated with effective donor-acceptor interactions between the halide anions and the graphene flake. This possible interaction between graphene and iodide ions in the electrolyte will reduce the number of iodide ions that supposed to regenerate the dye, and hence will diminish the amount of dye that can capture the incoming light, and eventually reduce the photocurrent (Fig. 3, Table 2).
In accordance with photocurrent value, the $V_{oc}$ of the DSSC incorporated rGO layer also shows a 4.47% improvement for A2 and A3 compared to the reference cell (A1). The $V_{oc}$ value strongly determines by the Fermi level between the TiO$_2$ conduction band edge and the potential energy of the redox couple. As the conductivity of A2 and A3 are higher than A1 (Table 1), the Fermi level then shifts upwards, inducing to an increase in $V_{oc}$ (Fig. 2, Table 1).

J-V curves for B1, B2, B3, and A1 as the reference cell and the tabulated parameters were shown in Fig. 3 and Table 2. The photocurrent shows an improvement with the incorporation of rGO in the TiO$_2$ composite or scaffold layer as much as 117.3% compared to the reference cell, due to increasing of light absorption at the N717 dye’s absorption peak [34]. Further addition of rGO into the composite reducing the photocurrent by 23.5% and 55.5% for B2 and B3 respectively, compared to B1. The photocurrent reduction with the increasing content of rGO is inversely proportional with the conductivity value of the thin film (Table 1). This result indicated that there is a limit for graphene content in the composite of TiO$_2$-rGO to produce a maximum photocurrent. At the right amount, the inclusion of rGO could induce the formation of macropores due to the formation of voids formed from the further reduction process in the volume of graphene oxide during annealing process of the photo-anode layer which acted as the scattering layer [8, 31], and opened up a channel for electrolyte infiltration.

The $V_{oc}$ of the DSSC with a composite of TiO$_2$-rGO photo-anode also shows an improvement of 1.49% compared to A1. The increasing amount of rGO in the composite give rise the $V_{oc}$ by 2.94% and 11.76% for B2 and B3 respectively, compared to B1. The incorporation of rGO has the distinct effect to the photocurrent value for both photo-anode configuration (stacked layer and composite), and only slightly changed the $V_{oc}$ value (Fig. 2, Fig. 3 and Table 2).

4. Conclusion
The improvements in PV performance in DSSC with the utilization of rGO in photo-anode has been verified. Furthermore, increasing rGO content in the photo-anode will increase its conductivity, however as the conductivity increases the overall conversion efficiency of the DSSC cells were decreased which is mainly attributed to the reduction of the photocurrent and fill factor. Lower fill factor value implies that incorporation of rGO in photo-anode increases series resistance of the cell, due to imperfection interaction at the interface of rGO and TiO$_2$.

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