Design and Fabrication of High Performance Photoanode of Fe$_2$(MoO$_4$)$_3$/RGO Hybrid Composites for Triiodide Reduction in Dye-Sensitized Solar Cells

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Received: 3 July 2021 / Accepted: 14 December 2021 / Published online: 30 January 2022
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Abstract
Here in, synthesis of Fe$_2$(MoO$_4$)$_3$/reduced graphene oxide (RGO) nanocomposite was prepared by simple hydrothermal approach and used as high efficient dye sensitized solar cells (DSSCs). The decoration of RGO into the Fe$_2$(MoO$_4$)$_3$ was proved by various physic-chemical studies such as XRD, SEM, TEM, Raman, UV, PL and BET analysis. The individual spherical shaped nanoparticles of Fe$_2$(MoO$_4$)$_3$ with sizes in the range of 25–35 nm was uniformly decorated on the surface of RGO nanosheets. Due to the synergic effect between the Fe$_2$(MoO$_4$)$_3$ and RGO the light absorption property is significantly improved as well the high surface area (112.5 m$^2$/g) and pore size (38.7 nm) was achieved than compared with bare Fe$_2$(MoO$_4$)$_3$ (88.5 m$^2$/g and 17.8 nm). The Fe$_2$(MoO$_4$)$_3$/RGO hybrid photoanode establish to display an outstanding catalytic activity toward the reduction of triiodide to iodide in a dye-sensitized solar cell (DSSC) and can provide a solar cell efficiency of 9.6 ± 0.001%, which is superior to a Pt-based DSSC (6.1 ± 0.002%). The better electro-catalytic ability of Fe$_2$(MoO$_4$)$_3$/RGO electrode is obtained by a synergistic effect that resulted in the high specific surface area and intrinsic reactivity of the materials.

Keywords Fe$_2$(MoO$_4$)$_3$ · RGO · High surface area · Photo-conversion · Electron–hole separation · Dye sensitized solar cells

Introduction
Due to the environmental degradation and rapid consumption of petroleum resources, researching into the development of clean energy sources has received widespread attention. Since the past two decades, intensive efforts have been paid to the so-called third generation of solar cells represented by dye-sensitized solar cells because this type of cells offer the compatibility with flexible substrates, low cost, easy fabrication, and high photo to electron conversion efficiency which allows them to compete with fossil fuel electrical generation [1–5]. The main components involved in the fabrication of DSSCs are (i) photoanode (most commonly TiO$_2$ layer-coated FTO glass plate), (ii) light harvesting material (dye), (iii) redox electrolyte and (iv) Pt counter electrode (CE). Titanium dioxide (TiO$_2$) was commonly considered to be fine photoanode content with excellent photochemical stability. However, TiO$_2$ has a large band gap of ~ 3.0 eV and hence can only be photo-excited by UV light (< 400 nm in
wavelength), which is just 5% of the solar light in energy percentage [6–10]. As a result, materials that show a narrower photo-response to the spectral light are strongly recommended to increase energy conversion efficiency, although the photo absorbance was partly contributed by the sensitizer dye molecules. Functional materials have received tremendous attention to date because of their attractive features. Functional materials with an inherent feature, charge carrying ability, specific chemical and physical attributes are provided special properties vs. bulk alternative [11–14]. Recently, molybdate of AMoO 4 forms (A = Zn, Mn, Co, Cu, Cd, Ni, Ba) of nanostructures were designed and synthesized using the hydrothermal process and systematically studied their electrochemical, luminescence and photocatalytic ability in the view of practical device applications [15–18].

Among them, Iron molybdate (Fe 2(MoO4)3), an important metal molybdate was generally used as an efficient catalyst due to their outstanding light absorption ability, luminescence properties, chemical stability, economy, nontoxic of iron and its open three dimensions framework. It also been widely used in different fields of applications such as, photocatalyst, solar cell, optoelectronic devices, alloys preparation, gas sensors and lithium ion batteries [19–24]. However, the lack of direct Fe–Fe or Fe–O–Fe interaction results in its poor electronic conductivity, low rate capability and bad cyclability, all of which have limit its further application. Graphene is a large monolayer sheet of sp2 bonded carbon, which has unique optical, electrical, mechanical, and electrochemical properties. The most important chemically derived graphene is graphene oxide (defined as single layer of graphite oxide). Reduced graphene oxide (RGO) has high aspect ratio and good electronic transport properties, which is expected to facilitate the kinetics of electrochemical reactions. Moreover, RGO has a high surface to volume ratio and strong electronic charge transport, which are supposed to promote transient electrochemistry. Besides, GO has variable optical, mechanical, and electronic properties that can be tuned by controlling the degree of oxidation. Herein we reported the high performance PCE based Fe 2(MoO4)3/RGO DSSC electrode by facile hydrothermal route. In typical DSSC, upon illumination, photo induced electrons from the excited dye transfer toward the conduction band of TiO2 photo anodes, accompanying the oxidation of redox species in the electrolyte (e.g., \( \Gamma^-/I_3^- \)), and simultaneously, the reduction reaction occurs at the counter electrodes by accepting the electrons. By substituting the Pt counter electrode with the GO, the photo induced electrons could be captured by GO and result in the reduction of GO. The Fe 2(MoO4)3/RGO hybrid photoanode establish to display an outstanding catalytic activity toward the reduction of triiodide to iodide in a dye-sensitized solar cell (DSSC) and can provide a solar cell efficiency of 9.6 ± 0.001%, which is superior to a Pt-based DSSC (6.1 ± 0.002%). The better electro-catalytic ability of Fe 2(MoO4)3/RGO electrode is obtained by a synergistic effect that resulted in the high specific surface area and intrinsic reactivity of the photocathode materials.

**Experimental Procedure**

**Materials**

All chemicals used, were of analytical grade, Fe(NO3)3·9H2O (purity 99.99%), (NH4)6Mo7O24·4H2O (purity 99.99%), graphite powder (purity 99.99%), purchased from Dae-Jung Korea. NaOH and ethanol was obtained from Merck chemicals.

**Synthesis of Fe 2(MoO4)3/RGO Composite Samples**

Modified hummers method [8] has been used to prepare the graphene oxide from graphite powders. Appropriate amount of Fe(NO3)3·9H2O and (NH4)6Mo7O24·4H2O was diluted with 50 mL of Milli-Q water and mixed completely. A various weight percentage of GO (1, 2 and 5%) was mixed with the above solution and ultrasonically dispersed for 30 min. Then the pH of the solution was adjusted by adding NaOH drop wise under strong magnetic stirring. The pH of the solution was measured with Digital pH meter and the values are 7.1 and 9.5 for before and after adding NaOH solution. Then the solution was transferred to stainless steel autoclave and the hydrothermal reaction was carried out at 180 °C for 24 h [25]. Finally, the precipitate was centrifuged and washed with distilled water for several times, and then dried at 80 °C for 10 h using vacuum oven. The samples with various ratio of GO (1, 2 and 5%) was named as FMG1, FMG2 and FMG5, accordingly.

**Characterization Method**

X-ray diffractometer (XRD) equipment type Philips PW 137; with CuKα source has been utilized to identify crystalline nature of the obtained products. SEM (Ultra Plus, Zeiss, Germany), TEM (Hitachi) and EDS (AZTEC/Xact) was employed to verify the morphology and elemental analysis of products. Raman spectra analysis was executed with the wavenumber region from 200 to 2000 cm\(^{-1}\) (Raman spectrometer). The optical transmittance of the nanopowders was recorded at room temperature by a Perkin Elmer UV/VIS/NIR Lambda 19 spectrophotometer in the wavelength range of 300–900 nm. Photoluminescence spectra of the samples were collected at room temperature by using Cary Eclipse (e102045776) Fluorescence spectrophotometer in the wavelength range of 400–800 nm.
with a He–Ne laser as an exciting source, and excitation wavelength of 350 nm. The Brunauer–Emmett–Teller (BET) surface area was measured by the Micromeritic ASAP 2020M+C.

Solar Cell Preparation

Fluorine-doped tin oxide (FTO) glass plates were washed (4 cm × 4 cm) in moderate alkaline medium, deionized water, isopropyl, and butyl acetate. The doctor-blade strategy used an opaque film (thickness 55 μm) as a strip to preserve a consistent contact area, these Fe₂(MoO₄)₃ layer-coated FTO sheets were cleaned with deionized water and alcohol and treated at 100 °C for 10 min. By dipping in 0.5 mM N719 dye soluble in tert-butanol/acetonitrile (v/v = 1:1) solution for 24 h under complete darkness, the as-prepared photoanodes (Fe₂(MoO₄)₃) were dye-sensitized at 70 °C. Now, with the aid of two connectors to create the solar cell, this photoanode and the counter electrode (commercial platinum) were sandwiched and was used to isolate the electrodes from touching each other to prevent the short circuit between both the electrodes’ of two conductive surfaces. The redox electrolyte consisted of 0.03 M I₂, 0.6 M 1-butyl-3-methylimidazolium iodide, 0.5 M 4-tert-butyl pyridine and 0.1 M guanidinium thiocyanate with solvent-type hydroxide acetonitrile. Eventually, with a Surlyn film coated with a plastic glass slip, the holes on the rear of the CEs are covered. For the photocurrent–voltage test with an active area of 0.16 cm², the sealed DSSCs were used.

Results and Discussion

Structural Properties

XRD was used to determine the structural and phase purity of the samples and the relevant diffraction pattern is shown in Fig. 1. As shown in Fig. 1, the GO exhibits a diffraction peak at 2θ of 10.75° attributed to (002) the GO plane, which emerges from graphite via the Hummers method. Well crystalline with sharp diffraction peaks were found in the pure Fe₂(MoO₄)₃, and all the peak positions are perfectly matched with monoclinic phase crystal structure of Fe₂(MoO₄)₃ (JCPDS 31-0642). Both GO and Fe₂(MoO₄)₃ diffractions were identified for all the composite samples. Moreover, when the content of RGO increases the diffraction intensity of Fe₂(MoO₄)₃ was significantly improved, which suggest that the incorporated RGO significantly improve the crystalline nature of Fe₂(MoO₄)₃. The average grain size was calculated from Debye scherrer’s formula:

\[ d = \frac{K\lambda}{\beta \cos \theta} \]  

where d is the mean crystallite size, K is the shape factor taken as 0.89, \( \lambda \) is the wavelength of the incident beam, \( \beta \) is the full width at half maximum and \( \theta \) is the Bragg angle.

![Fig. 1 Powder XRD pattern of the samples](image1)

![Fig. 2 Raman spectra of the samples](image2)
related) of the graphene sheet [26]. The typical characteristic peaks of \( \text{Fe}_2(\text{MoO}_4)_3 \) and RGO occur simultaneously, leading to the formation of \( \text{Fe}_2(\text{MoO}_4)_3/RGO \) nanocomposite. From the graph, the ID/IG value of GO and \( \text{Fe}_2(\text{MoO}_4)_3/RGO \) were found to be 1.02 and 1.13, respectively. The higher ID/IG ration of the composite sample is due to higher degree of reduction of GO. Moreover, the widening the D peak in the \( \text{Fe}_2(\text{MoO}_4)_3/RGO \) composite sample with respect to RGO is a further proof of the interaction between the graphenic sheets and the \( \text{Fe}_2(\text{MoO}_4)_3 \) nanoparticles.

**Morphological Analysis**

To evaluate the responsibility of GO scaffold in \( \text{Fe}_2(\text{MoO}_4)_3/RGO \) composites, SEM and TEM images were taken. Figure 3a–c shows the SEM images of bare RGO, \( \text{Fe}_2(\text{MoO}_4)_3 \) and \( \text{Fe}_2(\text{MoO}_4)_3/RGO \) (FMG5) samples, respectively. SEM image of bare RGO (Fig. 3a) shows that crumble like sheets morphology, which is advantageous to the deformation of other metal oxides. The clear uniform size spherical shaped nanoparticles sizes in the range of 25−35 nm are found in pristine \( \text{Fe}_2(\text{MoO}_4)_3 \) nanoparticles (Fig. 3b), which are consistently covered on the RGO sheet surface (Fig. 3c). The distribution of \( \text{Fe}_2(\text{MoO}_4)_3 \) NPs over all the surface of the RGO was further inveterate via high magnification TEM picture, as shown in Fig. 3d−f. The images reveal that neither of them surfaced NPs or isolated NPs outside of the RGO nanosheets have been found. Implied that the rapid expansion of ultrafine \( \text{Fe}_2(\text{MoO}_4)_3 \) NPs existed only on the GO surface. To confirm the stoichiometry of these samples, we mapped the elemental distribution of Fe, Mo, C and O on the surface of samples and the relevant images are shown in Fig. 3g–j. The elemental mapping clearly shows that the RGO was

![SEM images of a RGO, b \( \text{Fe}_2(\text{MoO}_4)_3 \), c FMG5, TEM images of d RGO, e \( \text{Fe}_2(\text{MoO}_4)_3 \), f FMG5; Elemental mapping of g C, h Fe, i Mo and j O](image-url)
successfully incorporated in Fe$_2$(MoO$_4$)$_3$. The weight percentage of Fe/Mo/C/O was found to be 22.4/36.3/27.8/13.5.

**Optical Properties**

UV–Vis DRS was further explored to investigate the optical characteristics of the photoanode materials. Figure 4a) shows the absorption spectra of all the photoanode samples. The absorption of pure sample lies in the visible light region (∼ 485 nm). Further the extended absorption in the red shift wavelength was noticed after loading the RGO content. From these spectra optical band-gap energy ($E_g$) was calculated using Kubelka–Munk equation [27–29]. $F(R \infty) \cdot h^2 = A \cdot (h \cdot E_g)$. The variation of $F(R \infty) \cdot h^2$ versus $h$ was plotted and the straight line range of these plots is extended on the x-axis ($h$) to obtain the values of optical band gap ($E_g$). Band gap energies of all the samples were estimated using the plot of $F(R \infty) \cdot h^2$ versus photon energy ($h$) (Kubelka–Munk equation). The band gap energy of pure Fe$_2$(MoO$_4$)$_3$, FMG1, FMG2 and FMG5 are 2.55, 2.47, 2.31 and 2.25 eV, respectively (Fig. 4b). The recombination of photoinduced electrons and the positive holes after illumination with UV or visible light leads to the emission of photons that produce the characteristic PL peaks. With the aim of studying the coupling photo-induced electron–hole pairing mechanisms, Photoluminescence (PL) spectral analysis is often used and the resultant spectrum is shown in Fig. 5. A clear emission band appeared around at 440–480 nm for all the photoanodes, which is good accordance with the absorption values from the UV results. Remarkably, the intensity emission was gradually decreased when the loading amount of RGO is increases, which suggesting that photo-generated electron–hole recombination system was suppressed due to the solid electron transfers potential of RGO. The introduction of RGO in the Fe$_2$(MoO$_4$)$_3$/RGO nanocomposites is associated with substantial decline in the intensity of the PL spectrum of Fe$_2$(MoO$_4$)$_3$ nanoparticles. This decrease in intensity is ascribed to the fact that RGO can transport the photogenerated electrons rapidly preventing the electron–hole pair recombination which is important in the enhancement of photo conversion efficiency of the fabricated device.

**Textural and Elemental Composition Analysis**

Brunauer–Emmett–Teller adsorption/desorption of the liquid nitrogen technique is used to explore the surface area and size of porous nature. The isotherm was taken for...
Fe$_2$(MoO$_4$) and Fe$_2$(MoO$_4$)$_3$/RGO (FMG5) samples and the related plot is shown in Fig. 6a). Both the samples exhibit the IV type adsorption isotherm, complemented by H3 hysteresis. This characteristics nature resembles that mesoporous of the materials [30–33]. Due to the synergic effect between the Fe$_2$(MoO$_4$)$_3$ and RGO the light absorption property is significantly improved as well the high surface area (112.5 m$^2$/g) and pore size (38.7 nm) was achieved than compared with bare Fe$_2$(MoO$_4$)$_3$ (88.5 m$^2$/g and 17.8 nm). Generally, photoanodes with specific surface and porous structure have attracted much attention, such a structure may render fewer grain boundaries, larger surface area and better light scattering properties. the heterostructure combination of Fe$_2$(MoO$_4$)$_3$/RGO (FMG5)
simultaneous possess the incompatible features such as high specific area, fast electron transport and a pronounced light-scattering effect and demonstrated a high energy conversion efficiency. The ultra thin RGO nanosheets with active surface definitely contribute to the enhanced photocurrent density and thus higher DSSC efficiencies. Chemical state of the samples was further elucidating by XPS analysis. Figure 7a) shows the XPS survey spectrum of Fe$_2$(MoO$_4$)$_3$/RGO (FMG5) samples, which is rendering the key elements of Fe 2p, Mo 3d, O1s and C1s. The Fe 2p$_{1/2}$ and 2p$_{3/2}$ states were presented at 724.1 and 711.1 eV, respectively (Fig. 7b). While the two binding energies were positioned at 235.5 and 232.4 eV, corresponding to the Mo 3d$_{3/2}$ and Mo 3d$_{5/2}$, accordingly (Fig. 7c). The O 1s state of the composite was found with equivalent binding energy of 530.1 eV (Fig. 7d). Core scale of binding C 1s energies of the spectrum shown in Fig. 7e could be the visible peaks at 284.5 eV and 288.6 eV, respectively. This could be due to the characteristic sp$^2$ hybridized graphite-like aromatic rings of RGO and atoms exist.

### Photovoltaic Studies

DSSC performance was evaluated for the constructed sandwich type device, which is shown in Fig. 8a). The DSSC setup with description was also mentioned in our previous published work [34]. The current (J)–voltage
V) characteristics of the DSSC was monitored under sun illumination (1.5 AM) conditions for each of these electrodes (Fig. 8b) and the results are summarized in Table 1. Each sample was tested by five different devices, and the average data were taken. In the J–V tests, the voltage scan range is 0.0–1.2 V with 100 sweep points and the dwell time was 30 ms. There are four parameters: short-circuit current density (Jsc), open circuit voltage (Voc), fill factor (FF) and PCE can be obtained from J–V tests. The error bars were calculated from the J–V curves of four DSSCs for each condition. The results reveal that Fe2(MoO4)3/RGO (FMG5) photoanode show high PCE of 9.6 ± 0.001% due to the higher current density (19.11 ± 0.001). While the PCE value of pure Pt, Fe2(MoO4)3, FMG1 and FMG3 are 6.1 ± 0.002, 4.3 ± 0.003, 6.8 ± 0.002 and 7.3 ± 0.001%. Due to the high reduction of triiodide and electrical conductivity by RGO, the PCE is significantly improved for the composite electrode. Incident photon-to-current conversion efficiency (IPCE) was further carried out to know the role of RGO on the performance of the DSSC and the relevant plot is shown in 8c). The IPCE values are clearly observed in the wavelength between 400 and 700 nm, which is characteristics behavior of N179 dye. The FMG3 photoanode shows high IPCE value of 87% than compared with other photoanode materials such as Fe2(MoO4)3, (41%) FMG1 (54%) and FMG3 (67%). The high stability of the device was further confirmed through multiple experiments of the J–V plot. The device loss only 2.3% of PCE value from its initial value. In addition, electrochemical impedance spectra (EIS) were tested to know the catalytic skill of the electrode samples. Figure 9a) shows the Nyquist plot of the electrodes with equivalent circuit (inset). The semicircle evidently illustrate that smaller Rs (5.4 Ω) and Rct (12.5 Ω)

| Parameters | Pt | Fe2(MoO4)3 | FMG1 | FMG2 | FMG5 |
|------------|----|------------|------|------|------|
| Jsc (mA/cm²) | 13.94 ± 0.002 | 9.77 ± 0.001 | 15.11 ± 0.003 | 17.95 ± 0.002 | 19.11 ± 0.001 |
| Voc (V) | 0.661 ± 0.002 | 0.588 ± 0.002 | 0.726 ± 0.002 | 0.819 ± 0.003 | 0.866 ± 0.003 |
| Fill Factor | 0.66 ± 0.003 | 0.56 ± 0.003 | 0.68 ± 0.001 | 0.73 ± 0.001 | 0.77 ± 0.003 |
| Efficiency η (%) | 6.1 ± 0.002 | 4.3 ± 0.003 | 6.8 ± 0.002 | 7.3 ± 0.001 | 9.6 ± 0.001 |

| Samples | Rs/Ωcm² | Rsh/Ωcm² | r/ns |
|---------|---------|----------|------|
| Fe2(MoO4)3 | 12.5 | 75.1 | 27 |
| FMG1 | 10.5 | 62.4 | 32 |
| FMG2 | 8.1 | 38.3 | 45 |
| FMG5 | 5.4 | 17.7 | 78 |
value of Fe₂(MoO₄)₃/RGO (FMG5) photoanode than other samples. The factors obtained from EIS was also depicted in Table 2. Moreover, the FMG photoanode has high electron life time (78 ns), which is caused by the good electrocatalytic activity to reduction in the triiodide. The photovoltaic mechanism of the proposed work is shown in Fig. 9b. The Fe₂(MoO₄)₃ nanoparticles are anchored to the RGO, which results in excited electrons are generated without impediment. The electrons obtained can be exported from Fe₂(MoO₄)₃. Hence, the conductive layer was effectively prevented by RGO bridges and therefore the recombination of electron–hole pair is effectively prevented.

Conclusions

The present study provides an example of RGO incorporated molybdate hybrid structure as photoanode materials for high performance DSSCs. The Fe₂(MoO₄)₃/RGO hybrid photoanode establish to display an outstanding catalytic activity toward the reduction of triiodide to iodide in a dye-sensitized solar cell (DSSC) and can provide a solar cell efficiency of 9.6 ± 0.001%, which is superior to a Pt-based DSSC (6.17%). The semicircle evidently illustrate that smaller Rs (5.4 Ω) and Rct (12.5 Ω) value of Fe₂(MoO₄)₃/RGO (FMG5) photoanode than other samples. This outcome again features the fact that surface interactions of RGO and Iron molybdate play a key role in the electro catalytic activity, and appropriate regulation of these interactions can be used to maximise to developing new, effective photoanodes in the applications of DSSCs.

Declarations

Conflict of interest Author declares that there is no conflict of interest in this manuscript.

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