



Exploration of graphene oxide nanoribbons as excellent electron conducting network for third generation solar cells

J. Kusuma, R. Geetha Balakrishna*, Siddappa Patil, M.S. Jyothi, H.R. Chandan, R. Shwetharani

Centre for Nano and Material Sciences, Jain Global Campus, Jain University, Kanakapura, Bangalore 562112, Karnataka, India

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ABSTRACT

The future of third generation Quantum dot sensitized solar cells (QDSSCs) can be optimistic if concerns of charge recombination and stability of cells can be worked upon. In this work, we report the role of Graphene materials in its different dimension in overcoming recombination and enhancing power conversion efficiency (PCE). Composite of Graphene oxide nanoribbons (GNRs) synthesized by oxidative unzipping of multi walled carbon nanotubes (MWCNTs) was used as an effective electron transport layer with TiO_2 as photo anode in QDSSCs. The best performing TiO_2 -GNR showed a significant enhancement in PCE of 3.7% for CdS based quantum dots, apart from increased efficiency reported earlier. Investigation of design of anode material reveals that the enhanced surface area and active sites of partially conducting GNRs facilitated uniform deposition of Titania particles (TiO_2) with less aggregation, excellent mobility, presence of organic functional group for anchor of TiO_2 , its 1D quasi structure add to enhanced electron transport (less resistance).

1. Introduction

The third generation quantum dot sensitized solar cells (QDSSCs) have drawn a lot of attention over last decade due to their unique properties like tunability of band gap for broad light absorption, multiple exciton generation, narrow emission, easy processing, low cost, high efficiency and its use as flexible panels [1,2]. Over this short span of time, QDSSCs have yielded an efficiency of 11.6% for Zn-Cu-In-Se quantum dot cells [3] and 13.7% for lead halide quantum dot based perovskites cells [4] which tops the current certified efficiency by NREL. The existing challenges of QDSSCs namely the recombination of exciton and degradation of counter electrode on long exposure to corrosive electrolytes has limited them from commercialization. Although TiO_2 is one of the promising wide band gap metal oxide semiconductor (because of its photo catalytic activity, stability and favorable energy alignments) recombination is one major concern as it forms an assembly of colloidal particles with higher grain boundaries that increases the charge carrier recombination [5]. Incorporation of Graphene materials into TiO_2 has been an established concept for photocatalytic applications. Graphene materials can render unique functionalities on incorporating into wide band gap semiconductor like extension of light absorption, sufficing continuous network for transport of electrons due to their morphology, excellent charge carrier mobility and its ability in separation of exciton for quick transfer of photo injected electrons to overcome recombination. Hence Graphene can be a promising material

for photovoltaic applications [6–8].

Graphene oxide (GO) are two dimensional sheets in morphology, possessing less or no conductivity. Rolling these sheets results in one dimensional tubes called carbon nanotubes (CNTs), and are well known for their strong mechanical property, high thermal conductivity and optoelectronic properties [9]. Moulding sheets into single (metallic or semiconducting) or multi-walled tubes (metallic) imparts them different optical properties [10]. One dimensional strip of these unzipped CNTs are known as Graphene oxide nanoribbons (GNRs). Unzipping of CNTs forms ribbons of few nanometers to hundred nanometers in width with different edge configurations namely arm chair and zig-zag. The angle of chirality of carbon atoms at the edges decides the semi-conducting (arm chair) and metallic (zig-zag) property respectively of GNRs [11]. The flat edge states in case of zigzag configuration allows high density of states to be located at the edges for greater reactivity [12] and lateral confinement of charge carriers in ribbons allows them to possess band gap, which decreases with increase in the width of ribbons [13]. The exceptional property of tuning GNRs to form a semiconductor (unlike insulating in GO), can be a very promising candidate for next-generation semiconductor materials. Since these graphitic structures are finite in dimension compared to infinite GO sheets, they possess edge boundaries which have higher aspect ratio [14] and also the three non-coordinated atoms at edges impart exceptional properties that control the electric and magnetic properties. Also wrinkling of strips leads to a number of fold lines enhancing electrical

* Corresponding author.

E-mail address: br.geetha@jainuniversity.ac.in (R.G. Balakrishna).

Nomenclature

QDSSCs	Quantum dot sensitized solar cells
GNRs	Graphene oxide nanoribbons
MWCNTs	Multi-walled carbon nanotubes
GO	Graphene oxide
ETL	electron transport layer

conductivity in GNRs [14]. These outstanding electronic properties add to the performance wings of electronic devices as an alternative to CNTs and GO sheets. It has been incorporated in some of the major applications like field effect transistors (FETs), sensors, super capacitors and energy based devices. To mention few, Liu et al. reported ribbons based meshes as wearable devices for sensor applications and it showed an excellent recoverable stability sensitive to different tensile strains [15]. Pachfule et al. investigated its usage in electrochemical storage and demonstrated an excellent electrochemical capacitive properties in a steady state as well as in the galvanostatic charge–discharge measurements [16]. Arnold et al. reported direct growth of GNRs onto Germanium semiconducting wafer on a large scale for its use in hybrid integrated circuits making them feasible for industrial applications. This not only promises to boost the performance of next-generation electronic devices significantly but it is going to be very useful material in industrial and military applications, such as sensors and photonic devices which manipulates light [17]. Ghosh et al. reported GNRs composite with copper sulphide as counter electrode to boost the performance owing to its electrical conductivity and is the only literature available for its use in photovoltaic devices [18]. The present paper focuses on study of GNRs and its role in third generation solar cells with comparison to two dimensional GO sheets and one dimensional MWCNTs. This work hence gains significance in providing efficient GNRs as an alternate to CNTs and GO in applications that need quick electron transport and efficient charge separation.

2. Experimental

2.1. Materials and methods

TiCl₄ (99.5%) was used as a titanium source for the preparation of the electron transport layer (ETL) procured from spectrochem and the others include the solvent ethanol, ammonium hydroxide, potassium nitrate, hydrochloric acid, ether, hydrogen peroxide, sulphuric acid, potassium permanganate, acetic acid from Merck. Fluorine doped tin oxide (FTO) glass, triton X-100, graphite powder, MWCNTs, zinc nitrate hexahydrate (Zn(NO₃)₂·6H₂O, 98%), cadmium sulphide powder (100 mesh 99.5%) sodium sulphide non hydrate (Na₂S·9H₂O, 98%), sulphur and Copper sulphide were procured from Sigma Aldrich.

2.2. Synthesis of ETL, GO and GNRs

Sol–gel technique was adopted to prepare TiO₂ (ETL) nano particles [19]. Briefly TiCl₄ was hydrolyzed with water and sulphuric acid. Ammonium hydroxide was used for gel to crystalline conversion at appropriate pH of 8 followed by drying and annealing for 6 h at 600 °C to obtain anatase phase of TiO₂ nanoparticles.

Graphene oxide was synthesized by Hummer's method [20]. Briefly Graphite with H₂SO₄ was cleaved into Graphene sheets with the use of KMnO₄ as strong oxidizing agent. The sheets were allowed for complete oxidation in presence of hydrogen peroxide and were given acid and water wash for purification.

GNRs was synthesized by unzipping of MWCNTs with almost 90% yield as reported by Higginbotham et al. [21] by an efficient chemical oxidation process with phosphoric acid as second acid along with KMnO₄ and H₂SO₄ to control the vacancies created in GNRs by masking

the diol groups.

2.3. Synthesis of graphene composites

The composites of TiO₂-GNR was prepared by solvothermal method as described in protocol by Wang et al. [9] with slight modification of using di-methyl formamide (DMF) as the solvent, because of better dispersion of Graphene. 0.1 g of TiO₂ and different wt% of GNRs (0.04 wt%, 0.08 wt%, 0.12 wt% and 0.16 wt%) were mixed and sonicated for 30 min with DMF. The mixture was placed in autoclave and was maintained at 180 °C for 6 h. The obtained mixture was given water and ethanol wash followed by vacuum drying overnight. The same was followed for other graphene composites.

2.4. Fabrication of solar cells

Cells were fabricated according to standard protocol [22]. In brief, fluorine doped tin oxide (FTO) glass substrates were sequentially cleaned by ultra-sonicating in surfactant, deionized water, and ethanol for impurity free substrates. Paste of TiO₂ was prepared by grinding 0.25 g of TiO₂ with 0.5 ml of acetic acid for 20 min. 1:3 mixture of deionized water and ethanol were added drop wise in intervals to the above paste for better suspension of particles and were further grinded by adding Triton-X (0.5 ml) as binder for 20 min to get a homogenous paste and for better adhesion on the substrate. The same was followed to prepare the composites. FTO glasses were sintered at 70 °C in oven by immersing the electrode in 40 mM solution of TiCl₄ for 30 min to get a compact layer, as this can enhance the short circuit current [23]. The prepared paste was coated on to the FTO substrate by doctor blade technique. The final TiO₂ coated films were sintered at 500 °C for 30 min to stabilize the films and as well to remove if any organic impurities were present. Sintering brings out better electrical connection between nanoparticles. It was then allowed to cool at room temperature. Two such layers were coated and the average thickness of the film was roughly around ~9 μm. The sintered FTOs were sensitized by cadmium sulphide (CdS) QDs by SILAR deposition. 0.1 M aqueous solution of cadmium nitrate (Cd(NO₃)₂) and sodium sulphide (Na₂S) was used as cationic and anionic source respectively. FTO substrates were first dipped in (Cd(NO₃)₂) solution for three minutes and later in (Na₂S) for 30 s and in between rinsing the electrodes with the distilled water to remove excess or unanchored particles for uniform deposition. This completes one SILAR cycle. Eight such SILAR cycles were carried out to get sensitized electrodes. Later it was passivated with inorganic layer (ZnS) of 4 cycles by SILAR deposition with Zn(NO₃)₂ and Na₂S as respective cationic and anionic source in between rinsing with the distilled water. This inorganic layer acts as a barrier avoiding chemical corrosion of QDs and prevents the photo degradation of CdS to CdSO₄ as they are more prone to undergo oxidation in ambient conditions. Polysulphide electrolyte was prepared using 1 M solution of Na₂S and Sulphur which acts as a redox couple or hole transporter with CuS as counter electrode obtained by sonicating CuS with ethanol and drop casting them on conducting side of FTO.

3. Characterization techniques

The Crystal structure and phase identification of these nanomaterials were done using Rigaku XRD using Cu K α radiation (scan rate of 3° min⁻¹). The functional groups were analyzed using Bruker ATR-IR spectroscopic analyzer. The absorption spectrum of samples were recorded using a Shimadzu 1700 PC UV–visible spectrophotometer, the absorbance wavelength was recorded and the band gap was calculated using $E_g = hc/\lambda$ at the absorbance band edges wavelength. Photoluminescence were recorded using Shimadzu RF 5301 PC spectra fluorometer using Xenon lamp. Surface morphology was carried by JSM 7100F JEOL FESEM and HR-TEM was carried out using JEOL/JEM 2100 at an accelerating voltage of 200 kV. The Brunauer-Emmett-Teller

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