



Synthesis, characterization of reduced graphene oxide nanosheets and its reinforcement effect on polymer electrolyte for dye sensitized solar cell applications

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ABSTRACT

High performance polymer electrolyte for dye sensitized solar cell application has been prepared using suitable polymer matrix reinforced by reduced graphene oxide (RGO) nanosheets. RGO nanosheet has been synthesized using appropriately modified Hummer's method. The matrix has been formulated as an optimized blend of polyethylene oxide and polyvinylidene fluoride – hexafluoro propylene (PEO/PVDF-HFP) to get expected high performance characteristics in terms of ionic conductivity, charge carrier concentration and diffusion coefficient. The same has been confirmed using electrochemical impedance spectroscopy and linear steady state voltammetry. Excellent stability of the electrolyte also has been recorded through efficiency retention study. Further, solar cell has been fabricated using the optimized formulation of PEO/PVDF-HFP/RGO nanosheet polymer electrolyte. The optimum performance in comparison with liquid electrolyte based solar cell has been authorized through photo voltaic performance study in terms of open circuit voltage (V_{oc}), short circuit current (J_{sc}) and solar conversion efficiency.

1. Introduction

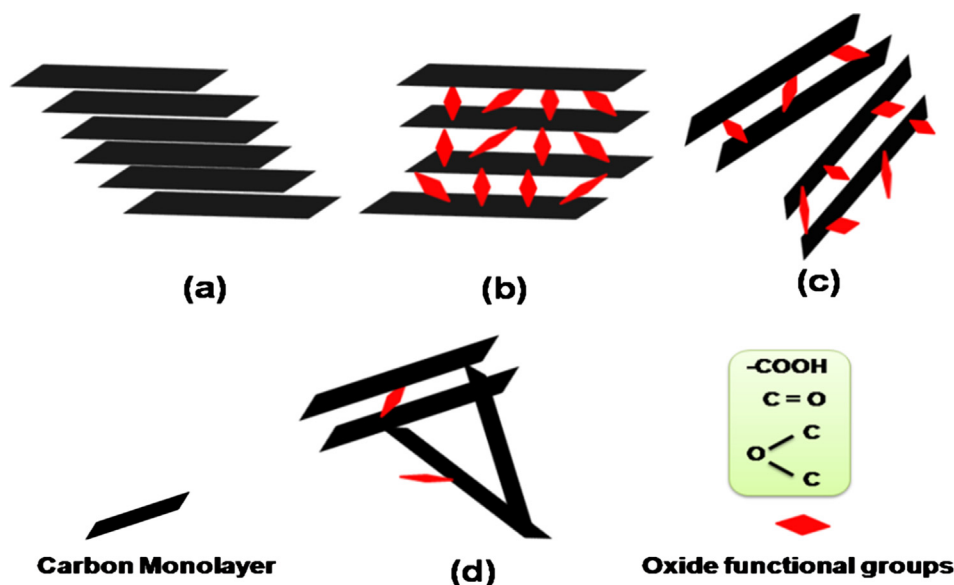
Since the invention of first dye sensitized solar cell (DSSC), the field has been attracting much attention from academic and industrial researcher community due to their superior qualities like simple structure, good plasticity and low cost as compared to the conventional silicon based p-n type solar cells (O'Regan and Gratzel, 1991; Vijayakumar et al., 2015). Typical DSSC is composed of a dye sensitized semiconductor photoanode, a liquid electrolyte comprises redox couple (triiodide and iodine) and platinum coated conducting glass as a counter electrode (catalyst layer) (Sarker et al., 2017; Bella et al., 2013). However, relatively poor stability due to solvent evaporation, electrolyte leakage and electrode corrosion restricts commercialization of the DSSC (Mathew et al., 2014). Various studies have been reported in this field to overcome this issue and proposed DSSC constructed from solid or gel-based polymer electrolytes (Liu et al., 2017). However, the photovoltaic conversion efficiency was found to be a great concern for polymer electrolyte based DSSCs too, even after it bring many other advantages over other electrolytes (Bella et al., 2013).

Commonly, polymers including polyethylene oxide (PEO), polyvinylidene fluoride (PVDF), polyvinylidene fluoride – hexafluoro

propylene (PVDF-HFP), polymethyl methacrylate (PMMA), polyurethane (PU), polyacrylonitrile (PAN), etc., have been well studied as polymer electrolyte for DSSC (Chathuranganie Senevirathne et al., 2018; Duan et al., 2015; Lee et al., 2010; Freitas et al., 2009). Besides this, biopolymers such as agarose, chitosan, acrylic-g-cellulose, and cyanoethylated hydroxypropyl cellulose have also been used for similar applications (Lee et al., 2011; Singh et al., 2013; Singh et al., 2010; Pinjiang et al., 2011; Huang et al., 2012). Among them, the synthetic polymers PEO and PVDF-HFP are promising polymers for polymer electrolytes in DSSC applications. In the present study blend of both the polymers opted as base matrix in view of their appreciable photochemical resistive properties. The properties have been tailored as per the requirement by blending both the polymers into suitable combination. In real the polymers can form a partially miscible blend through the interaction of CF_2 and C–O–C in functionalities from PVDF-HFP and PEO respectively (Prabakaran et al., 2015a).

Further, the properties can be shaped as per the requirement of electrolytic materials by incorporating of nanosized fillers. In this way, carbonaceous nanomaterials likes graphene, a flat single layer of carbon atoms with perfect sp^2 hybridized two dimensional carbon structure, reported as highly influential on the electrical/thermal conductance

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Scheme 1. Schematic diagram of RGO synthesis (a) Graphite (b) Graphite oxide (c) Graphene Oxide and (d) Reduced Graphene oxide.

and physical properties of the polymer due to its inherent properties such as high electrical conductivity, specific surface area (up to $2600\text{ m}^2/\text{g}$), mechanical strength, thermal and chemical stability ((Akhtar et al., 2013; Xu et al., 2008; Zarrin et al., 2011; Qin et al., 2015). Some of the studies have been reported graphene reinforced polymers used in super capacitor, fuel cell membranes, pressure sensors and actuators (Wang and Hu, 2012). It is expected that the addition of reduced graphene oxide (RGO) into the polymer/gel electrolytes can not only reduce crystallinity and glass transition temperature as like inorganic nanofillers through reduced cohesive force between the polymer macromolecules and recrystallization, but also can enhance the catalytic action within the electrolyte (Appetecchi et al., 2000; Wachtler et al., 2004; Yang et al., 2013; Prabakaran et al., 2015a,b; Wang and Hu, 2012). Thus, rapid reduction reaction can take place and provide more number of anions.

In the present work, reduced graphene oxide nano sheets have been synthesized from graphite powder by using modified Hummers method. The characteristic features of synthesized RGO nano sheets were confirmed Fourier infrared spectroscopy (FTIR), wide angle X-ray diffraction (WAXD), Raman spectroscopy, atomic force microscopy (AFM) and transmission electron microscopy (TEM). Further, the RGO sheets were incorporated into the PEO/PVDF-HFP blends by solution casting method. Further, the effect of RGO nano sheets reinforcement on structural, thermal, and electrochemical properties of PEO/PVDF-HFP electrolyte membranes was systematically investigated. Finally, solid state dye sensitized solar cells have been developed from the optimized composition PEO/PVDF-HFP/RGO electrolytes and their photovoltaic characteristics was performed.

2. Experimental procedure

2.1. Materials

Poly(vinylidene fluoride-co-hexafluoropropylene) (PVDF-HFP, Kynar flex 2801) with melt viscosity of 23–27 K poise was supplied by Arkema (India). Poly(ethylene oxide) (PEO) ($M_w = 5 \times 10^6$), Triton X-100 (laboratory grade), Titanium IV-n-butoxide (purity 99%) and Trisodium citrate dihydrate ($\text{HOC}(\text{COONa})(\text{CH}_2\text{COONa})_2 \cdot 2\text{H}_2\text{O}$) (purity 99%), graphite powder were purchased from Sigma India. Anhydrous Lithium Iodide (LiI) and Iodine (I_2) were purchased from thermo fisher scientific India. Di-tetrabutylammonium-cis-bis(isothiocyanato)bis(2,2'-bipyridyl-4,4'-dicarboxylato) ruthenium(II) (N-719) dye were

purchased from Solaronix, Switzerland. 4-*tert*-Butylpyridine (purity > 96%) and 1-Methyl-3-propylimidazolium Iodide (purity > 95%) were purchased from TCI chemicals, India. Zinc nitrate hexahydrate ($\text{Zn}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$) (purity 99%), sodium hydroxide (NaOH) (99%), Anhydrous dimethyl formamide (DMF) (99.5%), ethanol (99.9%), acetylacetone (99%), sulfuric acid (H_2SO_4 , 63%), sodium nitrate (NaNO_3), potassium permanganate (KMnO_4), hydrogen peroxide (H_2O_2), hydrazine hydrate were purchased from Himedia, India, and FTO glass plate ($R_{\text{sh}} < 10\ \Omega/\text{cm}^2$) supplied by M/s. Shilpa Enterprises, India.

2.2. Synthesis of reduced graphene oxide

GO was synthesized from graphite using Hummer's method with slight modifications. 0.325 g of graphite powder is slowly added into the solution contains 0.15 g of NaNO_3 in 7.5 ml of H_2SO_4 at $0\ ^\circ\text{C}$. The above solution was stirred and obtained a good wetting of graphite powder in the solution after stirring at room temperature for 30 min. Further, 1 g of KMnO_4 is slowly introduced into the above and became viscous mixture. Deionized water is added when exothermic reaction started (beaker kept in ice bath) and continued till exothermic reaction persisted to form a brown colored solution. 1 ml of H_2O_2 was added to the above reaction mixture to form yellowish green colored solution and stirred for 12 h at room temperature. Further, it was filtered and washed with 10% HCl and distilled water to make it neutral.

0.2 g of graphene oxide is added into 200 ml of distilled water under stirring at 500 r.p.m. Then, 1 ml of hydrazine hydrate is added and the solution is kept at $100\ ^\circ\text{C}$ for 12 h. Finally, the product was washed with 5 vol% methanol and distilled water until the product became neutral. The brown color of GO was turned to black color reduced graphene oxide. The product was dried in oven at $80\ ^\circ\text{C}$ for 24 h. Scheme 1 represents various stages of formation of reduced graphene oxide (RGO) via chemical oxidation and reduction processes.

2.3. Preparation of RGO-polymer electrolyte

PEO/PVDF-HFP electrolytes were prepared as per the optimized processing parameters reported in our earlier publication (Prabakaran et al., 2015a,b). Optimized composition of poly(vinylidene fluoride-co-hexafluoropropylene) (PVDF-HFP) and Poly(ethylene oxide) (PEO) (6:4 wt% ratio) were dissolved in DMF (99%): glycerol (1%) at $80\ ^\circ\text{C}$. Different wt.% of (0.2–1.4) of reduced graphene oxide nanosheets were

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