



# An electrocatalytic active lyocell fabric cathode based on cationically functionalized and charcoal decorated graphite composite for quasi-solid state dye sensitized solar cell



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## ABSTRACT

The state of the art of conductive functional textile woven fabrics have given rise to a demand for textile integrated electrodes. Herein, we report a highly conductive and flexible woven fabric electrode using highly absorbent lyocell fabric as the substrate and cationically functionalized and activated charcoal decorated graphite composite (AC-GC) as the coating film. This (AC-GC) coated lyocell fabric is used as a cathode for quasi-solid state dye sensitized solar cell (Q-DSSCs). Our suggested fabric based cathode shows sufficiently high conductivity and electrocatalytic activity (ECA) compared to platinum (Pt) based reference counter electrode (CE). This efficient CE demonstrates extremely low charge transfer resistance ( $R_{CT}$ ) of  $1.56 \Omega \text{ cm}^2$  with polyethylene oxide based quasi-solid electrolyte. The cationic charged enriched charcoal decorated graphite planner structure provide more availability of active sites for the reduction of negatively charged tri-iodide ( $I_3^-$ ) ions present in polymeric gel electrolyte. The formation of porous charcoal voids and conductive graphite channels entrap large amounts of gel electrolyte and provide fast diffusion of iodide/tri-iodide ( $I^-/I_3^-$ ) ions. Our organic system of AC-GC coated lyocell fabric based DSSCs assembly demonstrated 7.09% power conversion efficiency (PCE) when fabricated with quasi-solid electrolyte. This AC-GC coated fabric CE is also highly stable in water and electrolyte solution. The adequate electrocatalytic activity and cyclic stability demonstrate that this AC-GC coated fabric can be used to replace expensive Pt CE and can be used in flexible solar cells in future.

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## 1. Introduction

Electronic textiles (e-textiles) or self-powered textile fabric embedded with functional electrochemical properties provide a diverse range of smart textile applications, such as textile supercapacitors, flexible lithium ion batteries, textile based touch sensors, and solar energy devices (Sahito et al., 2016; Arbab et al., 2015b; Jung et al., 2016; Zebardastan et al., 2016). Textile structured electronic devices are needed for energy harvesting and storage, since conventional glass-based electrodes are rigid, bulky, and heavy to seamlessly assemble into wearable electronics. Compared to conventional solar cell devices, dye sensitized solar cells (DSSCs) possess advantages of having low cost, ease of fabrication, and

appreciable power conversion efficiency (PCE). The conventional DSSC is assembled with fluorinated tin oxide (FTO) glass based electrodes, separated with liquid-iodide based electrolyte. The photo anodes used in DSSCs are fabricated by pasting a mesoporous titania film adsorbed with N719 dye. In contrast, the counter electrode (CE) is fabricated by means of coating a highly electrocatalytic platinum (Pt) layer on FTO glass. The Pt layer requires a very high temperature ( $\sim 400^\circ\text{C}$ ) for its activation. The high thermal annealing of the Pt layer and expensive FTO glass contributes to around 30–60% of total DSSCs cost (Lee et al., 2010). Furthermore, Pt is one of the rarely found elements and Pt-FTO glass assembly makes it unsuitable for roll to roll production of solar cells (Kroon et al., 2007). Recently, textile substrates such as cotton and polyester have been reported as flexible CE material in fabrication of DSSC. However, lyocell (biodegradable manmade cellulose) commonly known for its superior adsorption ability and better mechanical properties as compared to cotton

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fabrics (Okubayashi et al., 2004; Kwon et al., 2014) has not been reported as a substrate material for CE preparation. The high strength, superb adsorption ability and wrinkle resistant characteristics of lyocell fabric makes it attractive material for application as textile structured electrode substrate. Although, the non-conductive nature of textile substrate requires a coating of highly conductive and electrocatalytic active materials for the application as counter electrode material.

Recently, various carbon based structures (Janani et al., 2015; Cai et al., 2012; Li et al., 2016a), such as multi-walled carbon nanotube (Arbab et al., 2015a; Li et al., 2015; Hou et al., 2016; Wu and Ceng, 2016; Memon et al., 2017a), single walled carbon nanotubes (Anwar et al., 2013; Yan et al., 2013), activated charcoal (Adachi and Hoshi, 2013; Beyer et al., 2013), graphite (Veerappan et al., 2011; Liu et al., 2011), and graphene (Mengal et al., 2016; Sahito et al., 2015; Kavan et al., 2014; Kavan, 2013) have been proposed for coating of flexible electrode system. Amongst these, graphite is the low cost and most promising materials to be utilised as electroconductive materials. Its high conductivity and chemical stability due to hexagonally stacked carbon layered structure display unique properties for electrochemical applications. The highly oriented graphite matrix structure is composed of edged and basal planes (Katagiri et al., 1988; Bu and Hu, 2016). However, its dominant basal planner morphology and defect-free matrix limits its electrocatalytic activity to the side edges (Banks et al., 2005), causing these graphitic planner structures to possess fewer catalytic sites for tri-iodide/iodide reduction reaction. As a result, different techniques have been proposed in the past to incorporate defects in the graphite structures (Terrones et al., 2010).

Conventional cationic functionalization techniques involves the use of harsh acidic treatments and highly concentrated solvents (Martincic et al., 2015). The chemical treatments fragment the graphite sheets and decrease their electrical conductivity. In addition, graphite structure have low adhesion towards the flexible substrate. Herein, we propose a cationic functionalization of graphite using enzymatic dispersion with acidic media using acetic acid at pH  $\sim$ 3. This cationic functionalisation evenly distributes the positively charge ions on the graphite sheet surface. Further more, cationic functionalization evenly disperses the graphite without affecting its planner morphology. The cationic charged graphite sheets will locate negatively charge iodide ions ( $I^-$ ) present in gel electrolyte, and will fasten the reduction mechanism of tri-iodide ions (Kaniyoor and Ramaprabhu, 2012).

In order to further enhance the surface area of graphite sheet, an inclusion of defect rich carbon materials were thoroughly studied. For this purpose, we used activated charcoal (AC) with 100 mesh size to incorporate defects rich edges into the planner morphology of graphite sheets. The amorphous AC possess a multi-edged surface with a porous structure (Chmiola et al., 2006; Miller and Simon, 2008; Memon et al., 2017b). Due to high surface area, AC possess high charge storage capacity and electrocatalytic activity with high charge transfer kinetics (Imoto et al., 2003; Li et al., 2009). However, in AC, the formation of grain boundaries decreases its conductivity (Li et al., 2016b). For this purpose, AC was used as supportive defect rich material for graphite sheets. The induction of mesoporous and electrocatalytically active AC on to the highly stacked basal plane of electroconductive graphite enhanced the number of electrocatalytic sites on the graphitic structure without compromising its electrical conductivity. Our proposed system of cationised functionalized graphite, followed by addition of AC provides synchronized characteristics of both high conductivity and ECA. This AC-GC structure is evenly distributed and absorbed into highly absorbent lyocell fabric. The AC-GC coated lyocell fabric electrode is further used as a platinized FTO free CE for quasi-solid state DSSC. Our proposed integrated system of AC-GC coated lyocell fabric CE is envisioned

to facilitate the fast charge transfer kinetics and improve the device photovoltaic efficiency.

## 2. Experimental

### 2.1. Materials

Plain weaved, 100% lyocell fabric was used as electrode substrate, provided by Tencel (Austria). For synthesis of electrocatalytic layer composed of activated charcoal decorated graphite composite (AC-GC), activated charcoal powder (100 mesh particle size, Sigma Aldrich), Graphite powder (particle size  $<100 \mu\text{m}$ , Asbury Carbon), lipase enzyme from rugose (type VII, Sigma Aldrich), Carboxymethyl cellulose (CMC, sodium salt of  $M_w$  250,000, Sigma Aldrich) as binder and acetic acid (Junsei chemicals, Japan) for acidic pH were used. Platinized FTO glass CEs were prepared using Chloroplatinic acid hexahydrate ( $\text{H}_2\text{PtCl}_6$ ) from Sigma Aldrich, and fluorinated tin oxide glass (FTO) with  $\sim 8 \Omega \text{cm}^{-2}$  resistivity from Pilkington Co. was used. For preparation of quasi-solid state electrolyte, polyethylene oxide (5%,  $M_w$  300,000) polymer, 1-butyl-3-methylimidazoliumiodide (BMII), Iodine ( $I_2$ ), Lithium iodide (LiI), 4-tert-butylpyridine (TBP), and anhydrous acetonitrile were purchased from Sigma Aldrich Co. For fabrication of photo anode, titania powder ( $\text{TiO}_2$ ) P25 (main layer, 20 nm) and G2 (scattering layer  $<500 \text{nm}$ ) was used. Dye sensitizer D719 cis-di-isothicyanato-bis (2,2'-bipyridyl-4,4'-dicarboxylato) ruthenium (II) bis (tetra butyl ammonium) from Everlight Co. was used. For sealing of fabric based DSSCs, thermoplastic ionomer Surllyn ( $60 \mu\text{m}$ ) purchased from Solaronix was used.

### 2.2. Fabrication of AC-GC coated lyocell fabric counter electrode

Initially,  $3 \text{mg mL}^{-1}$  cationised lipase aqueous solution was prepared. Acetic acid was added to maintain the acidic pH of the solution. Then 2 g of graphite powder was introduced and sonicated in 100 mL of cationised lipase solution for 2 h. By using cationised lipase solution, the positively charge ions distributed over graphite sheets. Afterwards, AC was gradually added into the solution and stirred for 24 h. For fabrication of highly conductive and electrocatalytic active electro catalyst carbon composite, different percentages (0%, 25%, 50%, 75% and 100%) of AC was added to graphitic solution. The dispersion was then ultra-sonicated for 1 h using sonic probe<sup>®</sup>, USA with 40% amplitude, to segregate any remaining chunks of carbonaceous materials. The homogenous carbon solution was then vacuum filtered using  $0.5 \mu\text{m}$  pore size PTFE polymer filter membrane. The carbon cake was thoroughly washed with DI water to remove excess impurities followed by vacuum filtration. An aqueous solution of 2% CMC binder was prepared and mixed in the carbon cake and ultra-sonicated to get a fully dispersed homogenous carbon paste. The as-synthesized carbon paste was stored overnight at room temperature for ageing. The lyocell substrate was then cut into small squares and washed with hot water to remove dirt. The as-synthesized paste was coated onto the cleaned and dried lyocell fabric by a simple tape casting method. The coated samples were dried at room temperature to avoid layer cracking. The thickness of  $\sim 60 \mu\text{m}$  was carefully controlled to avoid short circuit in the fabricated cells. For effective adhesion of AC-GC structure over fabric and to achieve a high interlayer sheet contact, the samples were dried at  $50^\circ\text{C}$  for 30 min followed by hot pressing at  $130^\circ\text{C}$ . Hot pressed AC-GC coated lyocell samples were cut down into  $15 \text{mm} \times 15 \text{mm}$  square shape and adhered to PET sheet using double sided adhesive tape. PET sheet was used as support material for the fabrication of DSSCs. The detailed schematic illustration of AC-GC coated lyocell fabric electrode preparation is given in Fig. 1. In addition, for comparison, Pt

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