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Dye-Sensitized Solar Cells with Cost-Effective Counter Electrode Based on Soybean Oil Lampblack

Umesh Lawaju[1], Prakash Joshi[1,*]

[1] *Physics Department, Bhaktapur Multiple Campus, Tribhuvan University, Bhaktapur, Nepal*

[*] *Corresponding author: prakash.joshi@bkmc.tu.edu.np*

Abstract. We have used soybean oil lampblack as a low-cost counter electrode (CE) material to replace expensive platinum in dye-sensitized solar cells (DSCs). The lampblack was prepared by obstructing the flame of the soybean oil lamp with a porcelain bowl and characterized with the techniques of X-ray diffraction (XRD) and Raman spectroscopy for structural analysis and scanning electron microscopy for surface morphology. The XRD of the lampblack indicated the presence of graphitic carbon in the lampblack while Raman spectroscopy of the carbonaceous material disclosed that the lampblack consists of both disordered and ordered forms of carbon. The lampblack was used with printer toner to prepare counter electrodes (CEs) of DSCs. The scanning electron microscopy of the film of the lampblack with toner showed that the toner serves as a binder to hold the particles of the lampblack on the CEs. The catalytic ability of the CEs was evaluated with electrochemical impedance spectroscopy (EIS). The charge transfer resistance R_{ct} at the toner-electrolyte interface determined by EIS was extremely high (5.32 x 10^5 Ωcm^2). Similarly, R_{ct} at the carbon-electrolyte interface and platinum-electrolyte interface were 13.46 Ωcm^2 and 2.29 Ωcm^2, respectively. The DSCs based on soybean oil lampblack achieved the light to electric energy conversion efficiency (η) of 2.58% and the platinum based solar cell achieved η of 3.67% .

Keywords: Counter electrode, Charge transfer resistance, Dye-sensitized solar cell, Electrochemical impedance spectroscopy, Lampblack

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INTRODUCTION

Dye-sensitized solar cells are the third generation solar cells which aim to enhance the light to electricity conversion efficiency while maintaining low manufacturing costs [1]. A DSC consists of two electrodes: photoelectrode and counter electrode. These two electrodes are separated by a liquid electrolyte [2].

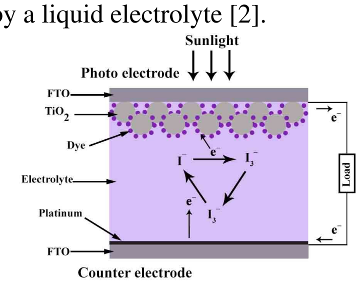

FIGURE 1. A schematic diagram of DSCs. Modified from ref. [3].

The photoelectrode consists of a thin film of nanocrystalline TiO_2 particles coated on a transparent conducting oxide film like fluorine doped tin-oxide-glass (FTO-glass) substrate and the TiO_2 particles are coated with light sensitive dye molecules [4]. The counter electrode (CE) is an FTO-glass substrate generally coated with a thin film of platinum. The liquid electrolyte is a solution of chemicals dissolved in a solvent containing ions like iodide (I^-) and tri-iodide (I_3^-) [4].

When dye molecules adsorbed on the TiO_2 surface of the photoelectrode are exposed to sunlight, photoelectrons are injected into the TiO_2 film. The photoelectrons pass through the porous nanocrystalline TiO_2 film to the transparent conducting oxide layer of the photoelectrode. Then the electrons flow through an external load to the counter electrode, where electrons are captured by tri-iodide ions of the electrolyte, and the tri-iodide ions are converted into iodide ions. The thin film of platinum coated on the CE acts as an efficient electrocatalyst for the reduction of I_3^- ions. The dye molecules oxidized due to loss of electrons are quickly regenerated by gaining electrons from iodide ions in the electrolyte and the iodide ions are converted into tri-iodide ions [2, 4, 5, 6].

Platinum has been extensively used as the CE material in DSCs because of its effective catalytic property for tri-iodide reduction [4]. However, the high cost of platinum can be a disadvantage in developing DSCs as low-cost solar electricity generating devices. Moreover, Olsen et al. reported the dissolution of platinum film in the liquid electrolyte containing LiI and I_2 [7].

In order to address these issues of platinum, several alternative materials have been proposed as CEs in DSCs [2, 4, 6, 8, 9, 10, 11, 12, 13, 14]. Among them, different types of carbonaceous materials and their composite such as carbon black [15], carbon nanotubes [16], carbon nanofibers [4, 17], graphene/polyaniline nanocomposite [18], and nickel/carbon nanofiber composite [19] have been investigated as counter electrode materials due to their low cost, efficient catalytic ability for the reduction of tri-iodide ions, and chemical stability in the corrosive electrolyte. Recently, Joshi et al. reported the use of mustard oil based lampblack as another low-cost carbonaceous CE material and demonstrated that the lampblack possesses a good catalytic ability for the reduction of tri-iodide ions [9]. Although the light to electricity conversion efficiency of the DSCs based on lampblack was slightly lower than that of the platinum based DSCs, the use of lampblack in DSCs has advantages like simplicity in the synthesis of the lampblack (it can be prepared by using merely a traditional oil lamp) and simplicity in coating the film of the lampblack onto an FTO-glass substrate (it can be coated on the FTO-glass substrate by the simple technique of doctorblading) [9]. Despite these benefits of lampblack as a CE material, only a limited study on lampblack (as another potential CE material) has been carried out so far.

In this research, we have introduced soybean oil lamp based lampblack as a low cost CE material. It was characterized by using Raman spectroscopy, X-ray diffraction (XRD), electrochemical impedance spectroscopy (EIS), and scanning electron microscopy (SEM), and used in the preparation of CEs to compare its catalytic ability for the reduction of tri-iodides ions to that of platinum.

EXPERIMENTAL METHODS

Preparation and characterization of lampblack of soybean oil

The methods of preparation of soybean oil based lampblack were similar to those described by Joshi et al. [9]. The flame of a soybean oil lamp was obstructed by keeping a porcelain bowl (mortar) just above the top of the flame and the lampblack (lamp soot) formed on the bowl's concave surface was collected by scratching the lamp soot with a spatula. Then the lampblack was grinded with a pestle.

The structural analysis of the lampblack was carried out by using the techniques of XRD and Raman spectroscopy. The XRD of the lampblack was carried out using D2 PHASER (Bruker) X-Ray diffractometer (at Nepal Academy of Science and Technology) and the Raman spectroscopy was carried out using RIHRM Raman Spectrometer (with 532 nm diode Laser at RI Instruments and Innovation, India). Similarly, the shape and size of the lampblack powder were investigated by scanning electron microscopy. The scanning electron microscope images were obtained by using the LEO 435VP scanning electron microscope (at the Indian Institute of Technology, Roorkee, India).

Fabrication and characterization of the counter electrode

Lampblack powder and printer toner powder (in the ratio of 1:1 by weight) were mixed in a porcelain mortar and the mixture was grinded with a pestle. The toner powder in the mixture was used as a binder rather than a catalyst as reported by Joshi et al. [9]. The paste of the soybean oil lampblack and toner powder prepared in ethanol was doctorbladed onto the conducting surface of cleaned FTO-glass substrates. Then the substrates were sintered at 80°C for several hours on a hot plate. And, the surface morphology of the film of the lampblack was analyzed by obtaining its SEM images.

To compare the catalytic activity of lampblack for the reduction of tri-iodide ions with that of platinum, the CE with thermally deposited platinum was also prepared. To prepare the CE with a thin film of platinum, a few drops of the solution named Platisol T (precursor of platinum purchased from Solaronix, Switzerland) was painted on an FTO-glass substrate, then the FTO was calcinated on a hot plate at a temperature of 450°C for about half an hour [9].

Fabrication of photoanode

The photoanodes were prepared in two steps. First of all, a thin film of transparent TiO_2 was prepared on FTO-glass substrates, then the TiO_2 film was sensitized with N-719 dye. The transparent TiO_2 film was prepared by doctorblading Ti-Nanoxide T/SP paste (purchased from Solaronix, Switzerland) onto the FTO-glass substrate and then calcinating the doctorbladed paste at the temperature of 450°C for 45 minutes. The transparent TiO_2 film was sensitized with the dye by immerging the film cooled at 80°C into the solution of Ruthenizer 535-bisTBA (N-719 dye purchased from Solaronix, Switzerland) with a concentration of $\sim$ 0.25 mM in ethanol for about 24 hours [9].

Device fabrication and characterization

For the fabrication of a working solar cell, the counter electrodes and the photoelectrodes were assembled using a sealant (parafilm sheet) while keeping a gap between them. The gap was subsequently filled with a liquid electrolyte containing iodide/tri-iodide ions [9]. The counter electrodes and photoanodes were dried before assembling. The counter electrodes based on the lampblack powder were dried by warming them at 80°C for about 15 minutes to remove water vapor absorbed by the carbon film. Similarly, the photoanodes were dried with a hot air drier after they were taken out of the dye solution and washed with ethanol.
In order to compare the photovoltaic performance of the DSCs with CEs based on the lampblack, the DSCs with CEs based on platinum were also prepared. The DSCs were tested by illuminating the solar cells with the stimulated light of 100 $mW cm^{-2}$ emitted from Abet SunLite Solar Stimulator 11002 at the laboratory of Kathmandu University, Nepal.

Fabrication of Dummy Cells for EIS

In order to evaluate the catalytic ability of the carbon film of the soybean oil lampblack, electrochemical impedance spectroscopy (EIS) of the dummy cell with the lampblack based carbon film was carried out. The dummy cell was prepared by assembling two symmetrical electrodes coated with the carbon film (the counter electrodes used in the fabrication of the carbon based DSCs) and filling the gap between the electrodes with the electrolyte containing iodide and tri-iodide ions [4, 20]. To compare the catalytic property of the carbon film, EIS of the dummy cells without catalyst (only with FTO), with toner film, and with platinum was also carried out. The EIS of the dummy cells was carried out in dark using the potentiostatic mode of Interface 1010E (Gamry Instrument, USA) at zero DC bias voltage and with a sinusoidal wave of 10 mV of amplitude and frequencies ranging from 0.1 Hz to 100 KHz.

RESULT AND ANALYSIS

Figure 2(a) is the X-ray diffraction of soybean oil lampblack. The intensity peaks at $2\theta \approx 25°$ of the XRD patterns indicate that the lampblack contains carbon with graphitic structure [4].

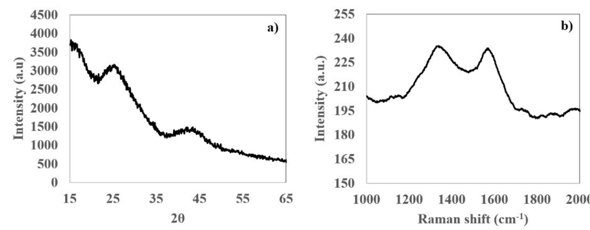

FIGURE 2. (a) X-ray Diffraction (XRD) and (b) Raman spectrum of soybean oil lampblack

Figure 2(b) is the Raman spectrum of the lampblack obtained from soybean oil. The Raman spectra have two characteristics bands. One of them is the “D-band”, which is centered at the wavenumber of $\sim$1339 cm^{-1}. This is related to disordered carbonaceous materials. The other is “G-band”, which is centered at the wavenumber of 1566 cm^{-1}. It is related to the ordered carbonaceous material (graphitic carbon) [4]. The finding of the Raman spectroscopy of the lampblack supports the XRD result that revealed the presence of graphitic materials in the lampblack. Previous researchers have reported that materials with graphitic form of carbon can be used as an efficient CE material [2, 4]. Thus, the lampblack can also be used as the catalyst for the tri-iodide reduction in DSCs.
Figure 3(a) is the SEM image of lampblack powder of soybean oil. The SEM image revealed that the lampblack has a porous and rough surface formed by circular particles having a dimension around 100-150 nm.

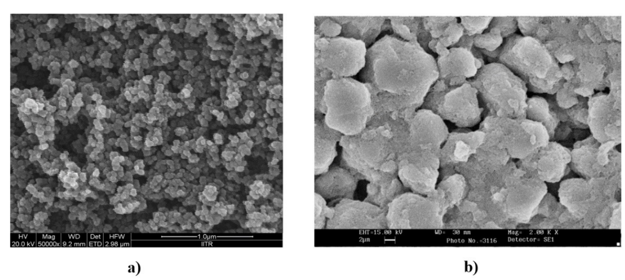

FIGURE 3. SEM images of (a) soybean oil lampblack powder and (b) Top view film of soybean oil lampblack with printer toner used in CE

Figure 3(b) is the top view of scanning electron microscopy (SEM) image of the film prepared from soybean oil based lampblack with printer toner. The SEM image shows that the film consists of irregular shaped large particles covered by fine particles (powder). The size of the irregular shaped large particles was around 10 μm which is much larger than the 100-150 nm sized and circular shaped carbon particles of lampblack. As the shape and size of the larger particles were different from those of lampblack powder as seen in Figure 3(a), the larger particles can be identified as the particles of the printer toner whereas the fine particles, which cover the printer toner, are the particles of the lampblack powder. Also, the SEM images show the presence of scattered micro-sized pores throughout the carbon films.

Previous researchers have mentioned that pores formed in the carbon film of the counter electrode will provide easy access for I_3^- ions to penetrate deep inside the film and enhance the reduction of the tri-iodide ions [5, 16]. Furthermore, the rough surface morphology of the film provides a larger electrolyte-catalyst interface, which may be beneficial for the fast reduction of the tri-iodide ions.

Figure 4(a) and 4(b) are the current density-voltage (J-V) curves of the DSCs with CEs prepared from soybean oil lampblack and platinum, respectively. The photovoltaic parameters of the DSCs extracted from the corresponding J-V curves are enlisted in table 1.

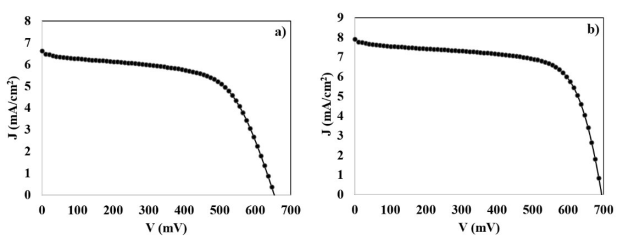

FIGURE 4. Current density-Voltage (J-V) curves of DSCs with CEs prepared from (a) soybean oil lampblack, (b) platinum, tested under the illumination of an intensity 100 $mWcm^{-2}$.

TABLE 1. Comparison of photovoltaic performance of DSCs with different CE materials.

CE materials used in DSCs	J_{sc} (mA/cm^{-2})	V_{oc}(V)	FF	$\eta(\%)$
Soybean oil lampblack with toner	6.61	0.657	~0.60	2.58
Platinum	7.91	0.697	~0.67	3.67

Among the photovoltaic parameters, short circuit current density (J_{sc}), open circuit voltage (V_{oc}), fill factor (FF), and overall light to electricity conversion efficiency (η), the value of power conversion efficiency is the most significant one, however, it is influenced by the other parameters. The light to energy conversion efficiency (η) of the soybean oil lampblack based DSC was 2.58%, and that of platinum based cell was 3.67%. So, the η of lampblack based DSC was less than the platinum based cell. This finding is similar to the result reported by previous researchers: Joshi et al. reported that the DSCs based on lampblack of mustard oil/printer toner composite yielded η of 3.20% and the DSCs based on platinum yielded η of 4.18% [9].

Figure 5 shows Nyquist plots of the symmetrical dummy cells with the lampblack carbon, platinum, FTO, and toner.

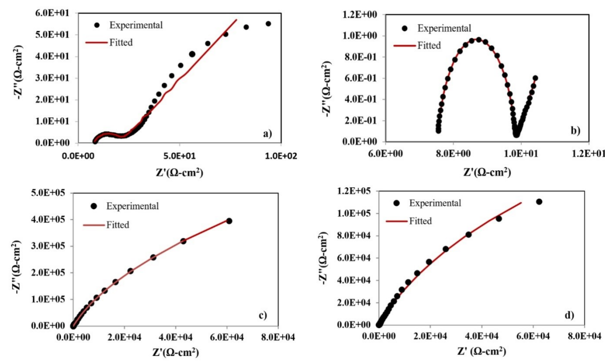

FIGURE 5. Nyquist plots of symmetrical dummy cells with a film of (a) soybean oil based carbon, (b) platinum, (c) FTO, and (d) printer toner.

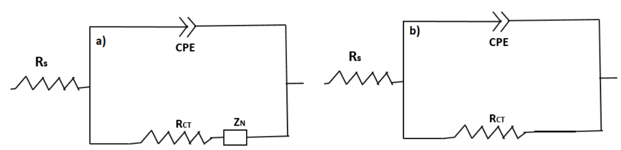

FIGURE 6. Equivalent circuits (a) including Nernst diffusion impedance and (b) excluding Nernst diffusion circuit (the Randle circuit used to fit the curve in high frequency region of the spectrum). Modified from ref. [20].

Figure 6 shows the equivalent circuits for fitting the Nyquist plots of the dummy cells. The equivalent circuit shown in Figure 6(a) is generally used for fitting the Nyquist plot of symmetrical dummy cells [20]. The equivalent circuit comprises R_s (series resistance), R_{ct} (charge transfer resistance), CPE (constant phase element), and Z_n (Nernst diffusion impedance) [4, 20, 21]. The R_{ct} and CPE are related to the charge exchange at the interface whereas Z_n is related to the diffusion of the ions in the liquid electrolyte [4, 20, 21, 22]. This equivalent circuit was used for fitting the Nyquist plots of the dummy cells with lampblack and platinum. In the case of the dummy cells prepared with FTO only and toner only, the equivalent circuit excluding Z_n (Randle circuit only with R_{ct} and CPE) shown in figure 6(b) was used aiming to extract the values of R_{ct} with better fitting of the Nyquist plots [20, 21].

The parameters obtained from the curve fitting by using GamryEchem Analyst (Gamry Instrument, USA) are enlisted in Table 2. R_s is basically due to the series resistance of the FTO [22]. Thus, the values of R_s of all

TABLE 2. Parameters determined from fitting of Nyquist plots

Symmetrical cell with	$R_s(\Omega)$	β	R_{ct} (Ωcm^2)
Soybean oil lampblack with toner	24.27	0.711	13.46
Platinum	23.77	0.894	2.29
FTO	22.24	0.959	46.2 x 10^5
Toner	21.96	0.846	5.32 x 10^5

of the four types of symmetrical cells are comparable. β is a parameter of the impedance of CPE, $Z_{CPE}=\frac{1}{(J\omega)^{\beta}(Y_0)}$ [4, 20] and it is related to the porosity or roughness of the film coated on the electrodes. The value of β lies between 0 to 1. For a smooth surface, it approaches 1 and for the film with porous and rough surfaces, it decreases [4]. The β value for the carbon film coated electrode was less than that for the other electrodes. Generally, the porous or rough surface of a CE can enhance the catalytic ability of the film by providing a larger electrolyte-catalyst interface for the fast reduction of the tri-iodide ions in DSCs. In spite of the smaller value of β, the catalytic property of the lampblack with toner film was inferior compared with that of platinum as indicated by a high value of the charge transfer resistance of the carbon based dummy cell compared with that of the platinum based dummy cell. The R_{ct} for the carbon-electrolyte interface was 13.46 Ωcm^2, which is larger than 2.29 Ωcm^2 at the platinum-electrolyte interface. Moreover, the values of the R_{ct} of the dummy cell with only FTO (without catalyst) and the cell with toner film were about 46.2 x 10^5 Ωcm^2 and 5.32 x 10^5 Ωcm^2, respectively. The R_{ct} of the toner based dummy cell was extremely high compared with that of the platinum based dummy cell and the lampblack with toner based dummy cell, and it is comparable with the dummy cell prepared with only FTO (without catalyst). This indicates that the contribution of the toner powder (the binder) mixed with the lampblack for the reduction of tri-iodide ions in the working solar cells will be negligibly small. The portion of the toner in the composite film was 50% by weight, and the toner can serve as a good binder to hold the lampblack particles with each other and bind them firmly on the FTO-glass substrate used in CEs as we reported in our previous paper [9], however, the toner particles (without catalytic ability) can occupy significant space reducing electrolyte-catalyst (lampblack) interface area in the composite film based counter electrode and hinder the catalytic ability of the composite based DSCs.

CONCLUSIONS

The soybean oil lampblack comprised of both ordered and disordered forms of graphitic carbon and the lampblack based CE exhibited catalytic ability for the reduction of tri-iodide ions. The toner power mixed with the lampblack while fabricating CE did not exhibit significant catalytic activity in the reduction of the tri-iodide ions and it merely served as a binder. Though the efficiency of the dye-sensitized solar cells based on the soybean oil lampblack was slightly less than that of the platinum based solar cell, the soybean oil lampblack can be a low-cost alternative counter electrode material to replace expensive platinum used in DSCs.

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