



Development of Electrode Deposition Methods for Scale-up of Dye Sensitized Solar Cells

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Highlights:

- Various deposition methods for scaled-up of Dye Sensitized Solar Cell (DSSC).
- The doctor-blade method shows excellence performance for the scale-up process.
- A higher active area shows lower performance despite similar deposition method.
- Need a good electrical contact in interface of electrodes - conductive glass.
- The screen printing method shows the lowest resistance in that interface.

Abstract. This research studied the effect of electrode deposition method on the performance of dye-sensitized solar cells (DSSCs). Four deposition methods (bar coating, doctor blade coating, screen printing, and spray coating) were compared. For commercial production purposes, applicability of the methods to a scaled-up DSSC was studied. In order to minimize the production cost, commercial activated carbon was utilized as counter electrode. The experimental results showed that the doctor blade technique provided the best DSSC performance among the investigated methods. Based on the study of cell I-V characteristics, the photoelectric conversion efficiency of the DSSC with an electrode active area of 70 cm² was significantly lower than with 5 cm² despite using the same deposition method. The electrochemical characteristics of the cells were further studied using electrochemical impedance analysis.

Keywords: *dye-sensitized solar cells (DSSCs); carbon-based; electrode deposition methods; electrochemical characteristics; size scale-up, doctor blade method.*

1 Introduction

Over the past decades, dye-sensitized solar cells (DSSCs) have gained substantial interest as third-generation solar cells due to their simple assembly procedure, cost-effective fabrication, environmental friendliness, scalable materials, and flexibility in realization [1-3]. The history of DSSCs began in 1972 with the development of the chlorophyll-sensitized zinc oxide (ZnO) electrode [4]. In 1991, O'Regan and Grätzel built the first dye-sensitized nanocrystalline solar

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cells with photoelectric energy conversion reaching 7.1% and an incident photon to electrical current conversion efficiency of approximately 80% [5]. Thenceforth, DSSCs have become an attractive subject in solar energy cell research from both applied and fundamental perspectives. As reported in 2015, the photoelectric conversion efficiency of recently developed DSSCs can reach up to 14.2% using carboxy-anchor organic dye LEG4 as collaborative sensitizer to a silyl-anchor dye [6].

DSSCs utilize the photovoltaic effect of semiconductor materials to directly convert solar light to electric current. The basic components of a DSSC are a photoanode, a sensitizer, an electrolyte, and a counter electrode. The design of DSSCs currently involves a set of serial-stacked components: glass substrate, transparent conducting layer, TiO₂ nanoparticles, dye, electrolyte, and counter electrode, which are gasket sealed [7]. Under solar illumination, photoexcitation of the dye molecules results in injection of electrons from the ground state of the dye to its excited state. Electrons subsequently injected into the conduction band of TiO₂ leave the dye in an oxidized state [8]. Consecutively, the electrons are transported to the fluorine-doped tin dioxide (FTO) and migrate to an external load. Finally, the counter electrode collects the electrons from the external circuit and catalyzes a reduction reaction of the electrolyte (e.g. triiodide to iodide). The reduced components of the electrolyte thus regenerate the dye from an oxidized state back to its ground state [2,9,10]. Thus, ideally, the DSSCs generate electric power from solar light without suffering any permanent chemical transformation.

For commercialization of DSSCs, the fabrication procedure and cost are the main constraints. The noble metal platinum (Pt) is commonly used as counter electrode material in DSSCs due to its outstanding electrical conductivity and catalytic properties. However, platinum's high price and limited availability may hinder its practical application for large-scale production of DSSC devices [10-12]. Therefore, the pursuit of Pt-free or low-Pt electrocatalyst materials with equivalent performance as with Pt in terms of conductivity and electrocatalytic efficiency have been a persistent objective for industrialization of DSSCs. Carbonaceous materials have been proposed as ideal substitutes for Pt electrodes in large-scale production of DSSCs due to their abundance, excellent catalytic activity, electrical conductivity, and superior chemical stability [10]. It has been reported that carbonaceous materials show catalytic activity, supporting the reduction of triiodide to iodide. Their high porosity leads to abundant reduction sites and hence low charge transfer resistance [13]. Carbon black, carbon nanotubes, and other types of nanocarbon structures have been explored [14].

The electrode fabrication procedure influences the performance of DSSCs. Using different deposition techniques affects the particle size, the surface area, the morphology as well as the catalytic and electrochemical properties of the

electrode [15]. Electrochemical deposition, thermal decomposition, chemical reduction [16], spray deposition [17], spin coating, doctor blade coating, screen printing, physical vapor deposition, etc. have been explored. Nonetheless, limited guidelines are available to determine which is the best deposition method [18].

The advantages of the different deposition techniques need to consider type of material and substrate, specific application, and cost. In this paper, therefore, investigations of suitable electrode deposition techniques for the scaling-up of DSSCs are presented. Precursors prepared from paste of TiO_2 and commercial activated carbon powder were deposited on fluorine-doped tin oxide (FTO). Four deposition methods (bar coating, doctor blade coating, screen printing, and spray coating) were compared. The performance of the prepared DSSCs was studied by I-V characteristics and electrochemical impedance analysis. It was found that the doctor blade technique provided the best-performing DSSCs. Decreasing the photoelectric conversion efficiency by increasing the single active area is still a major drawback during scale-up.

2 Experimental Set-up

Working and counter electrodes were prepared from particles of TiO_2 and commercial activated carbon, respectively. Before being deposited, the FTO glass resistance was tested by using a multimeter to determine the conductive side. The counter electrode was prepared from 0.25 g carbon powder and 15 mL dimethylformamide. The carbon-coated counter electrode was heated in an electric furnace at 160 °C for 40 minutes.

The working electrode was prepared from paste of 1 g of TiO_2 powder and 30 ml of acetic acid deposited onto a conductive glass surface. Sintering of the working electrode was conducted at a temperature of 450 °C for 30 minutes. Preparation of the electrodes was done using four different methods, i.e. bar coating, doctor blade coating, spray coating, and screen printing, as illustrated in Figure 1. Both the working and counter electrodes were prepared on conductive glass with dimension of 2.5 cm x 2.5 cm and 10 cm x 7 cm, as shown in Figure 2.

Eosin Yellowish solution was prepared by dissolving the dye into ethanol. The solution composition was 0.138 g Eosin Yellowish in 20 mL ethanol. The electrolyte solution consisted of redox couple of iodine and iodide (I/I_3^-). Preparation of the electrolyte solution using potassium iodide as the iodine source was conducted based on the technique described in [19]. First, 6 g potassium iodide and 3 g iodine were mixed and stirred for 1 hour. KI solution was added to a solution of iodine-acetonitrile with an iodine concentration of 0.05 M. The prepared solution was stored in a sealed container to prevent evaporation.

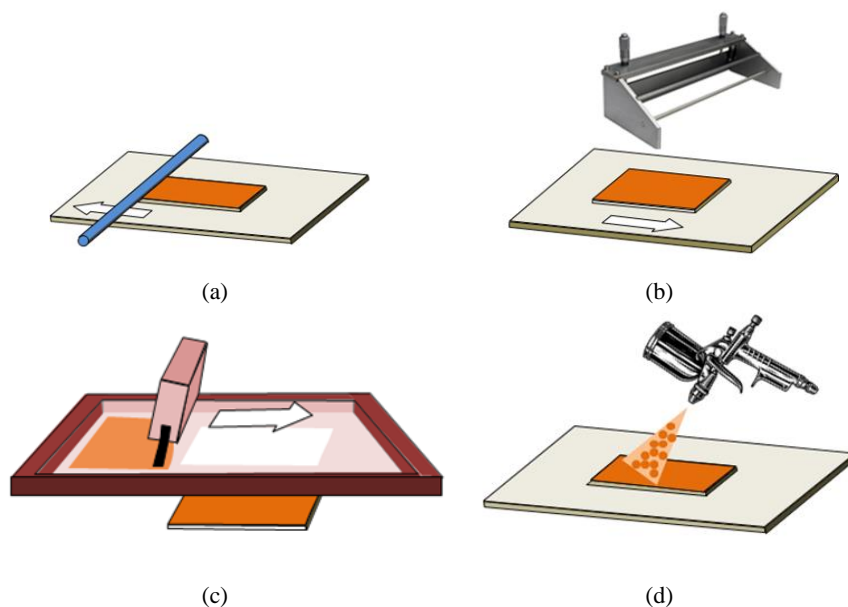


Figure 1 The methods used in this research: (a) bar coating, (b) doctor blade coating, (c) screen printing, and (d) spray coating.

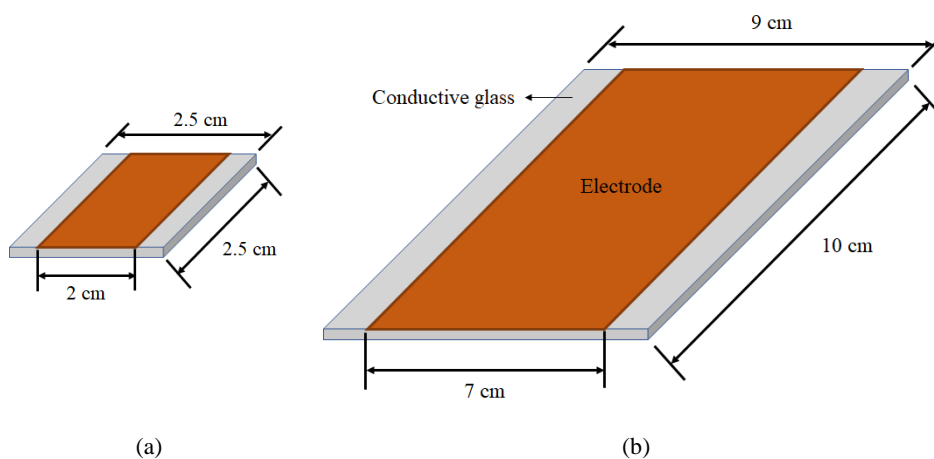


Figure 2 Schematic diagram of DSSCs with an electrode active area of (a) 5 cm^2 , and (b) 70 cm^2 .

The TiO_2 deposited working electrode was immersed in the dye solution to allow dye attachment. The dye was left to permeate and dry on the TiO_2 layer for 24 hours, after which the dye-coated working electrode was ready for the next stage. The working and counter electrodes were stacked to form a solar cell. The DSSC

was assembled into a sandwich-like structure by placing the dye-TiO₂-coated working electrode over the carbon-coated counter electrode [15]. Both electrodes were then clamped together using paper clips. Electrolyte solution was dropped into the gap between the electrodes using a pipette.

3 Results and Discussion

3.1 Morphology of Prepared Electrodes

Four methods, i.e. bar coating (BC), doctor blade coating (DBC), screen printing (SP), and spray coating (SC), were used to prepare a TiO₂ layer on transparent conductive glass. Meanwhile, carbon counter electrodes were prepared using the bar coating method. Thick slurry of both precursors of TiO₂ and activated carbon powders were utilized. The mass of the deposited precursor (before heat treatment) was controlled as similar as possible by measuring the mass of the bare conductive glass and the deposited glass.

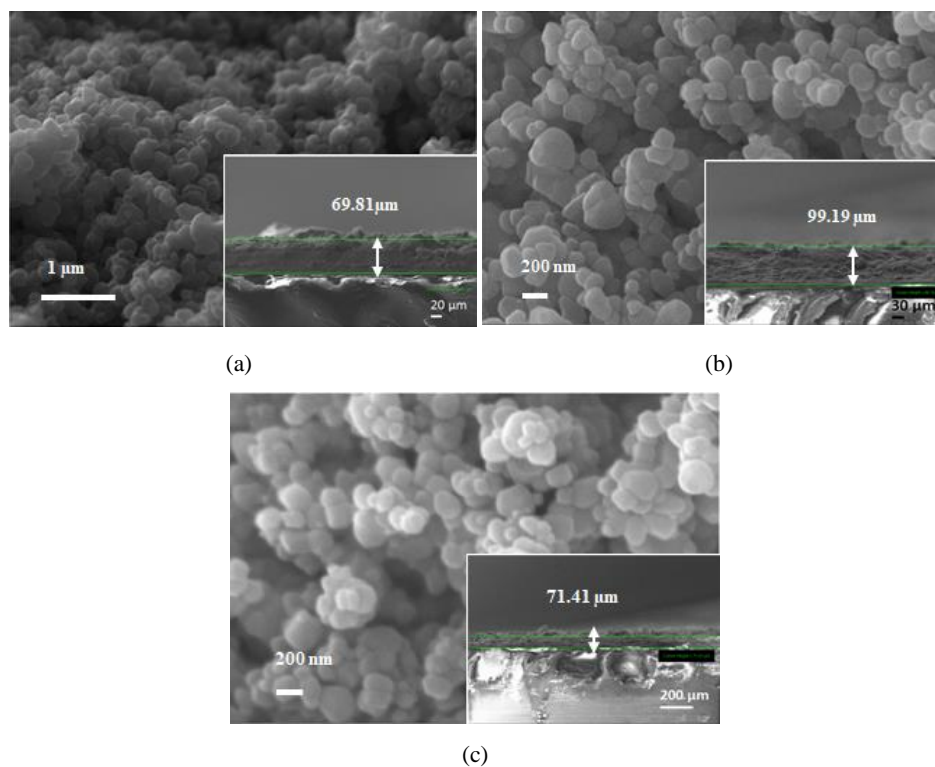


Figure 3 Typical scanning electron microscopy images of TiO₂ layers prepared using: (a) spray coating, (b) doctor blade coating, and (c) screen printing.

After deposition on the fluorine-doped tin oxide (FTO) glass and the heat treatment, the morphology of the deposited particles was studied using scanning electron microscopy (SEM). The cross-sectional SEM images in Figure 3 show that the TiO₂ layers have a thickness of 69 to 99 μm . Meanwhile, the carbon layers had a typical thickness of 210 μm , as can be seen in Figure 4. The surface images of the electrode show TiO₂ particles with the same size from the various deposition methods. This indicates that the deposition methods maintained the size of the precursor particles.

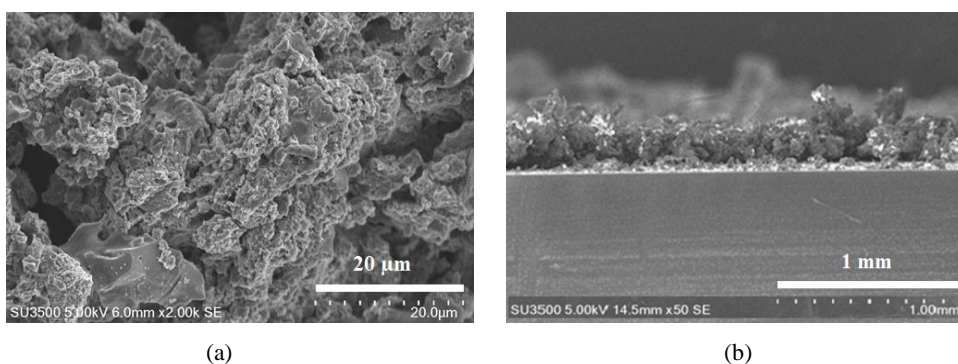


Figure 4 Typical scanning electron microscopy images of carbon layer prepared using bar coating: (a) surface (b) cross section.

3.2 Photovoltaic Performance

To study the performance of the DSSCs cells, the dye-immersed TiO₂ working electrode and carbon counter electrode were sandwich-stacked. A tungsten-halogen lamp (Philips QVF133 Halolite, 150 W) was used as light source. The typical emission spectrum of a tungsten halogen lamp is between 450 and 950 nm. Meanwhile, the effective light absorbance range of Eosin Y is 450 to 550 nm. The electrical current and voltage were measured using a multimeter under various load resistances.

All results showed that the prepared DSSCs had low photovoltaic performance. For comparison, Huynh, *et al.* could achieve an open circuit voltage (V_{OC}) of 0.77 V and a short circuit current density (I_{SC}) of 18.2 mA cm⁻² [20]. The experiment used Ruthenium 535 bis-TEA dye (N-719 Solaronix) instead of Eosin Y. It is known that the ruthenium dye family is the most suitable dye for TiO₂. Using Eosin Y, Wang, *et al.* developed DSSCs with a V_{OC} in the range of 0.451 to 0.813 V and an I_{SC} of about 5 mA cm⁻² [21]. Therefore, for the small-sized cells with an active area of 5 cm², our experiments using the doctor blade technique could obtain a sufficiently good V_{OC} of 0.32 V but a low I_{SC} of 0.036 mA cm⁻².

For the large cells, however, the V_{OC} and the photoelectric conversion efficiency (η) decreased significantly to approximately one fifth of the small ones. It is assumed that this low performance is due to the detachment of dye from the TiO_2 particles and poor adhesion of the TiO_2 to the conductive glass. The conductive glass suffers from deformation during the heat treatment at 450 °C, causing cracking and peeling of the TiO_2 layer. It has also been observed that the Eosin Y dye had been washed out by the electrolyte after experiment.

Figure 5 shows the best results for the current-voltage (I-V) characteristics of the large cells with an active area of 70 cm² prepared by using various deposition methods. The performance parameters of the prepared DSSCs are shown in Table 1.

Calculation of the solar conversion efficiency of the DSSCs was based on a formula stated elsewhere [22]. The experimental results showed the same trend (both in the small-size and large-size cells), where the doctor blade method provided the best performance. The series (R_S) and shunt (R_{SH}) resistances of the cells were then calculated from the reverse slop in I_{SC} and V_{OC} , respectively. Ideal solar cells should have series resistance close to zero and an extremely high shunt resistance [23]. The cell prepared using the doctor blade method had the lowest R_S (126 Ω cm²). Meanwhile, screen printing and spray coating resulted in a significantly higher R_S (616 and 2.66 x 10⁵ Ω cm²), respectively.

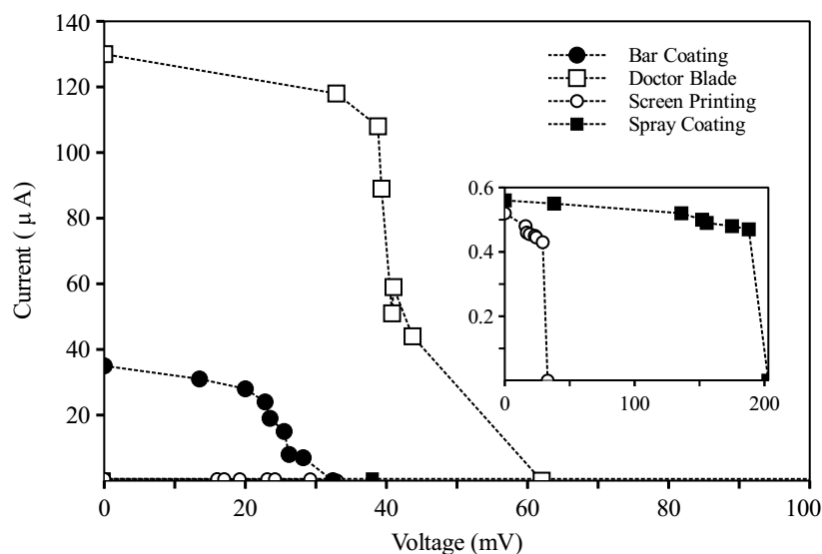


Figure 5 Effect of electrode coating method on I-V curve characteristics of dye-sensitized solar cells with an electrode active area of 70 cm².

Table 1 Performances parameters of prepared dye-sensitized solar cells.

Method	V_{oc} (mV)		I_{sc} (mA)		P_{max} (mW)		η (%)	
	S	B	S	B	S	B	S	B
Bar coating	165	32.4	0.04	0.04	2.94	0.54	0.08	0.014
Doctor blade coating	322	62	0.18	0.13	30.44	4.19	0.75	0.110
Screen printing	-	33	-	0.0052	-	0.088	-	0.002
Spray coating	-	203	-	0.0056	-	0.012	-	0.0003

Notes: S = small-size cells of 5 cm², B = large-size cells of 70 cm², V_{oc} = open circuit potential, I_{sc} = short circuit current, P_{max} = maximum power, η = conversion efficiency of cells

The shunt resistance of the DSSCs prepared using screen printing and spray coating was higher (2.25×10^4 and $4.08 \times 10^3 \Omega \text{ cm}^2$) compared to 56 and 28 $\Omega \text{ cm}^2$ for bar coating and doctor blade coating respectively.

R_s is influenced by the resistance for electron transport in the TCO substrate as well as at the electrolyte/counter electrode interface and Nernst diffusion impedance in the electrolyte [24]. Meanwhile, R_{SH} is attributed to the low electron transfer rate from the TiO₂ back to the electrolyte. It is affected by manufacturing impurities or defects. To elaborate the main factors that affect the resistance, an electrochemical impedance spectroscopy (EIS) analysis was performed on the DSSCs with an active area of 70 cm² using a Gamry Reference 3000 instrument.

3.3 Analysis of Electrochemical Impedance Characteristics

The EIS analysis was conducted in the frequency range of 10 mHz to 100 kHz under illumination. Figure 6 shows a Nyquist diagram of the prepared DSSCs. Interpretation of the diagram requires a suitable equivalent circuit model that simulates the physical and chemical processes in the DSSCs. We adapted the transmission line model suggested by Fabregat-Santiago, *et al.* [24], as shown in Figure 7.

The transmission line model represents phenomena of the series resistances of TCO (R_s), charge transport in TiO₂, and the TiO₂/electrolyte interface (Z_1), recombination of electron (Z_2), diffusion in electrolyte (Z_3), and charge transport in the carbon layer interface (Z_4). The impedance of porous TiO₂ electrode Z_1 can be modeled as a series of Randles circuits. In this work, a single Randles circuit was more representative than a series one. Z_1 consists of the resistance in TCO/TiO₂ (R_1 in series with a parallel circuit of R_{11} and a CPE). The impedance of CPE consists of capacitance Y_{11} and exponent α_{11} as described in Eq. (1). Meanwhile, the impedance of Z_2 represents the recombination of electrons on the surface of TCO that is imperfectly covered by a porous layer of TiO₂.

$$Z_i = (1/Y_i) / (j \omega)^\alpha \quad (1)$$

Diffusion resistance of active species, i.e. I^- and I_3^- , is represented by Z_3 . Simultaneous chemical reactions and diffusion mass transfer as Gerischer impedance is more suitable than the semi-infinite diffusion model of Warburg. Impedance Z_3 consists of a parallel circuit of capacitance Y_3 and effective transfer rate K_3 . Electrolyte regeneration in the porous activated carbon layer is represented by Z_4 , consisting of a parallel circuit of the resistance (R_4) and a constant phase element (capacitance Y_4 with an exponent of α_4).

Based on the EIS analysis, the DSSCs prepared using screen printing and spray coating showed high series resistance. This is consistent with the I-V performance analysis. For the screen-printed DSSC, an R_{11} of 1937Ω indicates high charge transfer resistance at the TiO_2 /electrolyte interface.

The low electron recombination resistance R_2 of 1.99Ω may indicate a high recombination rate in the TCO layer. In contrast, the R_{SH} calculated from the I-V curve is high. A very low output current of the DSSCs may cause this calculation of the R_{SH} from the curve to become inaccurate. This high value of R_{11} and low value of R_2 decreased the performance of the screen-printed DSSCs.

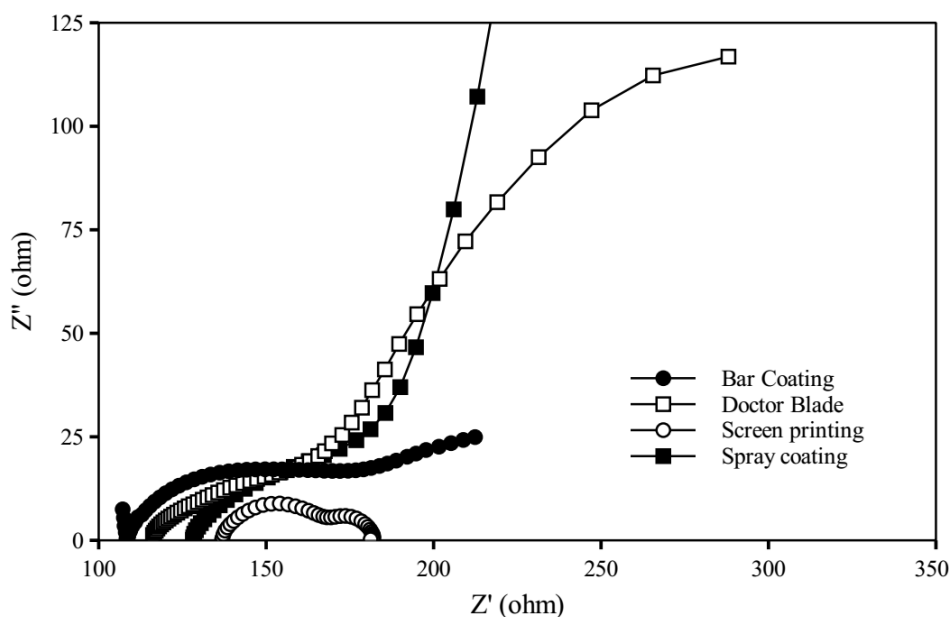


Figure 6 Electrochemical impedance spectroscopy Nyquist plot of dye-sensitized solar cells with an electrode active area of 70 cm^2 .

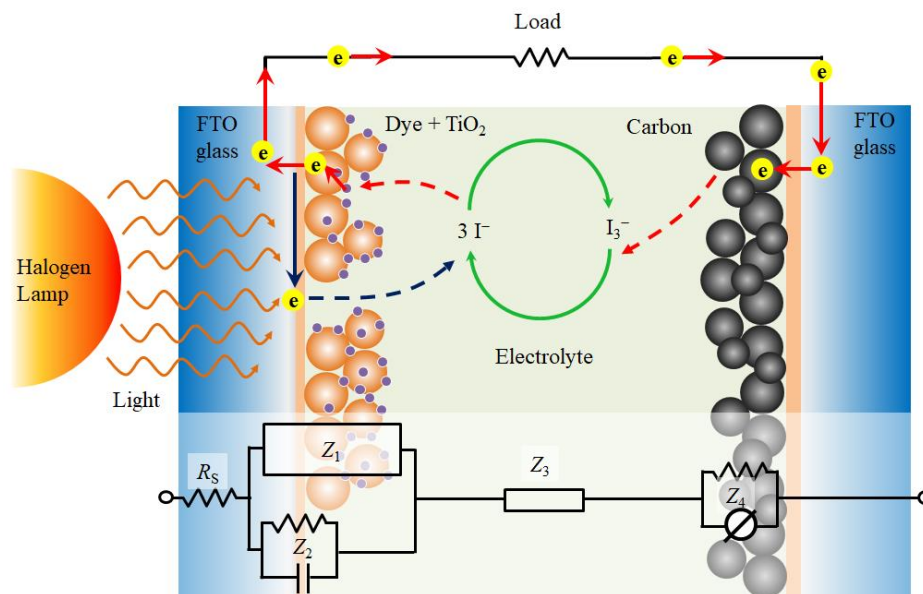


Figure 7 Equivalent circuit of the prepared dye-sensitized solar cells.

For the spray-coated DSSCs, the resistance R_1 of 1794 Ω indicates high charge transfer resistance at the TCO/TiO₂ interface. Further, we suppose that the low output current of the DSSCs was caused by detachment of dye from the TiO₂ surface, which was more noticeable here than in the other cells. Reaction of the dye with electrolyte therefore became intense, as indicated by the high transfer rate K_3 of 18430 s⁻¹.

Doctor-blade coated DSSCs showed a high R_2 resistance of 23890 Ω , suggesting a low recombination rate of the electrons on the TCO surface. As a result, the electrons can be effectively transferred from the working electrode to the counter electrode through an external load.

Electron loss due to the recombination reaction is minimized, reflected in a higher current in the I-V curve. Further, the R_2 of the doctor-blade coated DSSCs was much higher than that of the bar-coated cells (23890 Ω to 35.96 Ω), while the R_1 was lower (15.4 Ω to 310.5 Ω). This indicates that TiO₂ can be attached better to TCO using the doctor blade coating than the bar coating method, in accordance with the cross-sectional SEM images.

We assume that pressure should be applied to the TiO₂ layer to achieve a low resistance of the TCO/TiO₂ interface. In the screen printing method, where pressure is applied during coating, the resistance R_1 was much lower, i.e. 2.08×10^{-3} Ω . Hyunh, *et al.* pressed the TiO₂ layer with a flat glass at 50 kgf cm⁻² for 30

s [20]. This compacted layer on the TCO reduces the series resistance, thus increasing the performance of the DSSCs [23].

Table 2 Equivalent circuit parameters of prepared dye-sensitized solar cells.

Parameters	Unit	Method			
		Bar coating	Doctor blade coating	Screen printing	Spray coating
R_s	Ω	115.3	108.7	123.7	132.1
R_1	Ω	310.5	15.4	2.08×10^{-3}	1794
R_{11}	Ω	1.46×10^{-4}	38.0	1937	7.68×10^{-4}
Y_{11}	S. s^α	3.41×10^{-3}	1.17×10^{-4}	1.19×10^{-9}	4.93×10^{-3}
α_{11}	-	1.23×10^{-2}	0.56	0.93	3.77×10^{-4}
R_2	Ω	35.96	23890	1.99	53.55
C_2	F	1.71×10^{-4}	2.41×10^{-7}	0.10	3.32×10^{-4}
Y_3	S. $s^{0.5}$	0.59	6.39×10^{-3}	1.11×10^{-2}	0.55
K_3	s^{-1}	5.1	2.71×10^{-3}	40.5	18430
R_4	Ω	108.2	62.2	21.8	456.3
Y_4	S. s^α	1.60×10^{-3}	2.12×10^{-3}	3.39×10^{-5}	1.92×10^{-4}
α_4	-	1	0.42	0.78	0.80

4 Conclusions

We conclude that doctor blade coating is the most suitable method to prepare scaled-up working electrodes for DSSCs from a thick paste of TiO_2 precursor compared to the other studied methods. The performance of the scaled-up DSSCs with an electrode active area of 70 cm^2 was still significantly lower than that of the DSSCs with an electrode active area of 5 cm^2 due to manufacturing defects.

We also found that compared to the doctor blade method, the screen-printing and spray coating methods provided low TCO/ TiO_2 resistance and TiO_2 /electrolyte resistance, respectively. Therefore, a combined deposition method to increase DSSC performance needs to be explored in a future research.

Acknowledgements

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