

Finely tuning electrolytes and photoanodes in aqueous solar cells by experimental design



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ARTICLE INFO

Keywords:

Dye-sensitized solar cell
Aqueous electrolyte
Sustainability
Design of experiments
Multivariate approach
Electrode/electrolyte interface

ABSTRACT

If opportunely developed and optimized, aqueous dye-sensitized solar cells can be considered a truly low impact photovoltaic device, with no toxic components. Here we report the use of design of experiments as a useful chemometric technique for the concurrent investigation of a series of experimental factors that directly influence the proper operation of these photoelectrochemical cells. Results obtained enlighten that a solid mathematical-statistical approach is fundamental to support the researchers and effectively drive the experiments towards the achievements of optimal operating conditions of any new energy device, thus bypassing the energy/time consuming steps of traditional univariate one-factor-at-a-time method.

1. Introduction

The introduction of aqueous electrolytes is contributing to the re-birth of dye-sensitized solar cells (DSSCs), the low-impact photoelectrochemical device for the conversion of solar energy into electricity (Bella et al., 2015). Aqueous DSSCs represent a truly sustainable and very limited environmental impact technology, which might successfully pinpoint the concept of artificial photosynthesis often been evoked within the scientific community in the last two decades (Bella et al., 2017, 2018; Click et al., 2017; Imperiyka et al., 2014; McConnell et al., 2010; Yang et al., 2010).

Several research groups worldwide recently focused their efforts towards the investigation of a series of new factors, such as photoelectrode wetting (Dong et al., 2014; Galliano et al., 2017), novel sensitizers (Leandri et al., 2014; Li et al., 2017; Lin et al., 2015), the solubility and the diffusion behavior of various redox pairs in water (Daeneke et al., 2012; Xiang et al., 2013; Yang et al., 2015), the cathodic electrocatalytic reaction (Ellis et al., 2016). Clearly, in a new system (like the aqueous DSSC), the possibility to simultaneously study several of these experimental factors at the same time, bypassing the traditional one-factor-at-a-time (OFAT) process, would permit to effectively drive research towards well-defined paths supported by a solid

mathematical/statistical justification. In this respect, despite being scarcely adopted by the energy-related research community (Gianotti et al., 2014; Miccoli et al., 2016), multivariate chemometrics, such as design of experiments (DoE) (Kennard and Stone, 1969), would represent a key-enabling method.

In this work, we propose a DoE for the simultaneous investigation of the characteristics of photoanode, electrolyte and photoanode/electrolyte interface of aqueous DSSCs. A simple experimental matrix based on 20 photoelectrochemical cells enables to comprehensively weight each of the experimental factors involved, and to easily identify the best conditions to maximize sunlight conversion efficiency. Amongst the powerful and intriguing aspects of the adopted approach, the ability to find two performance maximization strategies must be mentioned, which is the fundamental achievement of this work.

2. Experimental

Sodium iodide (NaI), iodine (I₂), chenodeoxycholic acid (CDCA), ethanol, acetone, tert-butanol (*t*-BuOH) and acetonitrile (ACN) were purchased from Sigma-Aldrich. Deionized water (DI-H₂O, 18 MΩ cm⁻¹ at 25 °C) was obtained by Direct-Q 3 UV Water Purification System (Millipore). Sensitizing dye 2-[[4-(2,2-diphenylethenyl)phenyl]-

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<https://doi.org/10.1016/j.solener.2018.02.009>

Received 19 November 2017; Received in revised form 1 February 2018; Accepted 2 February 2018

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Table 1

DoE model matrix consisting of 20 experiments, useful to investigate the three selected experimental variables (x_1 , x_2 and x_3). The measured responses for J_{sc} , V_{oc} , FF and PCE are also shown.

Exp.	CDCA:Dye ratio [x_1]	[Iodine] (x_2 , mM)	[Iodide] (x_3 , M)	J_{sc} (mA cm^{-2})	V_{oc} (mV)	FF (%)	PCE (%)
N1	18:1	10	1.0	1.48	564	54.7	0.46
N2	50:1	10	1.0	2.21	607	54.7	0.73
N3	18:1	30	1.0	0.92	476	54.9	0.24
N4	50:1	30	1.0	1.71	540	59.5	0.55
N5	18:1	10	5.0	3.55	528	56.0	1.05
N6	50:1	10	5.0	1.73	532	44.2	0.41
N7	18:1	30	5.0	2.67	450	58.3	0.70
N8	50:1	30	5.0	2.27	465	45.6	0.48
N9	34:1	20	3.0	0.70	425	53.4	0.17
N10	34:1	20	3.0	1.35	453	56.9	0.35
N11	18:1	10	1.0	1.56	549	55.6	0.48
N12	50:1	10	1.0	2.57	613	57.1	0.90
N13	18:1	30	1.0	1.08	494	57.3	0.30
N14	50:1	30	1.0	1.88	557	59.7	0.63
N15	18:1	10	5.0	3.47	502	56.1	0.98
N16	50:1	10	5.0	1.72	534	44.0	0.42
N17	18:1	30	5.0	2.82	434	58.2	0.71
N18	50:1	30	5.0	2.58	474	44.6	0.55
N19	34:1	20	3.0	1.12	463	56.2	0.29
N20	34:1	20	3.0	1.44	468	57.8	0.39

1,2,3,3a,4,8b-hexahydrocyclopento[b]indole-7-yl)methylidene]-cyanoacetic acid (D131) was purchased from Inabata Europe S.A. FTO-glass plates (sheet resistance $7 \Omega sq^{-1}$, purchased from Solaronix) were cut into $2 cm \times 1.5 cm$ sheets and used as substrates for the fabrication of both photoanodes and counter electrodes.

FTO covered glasses were rinsed in mixed acetone/ethanol in an ultrasonic bath for 10 min; solvent traces were removed by flash evaporation at $450^\circ C$ on a hotplate. Front electrodes were prepared by depositing a single layer of porous TiO_2 on top of the conductive substrate by means of a manual screen printer with a 43 T mesh frame. After deposition of the paste (18NR-T, Dyesol) and 20 min rest to let it bed thoroughly, the TiO_2 layer was dried at $80^\circ C$ for 20 min; finally, it was sintered increasing the temperature up to $480^\circ C$ in 45 min. The

fabricated photoanodes had a thickness of $\approx 6 \mu m$ and active area of $0.25 cm^2$. They were finally reactivated by heating at $450^\circ C$ for 20 min and, subsequently, soaked into a D131 dye solution (0.50 mM in *t*-BuOH:ACN 1:1); CDCA was added to the dye solution as coadsorbent (its concentration represents one of the factors investigated in this work). Dipping in dye solutions was carried out at $22^\circ C$ for 5 h under dark conditions and shaking in a Buchi Syncore platform equipped with a cooling plate. After dye loading, photoanodes were washed in acetone to remove residual dye not specifically adsorbed onto the TiO_2 layer.

As regards the preparation of counter electrodes, FTO conductive glasses were platinized by spreading a H_2PtCl_6 5.0 mM solution on the plate surface and heating up to $400^\circ C$.

Photoanodes were faced to the counter electrodes exploiting Surlyn[®] thermoplastic frames (internal area $0.6 cm \times 0.6 cm$) as spacers (60 μm thick), taking care of the overlapping of the active areas. All of these components were assembled by hot pressing (20 s) at $110^\circ C$. The electrolyte solution (consisting of NaI and I_2 dissolved in water) was injected by vacuum backfilling process through a hole in the Surlyn[®] frame, which was then sealed by a commercial epoxy glue. The amounts of NaI and I_2 represent relevant factors in the aqueous DSSC field, thus they are investigated in this work.

Photovoltaic performances were evaluated recording three consecutive *J-V* curves on a Keithley 2420 Source Measure Unit. Cells were irradiated under simulated 1 sun light intensity ($100 mW cm^{-2}$, AM 1.5G) after calibration by silicon diode, as it is typically done in the DSSC framework (Bella et al., 2017; Fan et al., 2017; Liu et al., 2017; Maçaira et al., 2017; Mengal et al., 2017). Electrochemical impedance spectroscopy (EIS) data were recorded using a potentiostat (CH Instruments Inc., Model 680) in the frequency range between 10 kHz and 0.1 Hz. The amplitude of the AC signal was 10 mV. Spectra were recorded under dark conditions at applied DC potentials equal to the previously measured V_{oc} values under 1 sun (Buraidah et al., 2017; Mohan et al., 2017; Paušová et al., 2017; Subramanian et al., 2017; Vekariya et al., 2017).

3. Results and discussion

An experimental design was compiled to simultaneously investigate the effect of two key parameters, such as the concentration of the redox

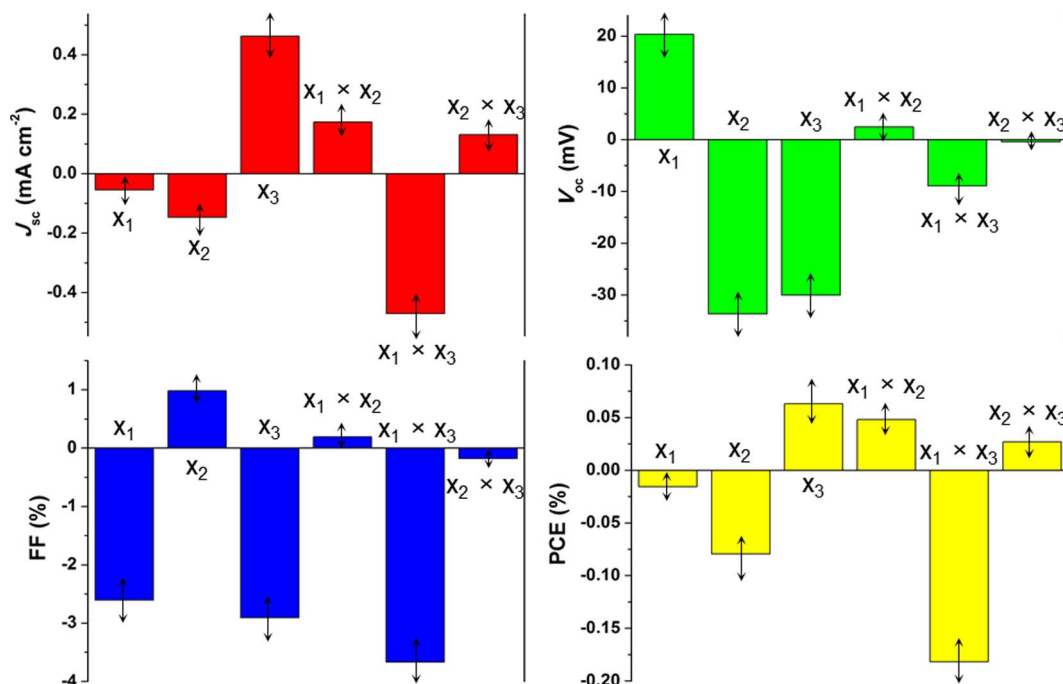


Fig. 1. Coefficient plot for the DoE. Each plot displays the coefficients, when changing from level 0 to a higher level, for the selected response (i.e., the photovoltaic parameters).

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