



# Highly transparent metal selenide counter electrodes for bifacial dye-sensitized solar cells



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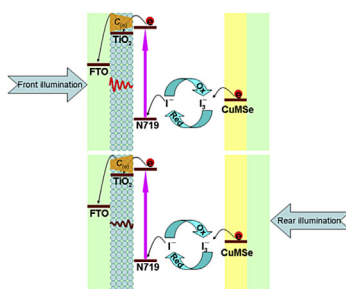
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## HIGHLIGHTS

- Transparent CuMSe (M = Fe, Co) alloys are synthesized by a hydrothermal method.
- The transparent CuMSe are used as CE catalysts for bifacial DSSCs.
- The CuMSe CEs have superior optical transparencies and activity activities.
- Front and rear efficiencies of 7.81% and 5.95% are recorded on the optimal DSSC.

## GRAPHICAL ABSTRACT



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## ABSTRACT

Creation of transparent counter electrode (CE) electrocatalysts for bifacial dye-sensitized solar cells (DSSCs) is a persistent objective for reducing cost of photovoltaic conversion. We present here the experimental realization of highly transparent CuSe CEs by a mild solution method for liquid-junction bifacial DSSCs. The resultant CuSe CEs show superior electrocatalytic activity toward  $I_3^-$  reduction reaction. By optimizing the pH values in synthesizing CuSe electrodes, the maximal front efficiency of 6.21% and rear efficiency of 4.72% are recorded on the corresponding bifacial DSSC. Both catalytic activity and photovoltaic performances can be further elevated by alloying CuSe with Co or Fe, yielding promising efficiencies of 7.81% and 5.38% under front and rear irradiations, respectively.

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## 1. Introduction

Dye-sensitized solar cell (DSSC) [1–3], a photoelectrochemical device of converting solar energy into electricity by complicated photoelectrochemical reactions, has been considered as a promising solution to energy crisis and environmental pollution. The

classical DSSC can realize electricity output by irradiating the  $TiO_2$  photoanode. It has been demonstrated that bifacial DSSC platform is a new emerging architecture to reduce the cost of photovoltaic conversion [4,5]. However, traditional Pt CE has metallic luster, leading to a high reflection for incident light from rear side. In the fashion, the creation of highly transparent CEs without reducing catalytic activity seems to be crucial for realizing electricity generation from either front or rear side. Graphene has been employed in transparent CE [6], one of the drawbacks is that the limited catalytic activity to redox electrolyte has dragged the cell efficiency.

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Conducting polymers such as polyaniline is colorful but transparent in visible-light region [7–9], therefore, it is also applicable for bifacial DSSCs. A remaining issue for transparent polyaniline electrode is its nature of organic semiconductor, allowing for poor electron collection from external circuit. Recently, many efforts have been placed on integrating graphene and conducting polymers to combine the good electron-conducting ability of graphene and excellent catalytic activity of conducting polymer into a single CE [10–12]. However, the reduced optical transmission is not beneficial to the enhancement of rear efficiency.

Previously, we have developed a series of metal selenide (MSe, M = Fe, Co, Ni, Ru, Mo) CEs by hydrothermal or electrodeposition techniques [13–18]. Due to the high optical transparency and catalytic activity, the DSSC performances are markedly enhanced in comparison with pristine Pt electrode based device. Moreover, the long-term stability is also increased, arising from the competitive dissolution reaction of incorporated transition metals [19,20]. To explore other cost-effective and efficient CE electrocatalysts for bifacial DSSC platforms, here we would like to create CuMSe (M = Co, Fe) CEs by a mild solution method. The dependences of synthesis conditions and M species on electrochemical behaviors of CEs and photovoltaic performances of their bifacial DSSCs are studied to optimize the processes. The preliminary results demonstrate that the electrochemical and photovoltaic performances of CuMSe CEs and corresponding devices have been markedly elevated, arising from electron-enriched surface as well as gigantic active sites for electrolyte reduction.

## 2. Experimental

### 2.1. Preparation of transparent CuSe CEs

0.02 g of Se ultrapowers and 0.0216 g of  $\text{CuCl}_2 \cdot 2\text{H}_2\text{O}$  were dissolved in 10 mL of deionized water. When being transferred into a 50 mL Teflon-lined autoclave, 2 mL of  $\text{N}_2\text{H}_4 \cdot \text{H}_2\text{O}$  (85 wt%) was dropped into the mixture. The total volume of reagent was controlled to 35 mL by adding deionized water, and the pH was adjusted to 8, 9, 10, or 11 using HCl or  $\text{NH}_3 \cdot \text{H}_2\text{O}$  aqueous solution. Before hydrothermal reaction at 120 °C for 12 h, the freshly rinsed FTO glass substrate with a sheet resistance of  $12 \Omega \text{ sq}^{-1}$  (purchased from Hartford Glass Co., USA) was immersed in the solution with conductive surface downward. Finally, the FTO glass supported CuSe CEs were thoroughly rinsed with deionized water and air-dried.

### 2.2. Preparation of transparent CuMSe (M = Co, Fe) CEs

The synthesis procedures for FTO glass supported CuMSe were similar to that for CuSe electrodes. The dosages of Se ultrapowers,  $\text{CuCl}_2 \cdot 2\text{H}_2\text{O}$ , and  $\text{CoCl}_2 \cdot 6\text{H}_2\text{O}$  for synthesizing CuCoSe electrode were 0.02 g, 0.0108 g, and 0.015 g, respectively. However, the dosages of Se ultrapowers,  $\text{CuCl}_2 \cdot 2\text{H}_2\text{O}$ , and  $\text{FeSO}_4 \cdot 7\text{H}_2\text{O}$  for synthesizing CuFeSe electrode were 0.02 g, 0.0108 g, and 0.0284 g, respectively. To realize the reaction, the pH of reagent was adjusted to 10 using  $\text{NH}_3 \cdot \text{H}_2\text{O}$  aqueous solution.

### 2.3. Solar cell assembly and measurements

Mesoporous  $\text{TiO}_2$  film with a thickness of 10  $\mu\text{m}$  and active area of  $0.25 \text{ cm}^2$  synthesized according to our previous report was utilized as a photoanode [21]. After being sensitized with 0.50 mM N719 dye (DYESOL LTD) in ethanol solution for 24 h, the bifacial DSSC device was fabricated by sandwiching redox electrolyte having  $\text{I}^-/\text{I}_3^-$  redox couples into  $\text{TiO}_2$  anode/transparent CE architecture. A surllyn film with a thickness of 30  $\mu\text{m}$  was used to seal the

solar cell under hot-pressing.

Photocurrent density-voltage (*J-V*) curves of the bifacial DSSCs were measured under  $100 \text{ mW cm}^{-2}$  from a 100 W Xenon arc lamp (XQ-500 W) by irradiating either front or rear sides. The active area of the solar cell was controlled at  $0.25 \text{ cm}^2$ . A black mask with an aperture area of around  $0.25 \text{ cm}^2$  was applied on the top surface of DSSC devices to avoid stray light. Each curve was repeatedly measured for at least five times to control the experimental error within  $\pm 5\%$ . The EIS spectra of DSSCs were determined at an ac amplitude of 10 mV, and the corresponding electrochemical data were extracted by fitting the EIS plotted with Z-view software.

### 2.4. Electrochemical characterizations

Cyclic voltammetry (CV) curves of the CEs were recorded in a traditional three electrode system at a scan rate of  $50 \text{ mV s}^{-1}$ . Prior to measurement, the supporting electrolyte having  $\text{I}^-/\text{I}_3^-$  couples was degassed by bubbling  $\text{N}_2$  gas for 5 min. Tafel polarization measurements were performed in a symmetric cell consisting of CE|redox electrolyte|CE at a scan rate of  $10 \text{ mV s}^{-1}$ .

### 2.5. Other characterizations

The morphologies of the resultant CEs were observed on an S4800 scanning electron microscope (SEM) and a JEM2010 transmission electron microscopy (TEM). The optical transparencies of resultant CE electrocatalyst were recorded on a UV-vis spectrophotometer using bared FTO glass as a benchmark. X-ray diffraction (XRD) patterns of the resultant CEs were recorded on a Philips X-ray powder diffractometer with  $\text{Cu K}\alpha$  radiation. XPS spectra were determined on a RBD upgraded PHI-5000C ESCA system (Perkin Elmer) with  $\text{Mg K}\alpha$  radiation ( $h\nu = 1253.6 \text{ eV}$ ). The actual compositions of the resultant metal selenide CEs were confirmed by inductively coupled plasma-atomic emission spectra (ICP-AES).

## 3. Results and discussion

Fig. 1 shows the top-view SEM images of resultant CuCoSe and CuFeSe CEs. At the first glance, the CuCoSe in Fig. 1a has a low coverage on FTO glass which may be attributed to the high optical transparency. Deep examination in Fig. 1b reveals that the CuCoSe electrocatalysts are aggregations. However, the FTO substrate has been almost covered by CuFeSe catalyst, as shown in Fig. 1c,d. The compositions were determined to be 1.000:0.82:3.51 and 1.000:0.98:3.18 for CuCoSe and CuFeSe, respectively.

In order to better characterize the morphologies of CuCoSe and CuFeSe electrocatalysts, the ultrafine powders have been collected from the Teflon-lined autoclave instead of scraping from FTO glass. Fig. 2a reveals that the resultant CuCoSe aggregations comprise of graphene-like nanosheets with loose structure. This extraordinary architecture is beneficial to  $\text{I}^-/\text{I}_3^-$  absorption and therefore  $\text{I}_3^-$  reduction reaction [22]. As shown in Fig. 2b, the CuFeSe catalyst has bulky stacking, reducing the specific surface area and therefore active sites for catalyzing  $\text{I}_3^-$  to  $\text{I}^-$  species.

The compositions of resultant CE materials are important in impacting the electrocatalytic activity and therefore cell performances. By addressing this issue, we have characterized the CuCoSe and CuFeSe CEs by XRD patterns and XPS spectra, as shown in Fig. 3. As a reference, the corresponding curves for the CuSe synthesized at pH of 10 have also been provided. Apparently, the as-synthesized Cu–Se at pH of 10 includes CuSe (PDF-# 02-0487) and  $\text{CuSe}_2$  (PDF-# 12-0115) species, and the XPS analysis reveals the atomic Cu/Se ratio is 1:2.16, closing to 1:2. In this fashion, the resultant Cu–Se synthesized at pH of 10 can be expressed as  $\text{CuSe}_2$ . The diffraction peaks of CuSe and  $\text{FeSe}_2$  (PDF-# 65-1455) can be detected in the

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