First application of benzotriazole as a dye additive to dye-sensitized solar cells: electrochemical device characterization

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Abstract
Dye-sensitized solar cell, known as an emergent technology, has been attracted attention around the world. It has been easily produced by a mesoporous semiconductor oxide (SO), with a dye. On the other hand, some unexpected reactions as the back-electron recombination, reducing the solar cell efficiency. The presence of some molecules to act as an oxide passivation might rise up the collection reaction rate, and enhance the photoelectrochemical solar system parameters. The molecule used as a dye additive, must effortlessly adsorb in SO and potentiate the dye deaggregation. This work aims to study the benzotriazole (BTAH) insertion in DSSC with TiO2, evaluating its capability as a passivator molecule in such PV technology. The electrochemical results showed better electrochemical energy conversion efficiency with lower resistance with BTAH at 2.0 mg mL−1, achieving a $j_{sc} = 9.92 \pm 0.26$, $V_{oc} = 0.770 \pm 0.022$, FF = 0.557 ± 0.014, $\eta = 4.26 \pm 0.13$ with superior electron lifetime and amazing recombination prevention.

Keywords PV system · BTAH · IMPS · IMVS · DSSC

Introduction
The use of semiconductor oxides with solar incidence is a promising technology to energy conversion [1–3]. Recent application of chenodeoxycholic acid-substituted dyes, in the third generation of dye-sensitized solar cells (DSSCs) raised up a new study area in PV systems [4]. The capability of suppressing dye-dye interactions and anchoring in the oxide surface become an excellent alternative in DSSCs development.

Dye-sensitized solar cells are made by the use of an electron transport material on transparent conductor oxide (TCO), acting as a scaffold material. There is also the presence of a dye, a photosensitive molecule that inject electron by solar irradiation, providing a charge flow responsible for the solar energy conversion [1–3].

Simply, the solar cell operating is based on the dye ability to inject electrons in the conduction band of scaffold oxide, usually TiO2 [5], ZnO [6], and SnO2 [7] with solar energy incidence. The injected electrons, moves through a TCO to a cathode catalyst, generating a charge flow as demonstrated in Reaction 1, (where SO is the semiconductor oxide).

$$SO (e^-) + \text{Cathode} \rightarrow SO + \text{Cathode} (e^-) \quad (\text{Reaction 1})$$

$$SO (e^-) + \text{electrolyte}^+ \rightarrow SO + \text{Electrolyte} \quad (\text{Reaction 2})$$

The intermediation of redox species is provided by an electrolyte that has been able to capture the electron in the cathode, reducing the dye [8, 9]. However, another reaction has been able to occur in the oxide/dye/electrolyte interface, the back-electron recombination (Reaction 2) [5, 10].

Suppressing the oxide-electrolyte recombination is one of the most studied ways to produce DSSC with improved photoelectrochemical parameters [11]. From this standpoint, many technologies have been used to reduce Reaction 2: push–pull dyes [12], using mix oxides [13, 14], and electrolyte additives [15]. Recently, Rodrigues and co-workers, [11]

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applied the poly (4-vinyl pyridine), observing a TiO₂ surface passivation, able to suppress the back-electron recombination and increasing the current, even at low dye loading [16, 17]. This related study shows that is possible to use organic additives in dye solar cells, such as benzotriazole (BTAH).

Benzotriazole is used as an effective inhibitor for several metal alloys and demonstrates high solubility in many aqueous media, good thermal stability, and has been able to chemical adsorbed in metal surface, due to nitrogen presence. In other words, some impressive advantages over the use of chenodeoxycholic acid. Furthermore, the equilibrium involving BTAH⁻ and BTAH²⁻ can contribute to physisorption and modulation of a space charge layer in a semiconductor/electrolyte interface [15, 16].

With a dye, BTAH might act efficiently avoiding dye aggregation, and shifting the oxide fermi level (EF), influencing the charge separation and photoelectrochemical values of the cells [15, 17, 18]. Regarding toxicity and recent applications, concentrations lower than 2.10⁻² mol L⁻¹ are not toxic and relate to its use, beyond the corrosion inhibitor, BTAH has been used as protease inhibitor to several virus classes, such as Corona Virus (SARS-CoV) [19, 20].

This work aims to first report the application of BTAH as an additive to DSSC and understand their role in photoanode/electrolyte interface, as a new alternative to produce systems with superior photoelectrochemical parameters.

**Experimental**

The oxide paste was produced with 3 g the TiO₂ (Aldrich®, 25 nm 100% anatase), 1 ml of polyethylene glycol, 0.1 mL of acetylacetone, 4 mL of deionized water, and 0.1 mL of Triton-X. TiO₂ suspension was coated by Doctor Blading method under FTO (~ 7 Ω sq⁻¹ Aldrich®). After deposition, the films were sintered at 450 °C for 30 min followed by immersion for 12 h in N719 commercial dye solution (2.5 × 10⁻⁴ mol L⁻¹ in ethanol) containing 0.148 mg mL⁻¹; 2 mg mL⁻¹ and 20 mg mL⁻¹ of BTAH [10].

The electrolyte was fabricated by mixing 0.5 mol L⁻¹ tert-butylpiridine, 0.6 mol L⁻¹ tetrabutylammonium, 0.1 mol L⁻¹ lithium iodide, and 0.1 mol L⁻¹ of resublimate iodine, in methoxypropionitrile [5]. The cathode catalyst was produced by Pt electrodeposition under FTO, using a three-electrode system, with FTO as a working electrode, Pt as a counter electrode, and Ag/AgCl as a reference electrode, using K₂PtCl₆ dissolved in HCl as electrolyte [18].

The solar cells were assembled in sandwich format, in an active area of 0.2 cm². The electrochemical measurements of j-V curves were taken in Zhenium Zahner® potentiostat with a solar simulator provided by a Xenon lamp at 100 mW cm⁻². Electrochemical Impedance Spectroscopy (10 mHz–10 kHz at 10 mV of perturbation), Intensity Modulated Photovoltage Spectroscopy (IMVS), and Intensity Modulated Photocurrent Spectroscopy (IMPS) (100 mHz–1 kHz at 10 mW of perturbation) were performed in an auto lab potentiostat from Metrohm®, coupled with a led lamp of 530 nm at 50 mW cm⁻².

**Results and discussion**

In Fig. 1 it is depicted the j-V curves to dye solar cells analyzed, and the photoelectrochemical results in Table 1.

| BTAH (mg mL⁻¹) | j_sc (mA cm⁻²) | V_oc (V) | FF | η (%) |
|----------------|----------------|---------|----|-------|
| 0              | 8.52 ± 0.48    | 0.802 ± 0.009 | 0.555 ± 0.068 | 3.78 ± 0.09 |
| 0.148          | 8.09 ± 0.38    | 0.780 ± 0.004 | 0.592 ± 0.003 | 3.74 ± 0.01 |
| 2              | 9.92 ± 0.26    | 0.770 ± 0.002 | 0.557 ± 0.014 | 4.26 ± 0.03 |
| 20             | 6.21 ± 0.89    | 0.767 ± 0.005 | 0.628 ± 0.045 | 3.01 ± 0.06 |

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Fig. 1. j-V curves to solar cells with different BTAH amounts

Table 1. Photoelectrochemical parameters to solar cells with different BTAH amounts
that BTAH amount in N719 dye has been able to shift the $E_t$ level to more positive potentials [22]. One possible key to improve $V_{sc}$ values is synthesizing new dyes containing BTAH in their own composition, not acting only as a spacer, even to composing the sensitizing material [4].

The photoconversion energy efficiency ($\eta$) was obtained using Eq. 1, where $j_{sc}$ is the short circuit current, FF fill factor, and $P_{in}$ the incident power [23].

$$\eta = \frac{j_{sc}V_{oc}FF}{P_{in}} \times 100 \quad (1)$$

It has been observed superior $\eta$ value (4.26 ± 0.03) to the cell prepared with BTAH in 2.0 mg mL$^{-1}$ condition when compared to the standard cell (3.78 ± 0.09). It demonstrated an efficiency boost of 12.8% in the energy conversion, due to superior photocurrent to the same BTAH condition. The $\eta$ values, when compared to literature were reduced since it was not made TiCl$_4$ treatment and the scattering TiO$_2$ layer was not used [24].

It is also observed, that BTAH in the concentration of 0.148 mg mL$^{-1}$ has not demonstrated a different behavior when compared to bare cells (due to deviation values). In higher concentrations of 20.0 mg mL$^{-1}$ and superior, it is expected an inhibition process of the charge collection, because BTAH molecules must occupy all the TiO$_2$ active layer, reducing the current and PCE values [25]. The space charge region in TiO$_2$/electrolyte interface (SCR) should be undergoing changes, improving electron collection rate (Reaction 1), and reducing the recombination reaction demonstrated by Reaction 2 [26]. For a better analysis of its supposed behavior, EIS measurements were taken and Bode diagram is depicted in Fig. 2.

In Bode diagram of Fig. 2, it is observed two different regions. Higher frequencies are related to Pt/Electrolyte interface and in lower frequencies, SCR interface [12]. It is noted a shift to reduced frequency values to the region of working electrode. As frequency and time are inversely proportional, lower frequencies mean a slowness in the charge transfer process, and to confirm this resistance, Nyquist plots (Fig. 3) were fitted using a simplified equivalent circuit (Fig. 1.SI) [11, 27–29]. The resistance values of TiO$_2$/Dye, BTAH interface ($R_{WE}$), and Pt/Electrolyte with the chemical capacitance ($C_{\mu}$) were estimated in Table 2.

By adding BTAH in 2.0 mg mL$^{-1}$ concentration, it is noted a great minimization of charge transfer resistance in TiO$_2$/dye, BTAH/electrolyte interface. This behavior demonstrates a more conductive and less resistive system which should facilitate the charge transport mechanism, being a great advantage when compared to similar additives applications, that showed an increase in $R_{WE}$ [8]. At higher values of added benzo triazole, $R_{WE}$ starts to rise up, proving that at high amounts (> 20 mg mL$^{-1}$) it is obtained a solar system with a charge process transfer minimized. Nyquist diagrams in Fig. 3 also demonstrate the same reduction in $Z_\mu$ values, with BTAH addition, which corroborates the $j_{sc}$ and $\eta$ values obtained from $j$-$V$ curves (Table 1).

For the chemical capacitance ($C_{\mu}$), it is observed proportional decreased values with the amounts of BTAH added, as also verified in $C_{\mu}$ versus $f$ curves of Fig. 2.SI. This behavior has been shown a reduce in the electrical double layer formed at working electrode interface.

With equal or higher amounts of BTAH (> 20.0 mg mL$^{-1}$), it is supposed to obtain an increased resistance value, due to TiO$_2$ pores filling that would be filled by the photosensitive molecule, decumulating the $C_{\mu}$. It is clear that there is a concentration threshold that potenti ates the solar cell photoelectrochemical parameters which must be well analyzed. A schematic overview about these discussions is shown in Fig. 4.

As shown in Fig. 4, a vacancy site, due to dye absence is an active site of recombination reaction, caused by electrolyte filling [30]. With BTAH insertion, there is a deaggregation of the dye, due to a competition for a TiO$_2$ surface site. As benzo triazole molecules present a reduced size, it is able to fill the vacancy sites, avoiding by a steric hindrance and oxide adsorption effect, the contact of TiO$_2$ and oxidized electrolyte. As demonstrated in some papers, BTAH has been able to easily adsorb in several materials [19, 20]. The charge recombination process should still occur, due to conjugated duple bonds, however with a slower speed.

As Reaction 1 and 2 occurs in TiO$_2$/electrolyte interface, it is not feasible to study the processes separately by EIS diagram. To well analyze the back-electron recombination and charge collection the IMVS and IMPS diagram were performed on cells and are depicted in Fig. 5A-B.
As the IMVS operates in open circuit condition and IMPS in short circuit, it is possible to calculate the electron lifetime \( \tau_e \) and collection lifetime \( \tau_c \) using Eq. 2 \([29, 31, 32]\).

The calculated results are shown in Table 3.

In Fig. 5A, it is observed a shift to lower frequencies of the maximum frequency point to 2.0 mg mL\(^{-1}\) of BTAH when compared to an unmodified device. This condition suggested a superior electron lifetime, which is a positive point to dye solar device and was confirmed by values in Table 2 \([10, 32]\). It is seen a \( \tau_e = 0.041 \) s and a \( \tau_c = 0.034 \) s to 2.0 mg mL\(^{-1}\) and 0 mg mL\(^{-1}\) of BTAH, respectively, showing a prevention of charge recombination between \( I_3^- \) with \( TiO_2 \) \([5]\). The presence of the organic structure in BTAH difficult to access of \( I_3^- \) species and these molecules also can occupy the vacancy sites in \( TiO_2 \) semiconductors, developing a slow back-electron recombination.

In the IMPS diagram, it is also noted a shift to higher frequencies with BTAH insertion, suggesting faster collection reactions. Using Eq. 2 \( \tau_c \) was calculated and an improvement in collection time was observed when BTAH was added to the dye. This behavior, evidence that the used additive has been able to boost the collection in \( TiO_2 \) semiconductor oxide \([18]\). Again, the steric effect is caused by benzene

![Nyquist plots with fitted values from DSSC with different BTAH amounts](image)

**Table 2.** - Resistances and chemical capacitance of the cells obtained by fitting the Nyquist EIS plots

| BTAH (mg mL\(^{-1}\)) | \( R_{WE} \) (Ω cm\(^2\)) | \( C_\mu \) (mF) |
|------------------------|--------------------------|---------------|
| 0                      | 34.2                     | 1.57          |
| 0.148                  | 30.2                     | 0.98          |
| 2.0                    | 21.8                     | 0.72          |
| 20.0                   | 28.6                     | 0.36          |

As the IMVS operates in open circuit condition and IMPS in short circuit, it is possible to calculate the electron lifetime \( \tau_e \) and collection lifetime \( \tau_c \) using Eq. 2 \([29, 31, 32]\). The calculated results are shown in Table 3.

\[
\tau_e = \frac{1}{2\pi f_{min}}
\]  

(2)

In Fig. 5A, it is observed a shift to lower frequencies of the maximum frequency point to 2.0 mg mL\(^{-1}\) of BTAH when compared to an unmodified device. This condition suggested a superior electron lifetime, which is a positive point to dye solar device and was confirmed by values in Table 2 \([10, 32]\). It is seen a \( \tau_e = 0.041 \) s and a \( \tau_c = 0.034 \) s to 2.0 mg mL\(^{-1}\) and 0 mg mL\(^{-1}\) of BTAH, respectively, showing a prevention of charge recombination between \( I_3^- \) with \( TiO_2 \) \([5]\). The presence of the organic structure in BTAH difficult to access of \( I_3^- \) species and these molecules also can occupy the vacancy sites in \( TiO_2 \) semiconductors, developing a slow back-electron recombination. On the other hand, the 20 mg mL\(^{-1}\) BTAH addition has not been presented the best \( \eta \) values to conditions tested, and despite superior \( \tau_c \), its present low \( C_\mu \).

In the IMPS diagram, it is also noted a shift to higher frequencies with BTAH insertion, suggesting faster collection reactions. Using Eq. 2 \( \tau_c \) was calculated and an improvement in collection time was observed when BTAH was added to the dye. This behavior, evidence that the used additive has been able to boost the collection in \( TiO_2 \) semiconductor oxide \([18]\). Again, the steric effect is caused by benzene
presence in a molecule, and also the N719 dye deaggregation, as the BTAH molecules might be positioning themselves between the dye molecules, rising up the charge separation and transportation in the semiconductor electrolyte interface. To 20 mg mL$^{-1}$ of BTAH, despite amazing $j_{sc}$ presumably reduce the number of dye molecules anchored in TiO$_2$ surface, supplying less electron under solar energy incidence, supported by $j_{sc}$ value in Table 1 [4, 11].

Then, it is important to emphasize that BTAH inserted in N719 dye solution is an amazing additive to be used in dye solar devices to enhance photoelectrochemical parameters.

**Conclusions**

BTAH molecules are effective additives in dye solar devices, able to enhance the photoelectrochemical parameters of the cell.

The back-electron recombination ($\tau_e$) can be restricted and the collection time ($\tau_c$) can be raised by BTAH presence. $\tau_e$ and $\tau_c$ enhancing are proportional to additive concentration, due to decreasing in recombination TiO$_2$/electrolyte sites. On the other hand, higher BTAH amounts,

### Table 3.

- Extracted data from the IMPS and IMVS diagrams with their respective time calculations

| BTAH (mg mL$^{-1}$) | $f_{IMVS}$ (Hz) | $\tau_e$ (s) | $f_{IMPS}$ (Hz) | $\tau_c$ (s) |
|---------------------|----------------|-------------|----------------|-------------|
| 0                   | 4.751          | 0.034       | 1.933          | 0.082       |
| 0.148               | 4.751          | 0.034       | 2.004          | 0.079       |
| 2.0                 | 3.900          | 0.041       | 3.141          | 0.050       |
| 20.0                | 6.2189         | 0.026       | 5.009          | 0.031       |

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Fig. 4. Schematic overview of BTAH influence in TiO$_2$ dye/electrolyte interface

Fig. 5. a–b. IMVS diagram in 5A and IMPS in 5B to solar cells with different BTAH amounts.
difficult the dye anchoring, reducing the photoelectrochemical parameters.

The best solar cell was achieved at concentration of 2.0 mg mL$^{-1}$ of BTAH, reaching a $j_{sc} = 9.92 \pm 0.26$, $V_{oc} = 0.770 \pm 0.022$, $FF = 0.557 \pm 0.014$, $\eta = 4.26 \pm 0.13$, $\tau_c = 0.034$ s and $\tau_c = 0.082$ s with lower charge transfer resistance.

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