



Effects of substitution position on electrochemical, electrochromic, optical, and photoresponsive properties of azobenzenecarboxylic acid alkyl ester derivatives



Qian Tang, Li-hua He, Yue-hong Yang, Jun-fei Long, Xiang-Kai Fu^{*}, Cheng-bin Gong^{**}

The Key Laboratory of Applied Chemistry of Chongqing Municipality, College of Chemistry and Chemical Engineering, Southwest University, Chongqing, 400715, China

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ABSTRACT

This study aimed to investigate the effects of substitution position on the electrochemical, electrochromic, optical, and photoresponsive properties of azobenzenecarboxylic acid alkyl ester derivatives. Therefore, a series of azobenzenen ester derivatives, including azobenzene-4,4'-dicarboxylic acid dialkyl ester derivatives (ADDEDs), azobenzene-3,3'-dicarboxylic acid dialkyl ester derivatives (ADADEs), and azobenzene-3,3',5,5'-tetracarboxylic acid four alkyl ester derivatives (ATFAEDs), was synthesized in this work, and their electrochemical, electrochromic, optical, and photoresponsive properties were characterized. Substitution position significantly influenced the electrochemical, electrochromic, and optical properties but not the photoresponsive properties of azobenzenecarboxylic acid alkyl ester derivatives. With regard to stability and response time, the electrochromic properties of azobenzenecarboxylic acid alkyl ester derivatives decreased in the following order: ADDEDs > ATFAEDs > ADADEs. However, ADDEDs, ADADEs, and ATFAEDs can all be reversibly and efficiently photoswitched between *trans* and *cis* states upon alternate irradiation at 365 and 440 nm under electrochromic conditions.

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1. Introduction

Electrochromic materials (ECMs) can modify optical properties as a persistent and reversible response to alternation in applied potential. Therefore, ECMs can be employed in numerous practical applications, such as electronic paper [1,2], smart windows [3–5], and antiglare vehicle mirrors [6,7]. ECMs can be divided into three categories: single molecular materials, organic and polymeric materials, and inorganic materials [8,9]. Given the fast response times, flexible device fabrication, relatively narrow absorption bands for displaying diverse and clear colors, and large-scale processability, organic ECMs have received significant research attention. In 1973, Schoot et al. [10] reported viologen as an organic electrochromic material for the first time. Since then, other viologen derivatives [11–17] and organic compounds, such as thiophene derivatives [18], polypyrrole derivatives [19], anthraquinones [20],

dithienylpyrroles [21], and polymer electrolytes [22], have been developed as ECMs.

Organic ECMs containing esters, such as isophthalates combined with diaryl ketones [23], aromatic oxygen, and sulfur diesters [24], demonstrate strong color changes from colorless to primary colors (red, blue, or yellow), which can be used to produce any desired color shade for displays [25]. In particular, *para*-substituted azobenzene derivatives called azobenzene-4,4'-dicarboxylic acid dialkyl ester derivatives (ADDEDs) (Fig. 1, compounds **1a–1f**) do not only exhibit color change from colorless to primary colors (magenta) with good stability, high color efficiency, short response time, and high optical contrast but can also present photoresponsive properties [26]. Hence, ADDEDs are excellent candidates for applications such as electronic paper, smart windows, optical memory devices, full-color EC display devices, and dual-stimuli-responsive systems. The current work aimed to investigate the effects of substitution position on the electrochemical, electrochromic, optical, and photoresponsive properties of azobenzenecarboxylic acid alkyl ester derivatives. However, Nakamura et al. [27] reported that the anion radicals of *ortho*- or *meta*-substituted diesters are less stable than *para*-substituted diesters. Sharmoukh et al. [28,29] found that 5-substituted isophthalate derivatives and bis-isophthalates exhibit

^{*} Corresponding author.

^{**} Corresponding author.

E-mail addresses: fxk@swu.edu.cn (X.-K. Fu), gongcbtq@swu.edu.cn (C.-b. Gong).

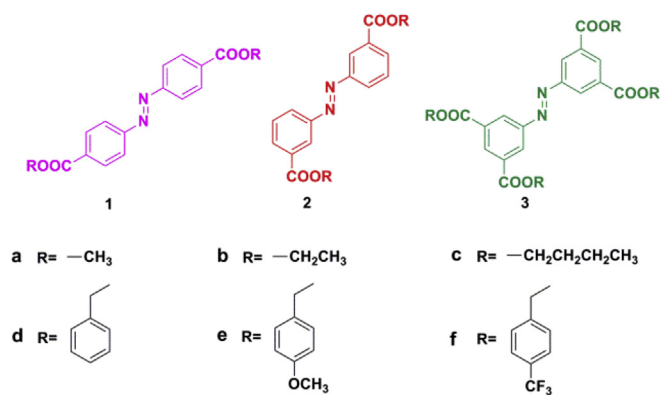


Fig. 1. Chemical structures of azobenzene carboxylic acid alkyl ester derivatives.

electrochromic properties. As such, azobenzene-3,3'-dicarboxylic acid dimethyl ester derivatives (ADADEs) (Fig. 1, compounds **2a–2f**) and azobenzene-3,3',5,5'-tetracarboxylic acid four alkyl ester derivatives (ATFAEDs) (Fig. 1, compounds **3a–3f**) were synthesized and their electrochemical, electrochromic, optical, and photoresponsive properties were characterized via cyclic voltammetry and UV–Vis spectroscopy. The influence of substitution position was investigated by comparing these data with *para*-substituted azobenzene derivatives.

2. Experimental

2.1. Materials and apparatus

All chemicals were purchased from Aladdin Co., Shanghai, China and used as received. All solvents were of analytical-reagent grade, commercially available, and used without further purification.

^1H NMR and ^{13}C NMR spectra were measured on a Bruker AV 300 (300 MHz) spectrometer at ambient temperature by using tetramethylsilane as an internal standard. HPLC-MS was performed on a Bruker Esquire 2000 HCT LC/MS system. FT-IR spectra (400 cm^{-1} to 4000 cm^{-1}) were recorded on a Perkin–Elmer Model GX spectrometer through KBr pellet method. UV–Vis spectra were obtained by a UV-4802 spectrophotometer [UNICO (Shanghai) Instruments Co., Ltd., China]. Cyclic voltammetry was performed on a CHI 650B electrochemical workstation by using a three-electrode system, and Ag/AgCl was used as reference electrode.

2.2. General procedure for synthesis of ADADEs and ATFAEDs

ADADEs and ATFAEDs were synthesized using previously reported methods [26,30] with certain modifications (Fig. 1). The detailed procedure and characterization are presented in ESI.

2.3. ECD construction

All ECDs based on ADADEs and ATFAEDs were fabricated, sealed,

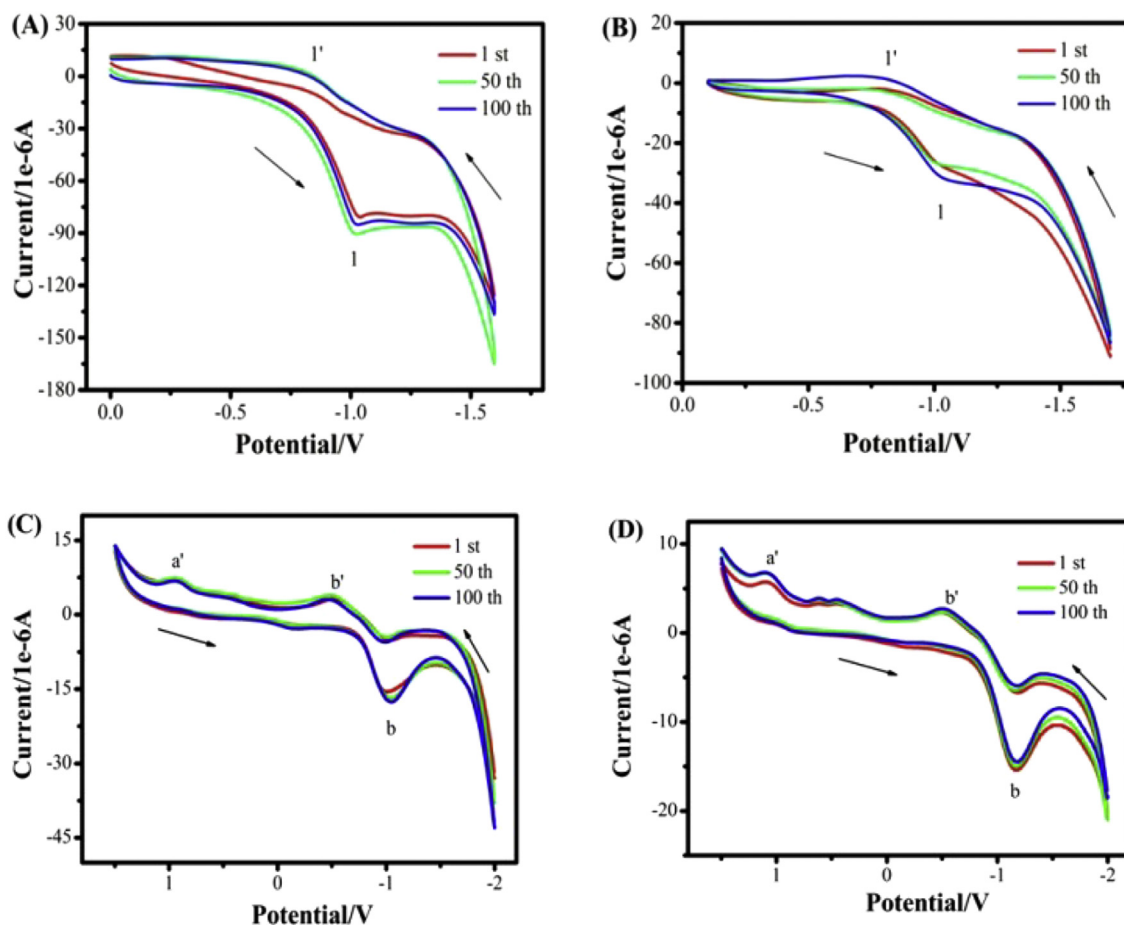


Fig. 2. Cyclic voltammograms of ADADEs and ATFAEDs [(A) and (B) for compounds **2e** and **2f**; (C) and (D) for compounds **3e** and **3f**] in DMF (with 0.05 mol L^{-1} TBAP) vs. Ag/AgCl at room temperature. The 1st, 50th, and 100th cycles are shown, and the scan rate is 50 mV s^{-1} .

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