

Electrical, optical and morphological properties of a new chromogenic calix[4]arene derivative



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ARTICLE INFO

Article history:

Received 20 May 2014

Received in revised form 11 August 2014

Accepted 5 September 2014

Keywords:

Chromogenic calix[4]arene

Thin films

Optical properties

Electrical properties

Morphological properties

ABSTRACT

We have studied electrical properties of ITO/azo-calix[4]arene/Al diodes by means of current–voltage measurement and impedance spectroscopy in a wide frequency range. The devices with an ITO/azo-calix[4]arene/Al structure could be modeled as a simple combination of resistors and capacitors. The impedance spectra can be discussed in terms of an equivalent circuit model designed as a parallel resistor R_p and capacitor C_p network in series with resistor R_s . We extract numerical values of these parameters by fitting experimental data. Relaxation processes have been identified too from the impedance spectroscopy. Their values deduced from fitting experimental data to the model have given a dielectric relaxation time in the ms range and an exponential trap distribution.

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1. Introduction

Organic materials have attracted much interest in recent years due to their low cost, the possibility of their deposition from solution onto large-area substrates, and the ability to tailor their properties [1–8].

Calix[n]arene materials have been in particular extensively studied for several potential applications because of their flexibility, good physical and chemical stability and suitability for host–guest interaction. The various available conformations of these molecules and ability to form complexes allow the utilization of calix[n]arenes as fundamental building blocks that can be tailored to make calix[n]arenes with desired functionalities for quantitative and qualitative applications.

Calix[n]arenes are macrocyclic aromatic molecules which originate from the synthesis of phenols and aldehydes, leading to the introduction of an index [n] indicating the number of the phenol aromatic cycles in the molecule. Currently, calixarenes are also known as the third generation supramolecules after cyclodextrins

and crown ethers [9]. Calixarene chemistry is a curious combination of academic and industrial research.

Calixarenes have commonly been utilized for the purpose of separation [10] and extraction of various analytes such as the selective removal of aromatic amines [11], heavy metal [12–14], dyes [15–17], and oxyanions [18,19]. They have also been utilized in several chemical sensor devices including ion and molecule-selective electrodes for various ions and molecules [20,21], fluorescent sensors [22,23], and nonlinear optical sensors [24].

Most potential applications require that these materials could be prepared as thin films form to develop a suitable technique [25].

Calixarenes and the related calix[4]resorcinarene derivatives in the form of thin films are of particular interest within the sensor community due to their selective recognition of different molecules. Films of controlled thickness of calix[4]resorcinarenes can be easily fabricated using the Langmuir–Blodgett (LB) technique [26] onto varying substrates to be used as chemical sensors.

In this study we have studied the optical characteristics of new chromogenic calix[4]arene derivatives whose synthesis was presented in a previous work [27]. We report a study of the electrical properties of ITO/calix[4]arene diode structures. The obtained results have been fitted to an electrical equivalent circuit in order to extract the relaxation time of these materials.

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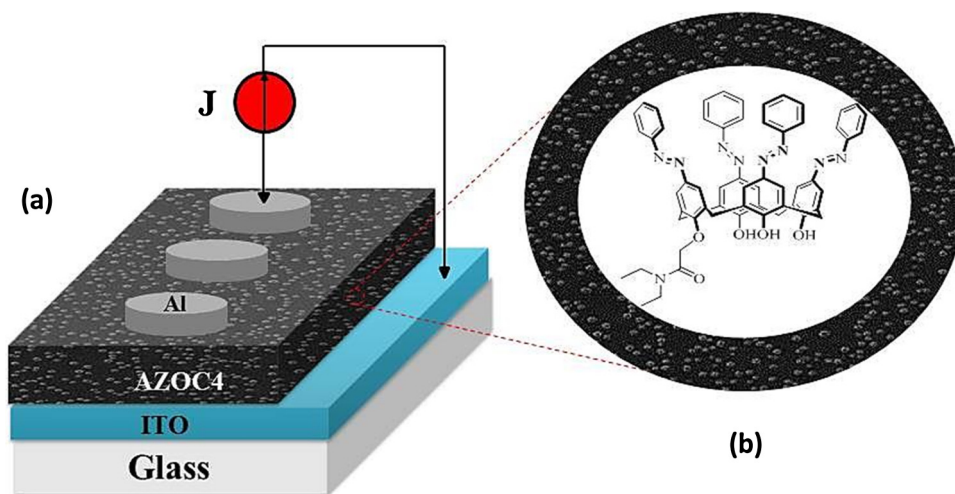


Fig. 1. (a) ITO/AZOC4/Al diode heterostructure, (b) chemical structure of azo-calix[4]arene derivative used in this work.

2. Experimental

2.1. Materials

Our study is based in a new chromogenic azo-calix[4]arene derivative characterized by the presence of an amide group at the lower rim and an azo group at the upper rim. This new material is represented in Fig. 1(b) and is synthesized according to the procedure presented in these references [28–30].

2.2. Organic diode fabrication

ITO-coated glass with a sheet resistance of $20 \Omega/\text{square}$ was used as an anode in the organic diode fabrication. In this process, the ITO glass was cleaned sequentially in an ultrasonic bath of acetone and isopropanol alcohol; it was then, sonicated in deionized water and finally blown dry with N_2 gas. The ITO coated glass substrates are used as the anode due to its superior properties, such as good transparency, high work function, high efficiency, and high conductivity.

A solution of azo-calix[4]arene was dissolved in acetonitrile solvent and then was spin-coated on the cleaned ITO pre-coated glass substrate at the speed of 1500 rpm for 60 s followed by heating on a hot-plate at 80°C for 30 min.

For processing the cathode, samples were put into an evaporator, in which Al metal electrodes (100 nm) were thermally evaporated at 2×10^{-6} Torr pressure through a shadow mask to produce simultaneously three diode structures. Fig. 1(a) shows the typical device structure of the diode investigated in this study.

2.3. Instrumentation and measurements

To avoid the absorption of the ITO substrate in UV region, a thin film of azo-calix[4]arene was deposited on a glass substrate. A Perkin-Elmer UV-vis Spectrophotometer (Lambda 35) has been used to characterize the optical properties of this material.

The current–voltage measurements were performed with a Keithley 236 source measure unit from an applied bias -12V to 12V . The impedance measurements were carried out with an impedance analyzer (Hewlett Packard 4192ALF) controlled by a computer acquisition. In general, the excitation potential for dynamic measurements is given by:

$$V = V_0 + V_{\text{mod}} \cos(\omega t) \quad (1)$$

where V_0 is a DC bias, V_{mod} is the oscillation level and $\omega/2\pi$ is the frequency. In our study, these measurements were released in the following conditions V_0 : 0–6 V, a V_{mod} of 50 mV over a frequency range of 5 Hz–13 MHz. All these electrical measurements were performed in dark and at room temperature.

The surface morphology of AZOC4 layers deposited on top of ITO were performed by scanning electron microscopy (SEM Hitachi S800) at a voltage of 15 kV and by atomic force microscopy (AFM) using a Nanoscope III in tapping mode.

All these measurements have been performed under room conditions.

3. Results and discussion

3.1. Optical properties of the azo-calix[4]arene derivative thin film

Before the fabrication and characterization of devices, the optical properties of the films based on the AZOC4 have been studied. The effect of the solvent used for the film preparation on these properties has been investigated.

The dispersion of the AZOC4 is in particular a critical issue and the solvent has a strong influence on the morphology of the produced films. Therefore an important step for the fabrication of suitable devices is to select the appropriate solvent to disperse the AZOC4 and achieve homogeneous films. After a rapid screening we have checked different solvents for the dispersion of the AZOC4. Tetrahydrofuran (THF) and acetonitrile are a rapidly evaporating solvent so that the deposited layer could be quenched in the dispersion state of the AZOC4 without any further reorganization.

The morphology of the AZOC4 layers deposited from the two solvents (THF and acetonitrile) has been studied by scanning electron microscopy (SEM) as it can be seen in Fig. 2. We can clearly see a tremendous difference between the surface morphologies of the film according to the solvent. We have already explained this difference in a previous work [31].

The UV–vis spectra of the AZOC4 performed in solution and or as thin films are shown in Fig. 3(a). Note that the red shift of the absorption of approximately 25 nm for AZOC4 as thin film versus solution suggests a high degree of macromolecular organization in the thin film phase.

Fig. 3(b) shows the UV–vis spectra of the AZOC4 prepared with two different solvents: THF and acetonitrile. It can be observed that the strongest absorption band is obtained for the last

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