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Original research article

# Structural investigation, thermal analysis and AC conduction mechanism of thermally evaporated alizarin red S thin films

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ABSTRACT

The differential scanning calorimetric (DSC) and the thermogravimetric analysis (TGA) of alizarin red S (ARS) in the powder form were investigated. Subsequently, the structural studies of thermally evaporated ARS thin films of were performed using the Fourier Transform Infrared spectroscopy (FTIR), X-ray diffraction (XRD) and the transmission electron microscope (TEM). Furthermore, the dielectric properties and alternating current conductivity, $\sigma_{ac}$, of ARS thin film in the frequency range 100 Hz–5 MHz through the temperature range 303–393 K were investigated. The activation energy, $\Delta E_{ac}$, was found to decrease as the frequency increases. Moreover, both of experimental and theoretical results of the frequency exponent, $S$, and $\sigma_{ac}$ as a function of temperature outweigh the overlapping large polaron tunneling model (OLPT) for explain the conduction mechanism for ARS thin films. Also, the Cole–Cole diagram for dielectric modulus and the complex impedance at different temperatures were investigated.

## 1. Introduction

Anthraquinone and their derivatives have been extensively studied for their promising electrical properties. They have many applications such as: wastewater treatment [1], photosensitizers [2], dye-sensitized solar cells (DSSC) [3], potential dyes [4] and synthetic fibers [5]. Particularly, Alizarin (1, 2-dihydroxy anthraquinone) and 1,4-dihydroxy anthraquinone thin films are n-type and photosensitive semiconductor [6].

According to theoretical calculation B3LYP/6-311 + G(d,p) using TDDFT methods, Ranjith et al. [3] calculated the energy gap transition of alizarin dye sensitizer from HOMO to LUMO of the molecule to be about 3.13 eV. Also, they reported that all absorption transitions were ascribed to n $\rightarrow\pi^*$ transition. Tuma et al. [7] used alizarin to enhance the optical constant of a poly methyl methacrylate films. The films doped with alizarin dye have indirect energy band gap which decreases with increasing the concentration of alizarin. Moreover, the absorbance of the prepared films increases with increasing the alizarin concentration.

Chandra et al. [8] have studied the electrical properties of alizarin doped anthraquinone in bulk form. They reported a direct activation energy $\Delta E_{dc}$ equals 0.57 eV and the alternating activation energy $\Delta E_{ac}$ equals 0.052 eV at 1 MHz. Also they investigated the conduction mechanism and found that the conduction mechanism of alizarin doped anthraquinone could be described in correlated barrier hopping model.

The electrical properties of some binary and ternary anthraqinone complexes in bulk form was discussed by Abd Elwahed et al. [9]. They found that the activation energy of ternary complexes is higher than its counterpart in binary complexes, but the conductivity was found to be the opposite. Moreover the activation energy of the hopping process lies between 0.021 and 0.079 eV [7].

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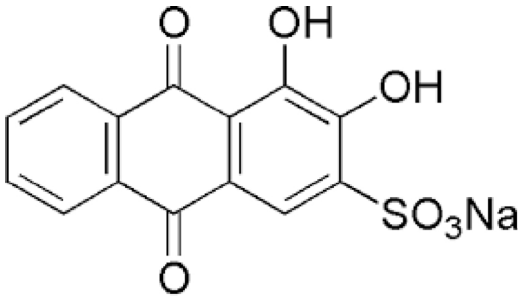


**Fig. 1.** The chemical structure of ARS.

Sun et al. [10] prepared alizarin complex (alizarin-3-methylimino-*N,N*-diacetic acid) as the sensitizers in dye-sensitized solar cells (DSSC). The cyclic voltammetry of alizarin complex showed that the first oxidation potential equals 0.28 V. The DSSC sensitized based on Alizarin showed energy conversion efficiency of 0.27%, with short circuit current of 0.89 mA and open circuit voltage of 0.45 V.

Alizarin red S (1,2-dihydroxy-9,10-anthraquinonesulfonic acid sodium salt) with linear formula $C_{14}H_7NaO_7S$ is classified as organic semiconductors dyes which belongs to a group of the anthraquinone dyes. It has been used as a chemical dye in paints and as a chemical indicator [11,12]. Faouzi et al. [13] executed the cyclic voltammetry for ARS. They found that a peak at 0.85 eV which is identical to the oxidation of ARS.

However, literature survey discloses that the AC conductivity and dielectric studies on ARS thin films have not been reported so far. Therefore this paper is devoted to study the temperature dependence of the dielectrical and AC conductivity of ARS thin films on the light of the thermal and the structural properties.

## 2. Experimental details

Alizarin red S (ARS) powder was obtained from Oxford laboratory reagent. The chemical structure of ARS is shown in Fig. 1. The thermogravimetric analysis (TGA) and the differential scanning calorimetric (DSC) measurements for ARS powder were carried out using Shimadza, TGA-60 heating rate 10°/min under $N_2$ atmosphere in the temperature range 293–873 K and DSC Q20 V24.11 Build 124 in the temperature range 303–573 K, respec twin, double tilt. Samples of the construction tively. Thereafter, the Fourier transform infrared spectroscopy (FTIR) measurements for the powder and thin films were carried out using Perkin-Elmer 1340 spectrophotometer. The X-ray diffraction patterns of the powder as well as thin films of ARS samples were recorded using A Philips X-ray diffractometer model X// Pert Pro, utilized by monochromatic Cu $K_\alpha$ radiation ($\lambda = 1.5418$ Å) the operating voltage and current were 40 kV and 25 mA, respectively. Transmission electron microscope (TEM) for samples of ARS thin films are carried out using Tecnai, G20, super twin, double tilt. Samples of the construction Ag/ARS/Ag were prepared onto optical flat glass substrates by the thermal deposition technique, using a high vacuum coating unit (Edwards Co., model E306 A, England). The vacuum in the chamber during deposition was kept at $5 \times 10^{-4}$ torr. Silver was evaporated using tungsten basket shaped filament and ARS was sublimated from boat-shaped molybdenum. The film thickness was measured during the evaporation using a quartz crystal thickness monitor (model TM-350 MATREX, Inc). A programmable automatic RLC bridge, model Hioki 3532 Hitester was used for the AC measurements in frequency range 100 Hz–5 MHz. The temperature of the sample was monitored by a k-type thermocouple.

## 3. Results and discussion

### *3.1. Structural investigations*

#### *3.1.1. Thermal analysis*

The thermogravimetric analysis (TGA) and differential of TGA, with respect to temperature (DTG), for ARS received powder are presented in Fig. 2 in the temperature range 303–538 K. According to DTG and TGA, the loss of the sample mass is about 7.5% of its original mass ($m_0$). This loss may be attributed to the loss of moisture in the first step of heating and loss of impurities at higher temperatures via decomposition. In the temperature range 538–673 K, a sharp decrease of sample's mass up to 50% was observed with an onset temperature of 593 K. This onset temperature may be related to the start of a boiling or decomposition process. Fig. 3 presents the DSC for ARS in the temperature range 303–573 K. The first exothermic broad peak at 368 K can be attributed to the crystallization of ARS powder, while the first endothermic peak at 418 K may be attributed to the melting temperature ($T_m$) of ARS. The endothermic peaks at 433 K and 453 K can be attributed to the melting of impurities.

#### *3.1.2. FTIR characterization of ARS*

Fig. 4 shows the FTIR spectra for the powder and the as deposited ARS thin films. The infrared technique has been used to confirm the molecular structure of the ARS compound. This result demonstrates that the thermal evaporation technique is a good method for the preparation of ARS thin films. Fig. 4 displays several peaks and assigned according to [14,15,3] as follows: the peak at 3487cm$^{-1}$

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