



ORIGINAL ARTICLE

Synthesis and optical properties studies of antipyrine derivatives thin films



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KEYWORDS

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Abstract 4-(4-Amino-1,5-dimethyl-2-phenyl-1,2-dihydro-pyrazol-3-ylideneamino)-phenol (L_1) and 4-(4-Amino-1,5-dimethyl-2-phenyl-1,2-dihydro-pyrazol-3-ylideneamino)-benzoic acid (L_2) have been synthesized by the condensation reaction of 4-aminoantipyrine (4-AAP) and 4-aminophenol or 4-aminobenzoic acid in ethanolic solution and are characterized by various physico-chemical techniques. Thin films of L_1 and L_2 have been prepared by the conventional spin coating technique. X-ray diffraction patterns (XRD) show an amorphous nature for both powder and thin films for L_1 and L_2 ligands. The optical absorption and refraction properties of L_1 and L_2 are investigated by spectrophotometric techniques at normal incidence of light in the wavelength range of 200–2500 nm. The absorption spectra show two peaks in the UV region which correspond to $\pi \rightarrow \pi^*$ transition and a peak in UV-Vis region which may correspond to $n \rightarrow \pi^*$ transition. The values of dispersion parameters E_o , E_d , ϵ_L , ϵ_∞ and N/m^* are calculated according to the single oscillator model. The presence of the OH group increases the value of ϵ_∞ from 3.21 to 3.32 and the value of N/m^* from 7.38×10^{53} to $2.08 \times 10^{54} \text{ m}^{-3} \text{ Kg}^{-1}$. The optical band transition is found to be indirect allowing fundamental energy gap values of 3.4 and 3.9 eV and onset energy gap values of 2.1 and 2.6 eV for L_1 and L_2 , respectively.

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1. Introduction

Heterojunction organics have attracted increasing attention due to their potential applications. Among these heterojunctions are antipyrine derivatives which are promising in optical communications, optoelectronic materials, and biofunctional compounds, especially, excellent nonlinear optical (NLO) responses [1–7].

Antipyrene derivatives (APDs) attracted attention as model compounds of functional materials due to their attractive functional properties such as antioxidant [8], antiputrefactive [9] and optical [10,11] characteristics in the field of chemical physics. For instance, these compounds have been considered as important biomodel compounds in biological and medical fields [12]. With the development of functional materials including bioelectronics [13,14], photovoltaics [15–17], photoluminescence [18,19], and nonlinear optics [20–22], the photoreponsive characteristics of these substances have become one focus area of scientists, gradually.

Sun et al. [6,23] and Zhang et al. [24] studied the crystallographic data of 4-(2-fluorobenzylideneamino) antipyrene (2-FBIAAP), 4-(3-fluorobenzylideneamino) antipyrene (3-FBIAAP), 4-(2,4-dichlorobenzylideneamino) antipyrene (2,4-DCBAP) and 4-(2,6-dichlorobenzylideneamino) antipyrene (2,6-DCBAP). They found that the studied molecules have an asymmetric structure consisting of the prazolone, imine bond and aromatic benzene rings. They concluded that these compounds are promising optical materials due to their functionally structural molecular device with four components concluding one prazolone, one imine bond and two benzene rings. The micro-variable F-substitution of (2-FBIAAP) and (3-FBIAAP) showed micro-differences in structural and partly photoreponsive characteristics, which may lead to the macro-differences in functional materials [23,24].

Arun et al. [25] reported that the Schiff base 3-hydroxyquinoxaline-2-carboxalidine-4-aminoantipyrene (HQCAAP), is polar and exhibits amide-iminol tautomerism. Its polarity decreased in solution. It exhibited positive solvatochromism in its absorption spectra but a negative solvatochromism in its emission spectra in various solvents. The results of the UV–Vis, fluorescence, cyclic voltammetry (CV) and differential scanning calorimetry (DSC) studies suggest that the present compound is a suitable candidate for application in organic electronics. The values of third-order non-linear absorption coefficient, β , imaginary part of the third-order non-linear optical susceptibility, $\text{Im } \chi^{(3)}$, and optical limiting threshold suggest that this compound might find application in the photonic age.

The electronic absorption spectra of APDs and molecular orbital coefficients analysis suggest that the electronic spectra are assigned to $\pi \rightarrow \pi^*$ and $n \rightarrow \pi^*$ electronic transitions [6,23–26]. Data extracted from the UV–Vis spectrum for some dissolved APDs accentuate both the fundamental and onset optical edges around 4.8 eV, 3.3 eV for two FBIAA isomers [23,24] and 3.6 eV, 2.5 eV for the HQCAAP thin film [25].

In spite of the importance of APDs in high-level technological applications, there are insufficient efforts paid for determining the optical constants such as refractive index, dielectric constant and energy gap. So we are interested in the synthesis of new APDs such as 4-(4-amino-1,5-dimethyl-2-phenyl-1,2-dihydro-pyrazol-3-ylideneamino)-phenol (L_1) and 4-(4-amino-1,5-dimethyl-2-phenyl-1,2-dihydro-pyrazol-3-ylideneamino)-benzoic acid (L_2) and in investigating their optical properties in thin film forms with the aim of using the results obtained in using these materials in many applications such as optoelectronic devices.

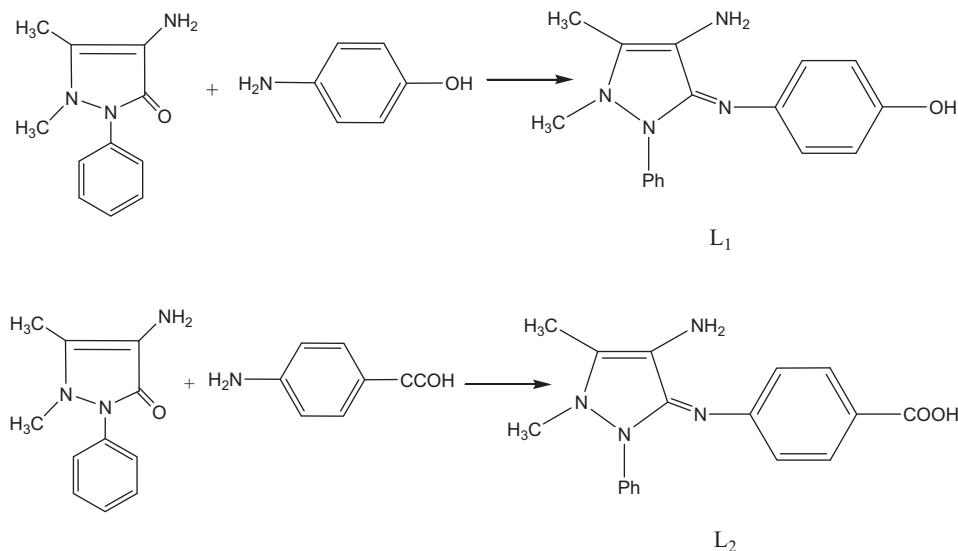
2. Experimental technique

2.1. Synthesis of ligands

Ethanol solutions of 4-aminoantipyrene (4-AAP) (0.1 mol) and *p*-aminophenol/*p*-aminobenzoic acid (0.1 mol) were refluxed together for 4 h over a steam bath (Scheme 1). The excess solvent was removed by evaporation and the concentrated solution was cooled in an ice bath with stirring. The Schiff base which separated out as a colored powder is collected and dried in vacuum [27].

2.2. Preparation of L_1 and L_2 thin films

Homogenous thin films of Schiff base L_1 and L_2 are prepared by a conventional spin coating technique onto pre cleaned optical flat glass substrates [28]. The prepared ligands are dissolved in 3 ml ethanol. The rotating speed of the spin coating system is controlled to be about 2800 rps. The thicknesses of



Scheme 1 The formation mechanism of Schiff base ligands (L_1 and L_2).

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