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Thermal and biological properties of the Schiff base N,N′-bis(salicylidene)-1,2-phenylenediamine, a potential adjuvant to antibiotic therapy

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ABSTRACT

Schiff base N,N′-bis(salicylidene)-1,2-phenylenediamine, salophen, is a substance that presents synergism when combined with amikacin against *Staphylococcus aureus* and *Escherichia coli*. Measurements of temperature dependence of the Raman spectra of salophen combined with thermal analysis investigations are presented. The room temperature crystalline structure seems to be stable up to the temperature where the phase transition from solid to liquid (433–443 K) is observed. The Raman spectra in the temperature range 433–443 K were observed to be characterized by the loss of external vibrational modes, in accordance with thermal analysis curves. According to thermogravimetric analysis, salophen shows a weight loss variation in the temperature range 300–453 K corresponding to 5% loss in weight, which is attributed to dehydration and materials melting temperature. The enthalpy (ΔH) obtained from the integration of the differential scanning calorimetry peak at melting (T_m = 438 K) and decomposition temperature (T_d = 484 K) is founded to be − 91 J/g and 239 J/g, respectively. Finally, it was carried out biological assays to evaluate the antibacterial potential of the salophen.

1. Introduction

Schiff base (–N=C–) derivatives are considered a promising organic class of material for technological application in several research fields, such as catalysis, used as precursor in organic chemistry or as molecular capsules to pacify normally reactive materials; in coordination chemistry and used as biologically inert metal – organic complex with different transition metals to activate proteins; in the pharmaceutics industry and electronic devices, among others [1–9]. N,N′-bis(salicylidene)-1,2-phenylenediamine, salophen, is also a relevant Schiff base ligand, which crystallizes in a monoclinic ($P2_{1/c}$) structure at room conditions, having various physical and chemical properties already established in literature [10–13].

In a previous work, the vibrational properties investigated through Raman and infrared spectroscopy and ab initio calculations allowed authors to identify most of the normal modes of salophen crystal [10]. The present temperature dependent-Raman study combined with thermal analysis (thermogravimetric analysis – TGA, differential thermogravimetric analysis – DTA and differential scanning calorimetry – DSC) is focused on the investigation of the thermal stability and eventual phase transition, as well as anharmonic effects on the phonon spectrum, conformational changes and temperature-induced structural disorder effects. Such a kind of work leads, in general, to the discovery of essential information from fundamental science with potential application in pharmaceutics industry. Related to salophen in particular, we show that the crystalline structure is stable under temperature variation. Additionally, the antibacterial and antibiotic modifying activity of the salophen is also reported. Because the incidence of resistance to

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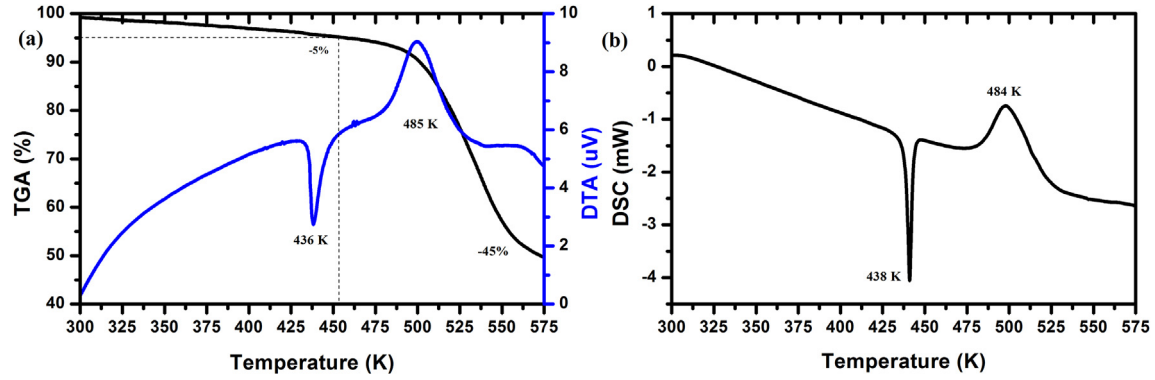

Fig. 1. Thermal analysis for salophen in the temperature range 300–575 K (a) TG-DTA curves and (b) DSC curve.

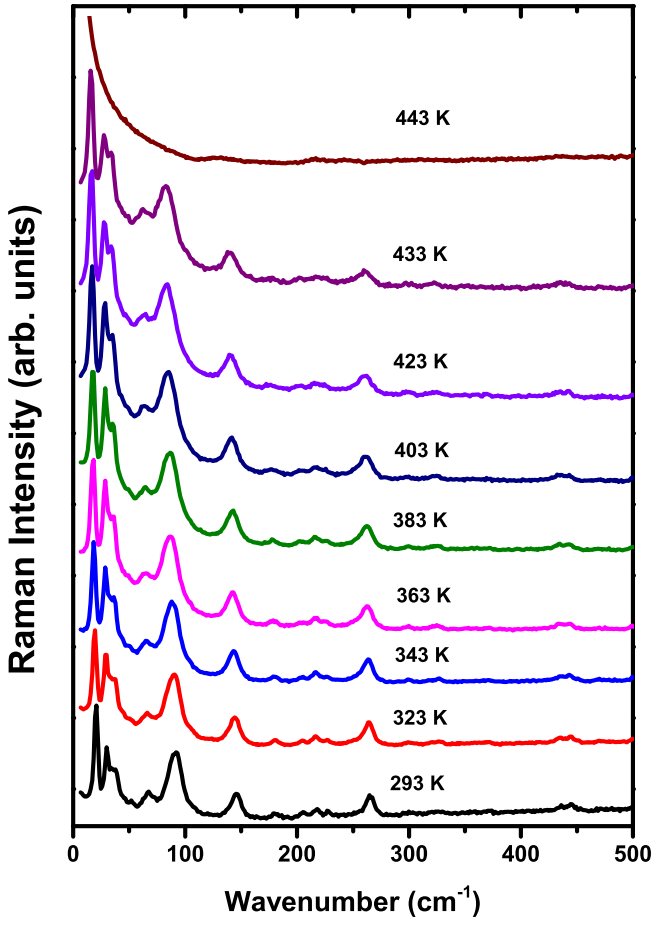

Fig. 2. Raman spectra of salophen for several temperature values in the region from 0 cm^{-1} to 500 cm^{-1}.

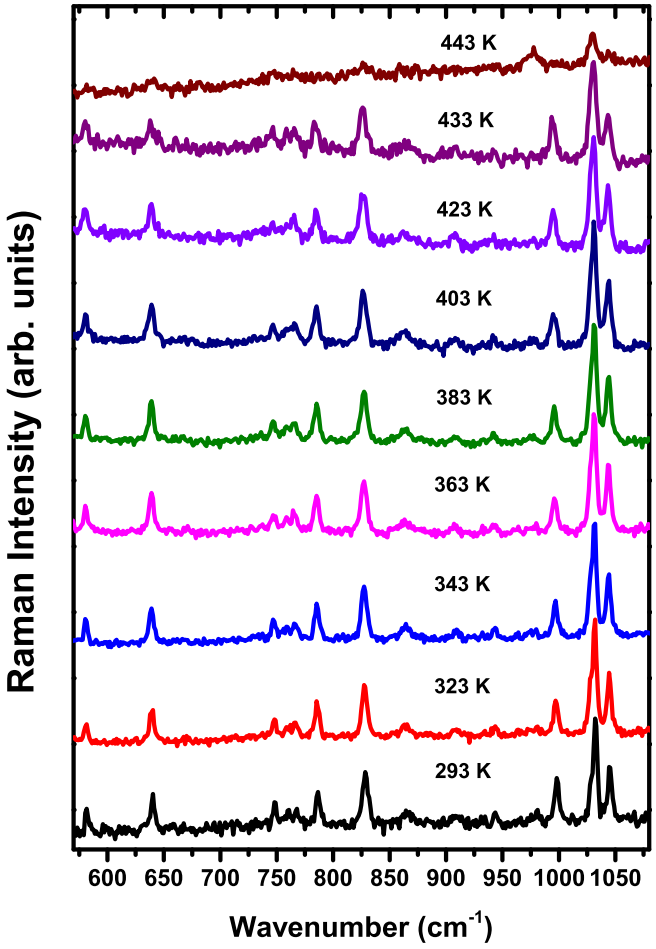

Fig. 3. Raman spectra of salophen for several temperature values in the region from 570 cm^{-1} to 1075 cm^{-1}.

antibiotics is increasing, research to find new products with antimicrobial activity is very important to decrease morbidity and mortality.

2. Experimental procedure

The synthesis process of salophen was previously reported in the literature [10]. The Raman scattering measurements were performed in a HR800 Evolution micro-Raman spectrometer from Horiba-Jobin-Yvon with a charge coupled device as light detector and the 633 nm line of a He–Ne laser as excitation source. For the high temperature measurements was used a Linkan TS1500 micro furnace. The simultaneous TG-DTA measurement were recorded in a Shimadzu DTG-60H equipment in the temperature range 303–603 K with temperature heat rate of 10 °C/min under an oxygen flow at 50 mL/min, while DSC measurement was recorded in Shimadzu DSC-60A model at the same experimental condition.

2.1. Antibacterial and modulation of antibiotic activity assays

The microorganisms employed in this study were provided by