

## Synthesis and characterization of sodium cinnamate

<sup>1</sup>Ravindran B, <sup>2</sup>Mariappan M, <sup>3</sup>Madhurambal G, <sup>1</sup>Ambikavathy S.

<sup>1</sup>Department of Physics, Thiru.Vi.Ka. Govt. Arts College, Thiruvarur, Tamilnadu, India.

<sup>2</sup>Department of Chemistry, Thiru.Vi.Ka. Govt. Arts College, Thiruvarur, Tamilnadu, India.

<sup>3</sup>Dean of Science, A D M College for Women, Nagapattinam, Tamilnadu, India.

\*Corresponding Author: E-Mail: ravitvk74@gmail.com

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### ABSTRACT

Crystals of sodium cinnamate were grown by slow evaporation of methanol solution at room temperature. Effect of sodium was studied by some analytical methods such as UV, FTIR and TG-DTA. The efficiency of SHG is more pronounced in the presence of sodium. Distribution of the electronic change were proved by the spectroscopic studies. Incorporation sodium results in increase of thermal stability ensuring the suitability of the grown crystals for NLO application upto 180°C.

**Keywords:** Solution growth, Sodium cinnamate, Single crystal, TG-DTA, UV, FTIR.

## 1. INTRODUCTION

Cinnamic acid derivatives are important metabolic building blocks in the production of lignins for higher plants in nature. Cinnamic acid composes a relatively large family of organic isomers which is a derivative of phenyl alanine [1-4]. The derivatives of cinnamic acid possesses antitumor activity and plays important pharmaceuticals for stroke prevention, high blood pressure etc [5-7]. The studies of cinnamic acid is not only for its biological activity but also for its structure. The carboxylic group is separated from the aromatic ring by a double bond and it causes conjugation between the C=C with  $\pi$  electron. The comparison is very interesting between the electronic structures of cinnamic acid and the structure of sodium cinnamate [8-12].

## 2. Experimental details

### 2.1. Sample preparation

The metal compound were prepared by mixing cinnamic acid in methanol with sodium thiocyanide in 1:1 stoichiometric ratio. The clear solution of reaction mixture is collected into a clean dry beaker after filtered by whatmann filter paper. The prepared solution was tightly covered with a perforated paper and kept in a constant temperature bath. Crystallization took place within 7 days due to slow evaporation.

## 2.2. Measurements

The FTIR spectra were recorded with a Bruker IFS spectrometer in the range of 400-4000  $\text{cm}^{-1}$ . Samples in the solid state were measured in KBr matrix pellets which were obtained with hydraulic press under 739MPa. The UV absorption spectra were recorded using a double beam UV spectrometer in the spectral range 100-400nm. The thermogravimetric and differential thermal analyses were carried out on a Netzsch STA 409C thermal analyser in the nitrogen atmosphere. The sample was heated between 30 and 1200°C at a heating rate of 10°C per minute.

## 3. Results and discussion

### 3.1. FTIR Spectral Studies

The FTIR spectra of cinnamic acid and sodium cinnamate, recorded in KBr pellet technique and in organic matrix are presented in Fig. 1 and 2. There is an intense, broad band near 3445  $\text{cm}^{-1}$  in sodium cinnamate assigned to the -ONa stretching vibration. The -CH stretching region shows a doublet at 2526 and 2359  $\text{cm}^{-1}$  (Fermi resonance).

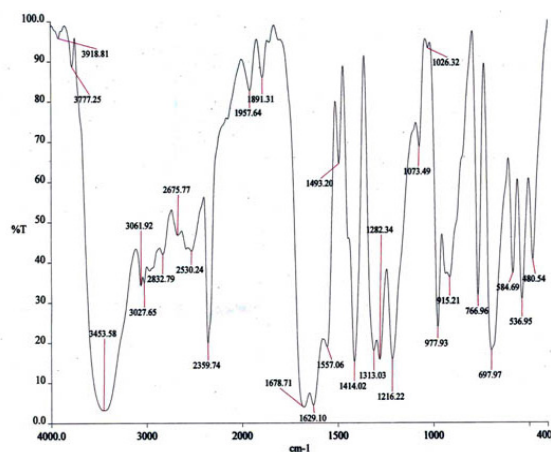


Figure -1: FTIR spectrum of Cinnamic acid.

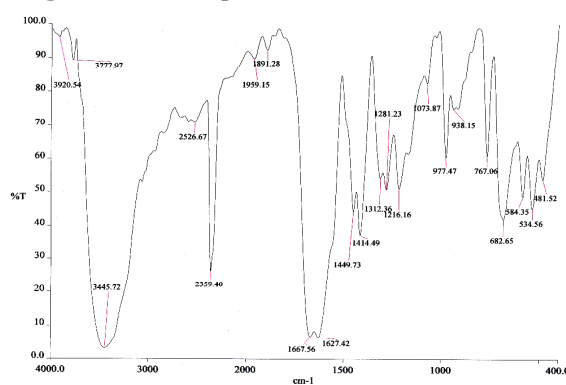


Figure - 2: FTIR Spectrum of Sodium Cinnamate.

There are splitting bands assigned to the stretching vibrations of the C=O group ( $1667$  and  $1627\text{cm}^{-1}$ ) ( $1678$  and  $1629\text{cm}^{-1}$ ) in the infrared spectra of sodium cinnamate and cinnamic acid. This increase in stretching frequencies of carbonyl group in cinnamic acid dimer indicates the formation of intermolecular hydrogen bonding between C=O.....H-O. Substitution of the carboxylic group hydrogen of cinnamic acid by a sodium metal ion causes a disappearance of the intermolecular hydrogen bonding and the characteristic changes in the IR spectra of sodium cinnamate appeared. The disappearance of bands which originate from stretching  $\gamma\text{OH}$  vibration and an appearance of bands of the asymmetric and symmetric vibration of the carboxylate anion  $\gamma_{\text{as}}(\text{COO}^-)$  were shifted towards lower wave numbers in FTIR spectra. This confirms the formation of sodium cinnamate.

### 3.2. UV spectral analysis

The UV spectra of cinnamic acid and sodium cinnamate are shown in Fig. 3 and Fig. 4. The  $\pi\text{-}\pi^*$  absorption in the sodium cinnamate spectrum on compared to cinnamic acid spectra. This shift arises due to the formation of bond between sodium with cinnamic acid. Hence the absorption shows the blue end of the spectrum

which reveals that greater energy is required for this transition.

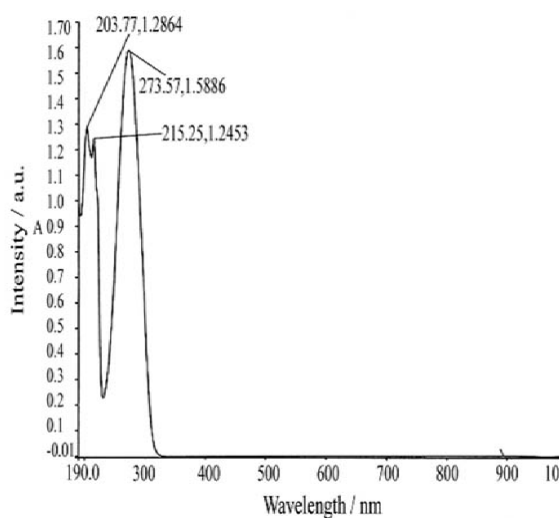


Figure -3: UV spectrum of Cinnamic acid.

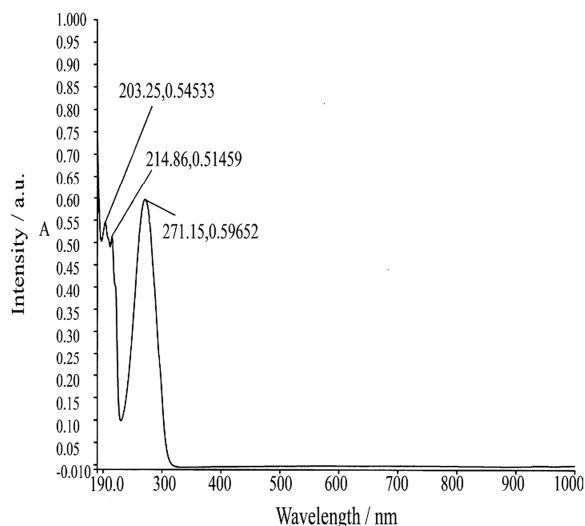


Figure - 4: UV Spectrum of Sodium Cinnamate.

### 3.3. Thermal analysis

Single crystals of cinnamic acid and sodium cinnamate were subjected to using a Netzsch STA 409C thermal analyser in the nitrogen atmosphere heated between  $30$  and  $1200^\circ\text{C}$  at a heating rate of  $10^\circ\text{C}$  per minute. The weight loss and thermal stability of the grown crystal were studied. From the Fig.5 and Fig. 6 the decomposition of cinnamic acid and sodium cinnamate were show to be a single degradation around a maximum temperature of  $180^\circ\text{C}$ . From the data it is confirmed that inclusion of sodium increases the thermal stability. This is because the replacement of hydrogen in cinnamic acid by the sodium results in increase of thermal resistance. Thus the incorporation of sodium helps to increase the thermal stability which ensures the suitability of material for NLO application around  $180^\circ\text{C}$ .

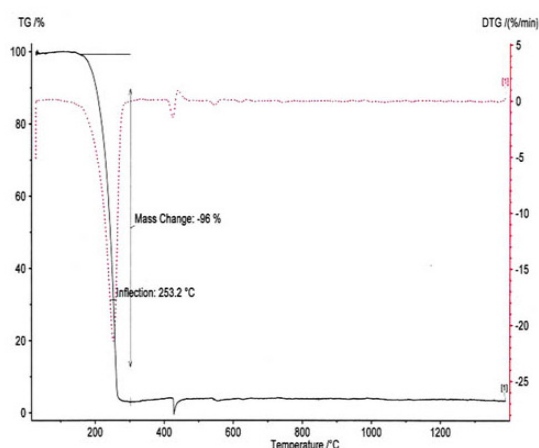


Figure - 5: TG-DTA of Cinnamic acid.

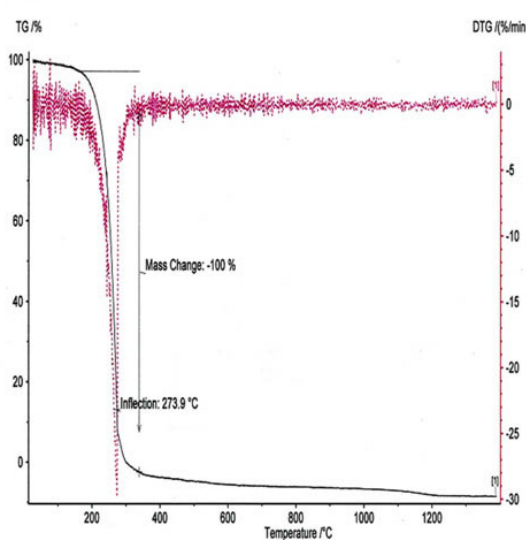


Figure - 5: TG-DTA of Sodium Cinnamate.

### 3.4. SHG

An Nd-YAG laser with modulated reaction of 1064nm was used as the optical source and was allowed to pass through the powder sample via filter. The frequency doubling was confirmed by the emission of green radiation of wavelength 532nm. The input radiation is 5.35 mj/pulse. The SHG gives an confirmation of NLO efficiency of the grown crystal. It has observed that the NLO efficiency is more pronounced in the presence of sodium.

### 4. CONCLUSIONS

The transparency of grown crystals of sodium cinnamate by slow evaporation method at room temperature were confirmed by UV and FTIR spectral analysis. FTIR spectral analysis reveals the molecular vibration and functional groups of the grown crystals. Sodium cinnamate is thermally more stable than compared to cinnamic acid. The thermal analysis confirmed that the crystals retain its texture upto 180°C. The

dopant produced catalytic effect on the NLO properties of cinnamic acid crystals.

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