GROWTH AND CHARACTERIZATION OF SUCCINIC ACID- TARTARIC ACID MIXED CRYSTALS

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ABSTRACT

Succinic acid mixed tartaric acid single crystals of good optical quality were produced from aqueous solution using the slow evaporation method. Grown crystals were used to analyse the various characterizations and disclose their properties. To confirm the compound, single crystal X-ray diffraction was used, and the obtained cell characteristics were compared to literature values. They show good transparency in visible and infrared radiation with wavelengths ranging from 200 to 1200 nm, according to UV-VIS-NIR spectra. SATA was confirmed using FTIR to identify functional groups. The thermal stability of grown SATA has been determined to be 190°C. The SHG change efficiency of the powder samples of the crystals was calculated to be about and, respectively.

Key words: Slow evaporation process, SATA, Single XRD, UV-Visible, FTIR, TGA/DSC, SHG.

I. INTRODUCTION

The crystallization of a variety of organic compound families could be useful for NLO applications in the UV and visible spectral regions. A variety of researches using inorganic, organic, and semi-organic materials for nonlinear optics have been described in recent years. The photanics applications are due to the increasing demand for low-cost, easily processible materials. Organic nonlinear materials will be critical components in future technology. For their applicability as NLO materials, a number of such materials have been documented in the literature. [1-2]. Tartaric acid, also known as dihydroxy butanedioic acid, is an important carboxylic acid in organic molecules. The photo acoustic method has gained popularity in recent years as a reliable instrument for analyzing the thermal transport properties of solid materials, particularly semiconductors. [3, 5] More than a few types of organic compounds may crystallise, which could have implications for nonlinear optical applications in the visible and UV range. It has similar optical and mechanical properties to KDP crystals [6]. L-Tartaric acid forms a nonlinear optical complex when combined with amino acids and bases [7-11]. In addition, when combined with KDP, it aids in influencing the nonlinear impact. [12]. In modern technologies, crystal growth has become increasingly important. Crystals are solids in which the atoms, the fundamental building units, are arranged in a space lattice with perfect geometrical symmetry. Single crystals have several uses in light emitting diodes, laser technology, thermography, optoelectronics, and other fields. As a result, the formation of single crystals has the greatest impact on future material research. A basic in the field of information as well as laser measurement is an optical substance.

Succinic acid mixed Tartaric Acid in this study. Single crystals of Succinic Acid mixed with Tartaric Acid were produced using a slow evaporation process. Single XRD, and UV-Visible were used to characterize the produced crystals. SHG, FTIR, TGA/DTA. This work discusses the growth, mechanical, optical, and thermal properties of the material.

II. EXPERIMENTAL METHOD

SA and TA single crystals were produced at room temperature using a slow evaporation process. The constituent chemicals were introduced to each other in a 1:1 ratio in each example. In a beaker containing doubly deionized water, equimolar amounts of Succinic and Tartaric acids were taken. The creation of succinic acid Tartaric acid is
depicted here. After 25 days, good grade crystals with fewer colours were harvested. In Figure 1, the size of the grown crystal is 19 X 8 X 6 mm.

\[
\text{C}_4\text{H}_6\text{O}_4 + \text{C}_4\text{H}_6\text{O}_6 \xrightarrow{\text{H}_2\text{O}} [\text{C}_4\text{H}_6\text{O}_4 \cdot \text{C}_4\text{H}_6\text{O}_6]
\]

![Fig : 1 As grown crystals of Succinic Acid mixed Tataric Acid](image)

### III. RESULTS AND DISCUSSION

#### 3.1 Single XRD Analysis

Single crystal X-ray diffraction measurements were performed on the crystals using an Enraf Nonius CAD-4/MACH 3 diffractometer with MOK radiation (0.71073 Å). Succinic acid and Tartaric acid single crystals were discovered to be Monoclinic in the current research. The values of the lattice parameters of the grown crystals are listed in table.

<table>
<thead>
<tr>
<th>Crystal system</th>
<th>Unit cell parameters</th>
<th>Volume</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Axial length</td>
<td>Interfacial angles</td>
</tr>
<tr>
<td>Monoclinic</td>
<td>(a = 5.09) Å (^{\circ})</td>
<td>(\alpha = 90.00) °</td>
</tr>
<tr>
<td></td>
<td>(b = 8.84) Å (^{\circ})</td>
<td>(\beta = 92.02) °</td>
</tr>
<tr>
<td></td>
<td>(c = 5.44) Å (^{\circ})</td>
<td>(\gamma = 90.00) °</td>
</tr>
</tbody>
</table>

#### 3.2 UV-VIS spectral analysis

A Perkin-Elmer Lambda-35 spectrometer was used to measure the transmittance of the produced crystals in the wavelength range of 200-1200 nm. SA and TA single crystals have optical transmittance figures of and, respectively. In two cases, a strong vibration of about 240 nm was observed. The effective transmission ranges from 240 to 1200 nm. This is required for applications that require blue/green light. It is a crucial prerequisite for the uses of NLO materials. Using the following relationship, the optical absorption coefficient (\(\alpha\)) was determined from the transmittance:

\[
\alpha = \frac{2.306 \log \frac{1}{T}}{t}
\]

Where T is the transmittance and t is the crystal thickness. For high energies, the optical energy gap, the crystal has an absorption coefficient that follows the following relationship:

\[
\alpha = \frac{A(bv - Eg)^2}{hv}
\]

Where A is a constant, the optical band gap of the crystal indicates Eg, the input photon's energy is hv, and the optical absorption coefficient is Plotting versus h (Fig 2b ) and extending the linear section towards the beginning.
of the absorption edge to the energy axis [13] were used to determine the band gap of the crystals. Succinic Acid and Tartaric Acid were discovered to have band gap energies of 5.0 and 4.7, respectively. The formed crystals of absorption spectrum are presented in (fig 2d), and it was determined from the UV absorption spectrum that there is very low absorbance throughout the visible range. It as a visible material for device fabrication because of its good optical absorbance and lower cutoff wavelength of 240 nm.

![Fig : 2a UV –VIS spectrum of Succinic Acid mixed Tartaric Acid](image-url)
Fig. 2b Tauc’s plot of SATA crystal.

Fig. 2c Photon energy Vs extinction coefficient.
3.3. FTIR analysis

Fig. 2d Optical absorption spectrum of Succinic Acid mixed Tartaric Acid

Fig 3: FTIR Spectrum of Succinic Acid mixed Tartaric Acid
Figure 3 shows the Fourier transform infrared spectra of Succinic acid mixed Tartaric Acid. The absorption of O-H stretching found shows as a broad band between 3240, 2740, 2535, and 2432 cm\(^{-1}\) in this spectrum. Table 2 shows the absorption peaks that characterize several functional groups, and they are in great agreement with published values [14-15].

Table: 2 Functional group assignments of Succinic Acid mixed Tartaric Acid

<table>
<thead>
<tr>
<th>S.NO</th>
<th>Frequency cm(^{-1})</th>
<th>Functional group assignments</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>3412.61</td>
<td>Asymmetric stretch (NH(_2) and NH(_3))</td>
</tr>
<tr>
<td>2</td>
<td>3270.72</td>
<td>O-H Stretching</td>
</tr>
<tr>
<td>3</td>
<td>2930.44</td>
<td>C-H Stretching</td>
</tr>
<tr>
<td>4</td>
<td>2740.69</td>
<td>O-H Stretch</td>
</tr>
<tr>
<td>5</td>
<td>2535.44</td>
<td>O-H Stretch</td>
</tr>
<tr>
<td>6</td>
<td>2432.62</td>
<td>O-H Stretch</td>
</tr>
<tr>
<td>7</td>
<td>1728.31</td>
<td>C=O Stretching</td>
</tr>
<tr>
<td>8</td>
<td>1693.28</td>
<td>C=O Stretching</td>
</tr>
<tr>
<td>9</td>
<td>1416.78</td>
<td>C-O-H in plane bending</td>
</tr>
<tr>
<td>10</td>
<td>1202.57</td>
<td>CO Stretching</td>
</tr>
<tr>
<td>11</td>
<td>913.32</td>
<td>Out of the plane bending of the bonded O-H</td>
</tr>
<tr>
<td>12</td>
<td>801.40</td>
<td>-C-H bending</td>
</tr>
</tbody>
</table>

3.4. TGA/DTA

In the aluminum crucible, TGA/DTA analyses were performed using SDT Q600 in the temperature range 28 - 1000°C at a heating rate of 20°C/min (Fig. 4). The compound SATA exhibits good thermal stability up to 190°C, according to the TGA curve. The material decomposes fast beyond 250°C, however the earliest decomposition states are at 190°C, which was exposed in the studies, however the existence of two endothermic peaks demonstrated that the SATA crystal thermal stability extends up to 190°C. The formed SATA crystal experiences two endothermic transitions about 190°C, according to DTA analysis. Up to 190 °C, no weight loss was observed. At 900 °C, about 90% of the weight was decomposed. TGA/DSC analysis was used to determine the thermal stability of the formed SATA crystal at a temperature of 190°C.

![Fig. 4 TG/DTA curves of SATA](image-url)
3.5. Second Harmonic Generation

The efficiency of the second harmonic generation change was reported by Kurtz and Perry [16] utilizing a customized set up. A 1064 nm fundamental beam from a Q switched Nd:YAG laser was employed with an input power of 2.3 mJ and a pulse length of 10 ns, with a repetition rate of 10 Hz. The sample's output was mono chromated so that only the second harmonic of 532 nm was accumulated. SA and TA have second harmonic signals of 4.0 and 9.5 mV, respectively. For an input pulse with energy of 1.1 mJ/pulse, the results were obtained. On the oscilloscope, the signal amplitude in milli volts represents the sample's efficiency. The conventional KDP crystals have the same input energy as that of SHG signal with a value of 10 mV/pulse. The output of the succinic acid mixed Tartaric Acid crystal was 4 mV.

IV. CONCLUSION

SATA single crystals of small size and optical transparency were successfully produced from aqueous solution using the slow evaporation approach. SA and TA crystals belonged to the monoclinic crystal system, according to single crystal XRD investigation. Using a Fourier Transform Infrared spectrum, the numerous functional groups present in the crystal were identified. The band gap UV–Visible tests revealed that the determination was also made by, and the slope obtained was evaluated as $E_g = 5$ eV. As a result, they are a good choice for optical device construction. The produced crystal exhibits good thermal stability up to 199°C, according to the thermal studies. The SATA crystal's SHG efficiency is 0.8 times that of the KDP.

REFERENCE