Spectroscopic, Optical, Thermal and Microhardness Studies of 4-FormylNitrobenzene

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Abstract— An interesting optical organic crystal of 4-FormylNitrobenzene crystals has been grown by slow evaporation solution growth technique using CCl₄ as growth medium. The solubility of 4-FormylNitrobenzene in carbon tetra chloride at various temperature ranges from 30°C to 50°C was determined. After solubility determination, the metastable zone width of 4-FormylNitrobenzene in CCl₄ was determined. The resulting crystals were subjected to FT-IR, UV-and-H1 NMR spectral studies. The purity and high thermal stability of the grown crystals were determined using Thermal analysis. Vickers microhardness studies provide information about the hardness of the grown crystal. Key words: Solution Growth, Spectral Characterization, Microhardness Studies

I. INTRODUCTION

For the past three decades, the field of opto electronics, optic communication has experienced tremendous advancements. Recently the Organic material are gain more attention due to their enormous application in opto electronic and optic communication technologies [1-5]. Organic compounds are often formed by very weak Vander walls and hydrogen bonds and possess high degree of delocalization. Hence they are optically more nonlinear than inorganic crystals. Recent researches have mentioned that organic crystals are bulk in size, hard, stable, and large. Nonlinear optical susceptibilities compared to the inorganic crystals but they have poor mechanical properties 4-FormylNitrobenzene has wide application in the very essential field of telecommunication and optical information storage devices. Slow evaporation solution growth technique has been widely used to grow organic nonlinear optical and several other types of crystals [6-11]. These crystals are used in the area of optical communication, optical information process and optical computing. Organic molecular non-linear crystals possess shorter wavelength, optical quality, sufficiently larger non-linear co-efficient spectral, structural and optical studies are important tools in the characterization of various materials. The harvested crystals were characterized by FT-IR spectral analysis, UV, X-ray diffraction (XRD), and Microhardness studies.

II. EXPERIMENTAL TECHNIQUE

The analar sample of 4-FormylNitrobenzene was further purified by repeated recrystallization. The extent of solubility of 4-FormylNitrobenzene in various solvents viz CH₃Cl, CCl₄ were determined. Finally 4-FormylNitrobenzene was grown by solution technique using CCl₄ as growth medium.

The saturated solution of 4-FormylNitrobenzene was prepared in a beaker at room temperature. Then it was allowed for systematic slow evaporation. Well needle shaped crystals were obtained in one to two days. The solubility of 4-FormylNitrobenzene in carbon tetra chloride at various temperature ranges from 30⁰ to 50⁰ was determined [2].

After solubility determination, the metastable zone width of 4-FormylNitrobenzene in CCl₄ was determined. Saturated solutions of 4-FormylNitrobenzene in CCl₄ at different temperatures were allowed for systematic slow cooling. The temperature at which the first nucleation was observed which corresponds to their width of metastable zone. The solubility of 4-FormylNitrobenzene and the metastable zone width of 4-FormylNitrobenzene in CCl₄ is shown figure 1

![Figure 1: The solubility and metastability of 4-FormylNitrobenzene](image)

Fig. 1: The solubility and metastability of 4-FormylNitrobenzene

III. RESULTS AND DISCUSSION

A. FTIR Spectral Analysis

The Infrared spectral analysis has been carried out to understand the chemical bonding and it provides useful information regarding the molecular structure of the compound [12-15]. The Fourier transform infrared (FT-IR) spectrum of 4-FormylNitrobenzene was recorded using KBr pellet technique between 400cm⁻¹ and 4000cm⁻¹ The FT-IR spectrum is shown in figure-2. The peaks at 1344cm⁻¹ and 1517.50cm⁻¹ are assigned to nitro group in aromatic ring. The peaks at 1603.52cm⁻¹ and 1576.48cm⁻¹. Further confirms the presence of aromatic ring. The peak at 2750 cm⁻¹ shows aldehyde functional group. The peaks at 529.83cm⁻¹ and 460.81cm⁻¹ Further confirms the presence of NO₂ group in aromatic ring.

![Figure 4: FT-IR spectrum of 4-FormylNitrobenzene](image)

Fig. 4: FT-IR spectrum of 4-FormylNitrobenzene

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B. UV – Visible spectral studies

UV-Visible Spectral study is very useful technique to determine the optical properties and transparency of a substance [16-21]. The UV-Visible spectrum of 4-Formylnitrobenzene crystal was recorded using Lambda 25 spectrometer is shown in figure 3. To determine the transmission range and hence to know the suitability of solution grown 4-Formylnitrobenzene crystals for optical applications, the UV – visible spectrum was recorded. The UV-visible spectrum of solution grown 4-Formylnitrobenzene is shown in figure. The spectrum further shows about 85 percent transparency of solution grown 4-Formylnitrobenzene for a wide range between 500 and 2500nm.

C. NMR Spectral Studies

NMR – spectroscopy is used to determine the molecular structure based on the chemical environment of the magnetic nuclei like H\textsuperscript{1}, C\textsuperscript{13}, P\textsuperscript{31} etc even at low concentrations. In the spectrum there are two characteristic peaks. In 4-Formylnitrobenzene there 2 sets of protons 2-ortho protons and 2-meta protons.

The doublet at 7.43 δ and 7.57 δ is assigned to two m-protons. The doublet at 8.12 δ and 8.24 δ is assigned to two o-protons [22-24].

D. Thermal Analysis

Thermal properties of harvested crystal of 4-Formylnitrobenzene were studied in powder form by recording TGA and DTA response curve in the temperature range between 0°C to 500°C. Thermal studies have been carried out using on SDTQ 600R 20.9 BUILD 20 Instrument at a heating rate of 10 °C/min under nitrogen atmosphere. The thermogram of 4-Formylnitrobenzene shows in figure 9. The DTA analysis shows a sharp peak at 103°C which corresponds to the melting point of 4-Formylnitrobenzene. There is an intense single weight loss at 160°C with a total weight loss at 430°C of about 98 %.

The thermogram further shows the thermal stability and crystalline nature of the grown crystal [25-30]. Because of the sharp endothermic peaks shows the good degree of crystallinity of the 4-Formylnitrobenzene.

E. Microhardness Analysis

Measurement of hardness is a useful nondestructive testing method to determine the hardness of the materials. The microhardness value correlates with other mechanical properties such as elastic constants and yield strength. The hardness of a material depends on different parameters such as lattice energy, Debye temperature, heat of formation and interatomic spacing [30].

Fig. 6: Microhardness values vs load for 4-Formylnitrobenzene

Vickers microhardness test was carried out on 4-Formylnitrobenzene crystal using microhardness tester fitted with a diamond indenter. The indentations were made using a Vickers pyramidal indenter for various loads. The diagonals of the impressions were measured using Shimadzu, Model HMV-2 hardness instrument.

The average diagonal length of the indented impression was calculated and the Vickers microhardness number (Hv) was found from the relation Hv=2P\sin(θ/2)/d\textsuperscript{2}. If P is the applied load (kg) and d is the average diagonal length of the indentation impressions (mm) and the angle between the opposite faces of the diamond pyramid is 0=136° then Hv in Kg/mm is given by

\[
Hv=1.8544\frac{P}{d^2} \text{ Kg/mm}^2
\]

Where, 1.8544 is a constant of a geometrical factor for the diamond pyramid

The variation of microhardness values with applied load is shown in figure 7. From the Vickers microhardness studies it observed that the hardness values increases up to load of 50g. For load above 50g crack started developing around the indentation mark which may be due to the
release of internal stresses. The presence of cracks confirms the decrease in microhardness.

\[
y = 0.2046x + 1.342
\]

\[R^2 = 0.9972\]

Fig. 8: The plot of log p vs log d

Work hardening coefficient n, a measure of the strength of the crystal is computed from the log p-log d plot and it is found to be 3.5. Onitsch [31] inferred that the values of n lies between 1 and 1.6 for hard materials and for soft materials it is above 1.6.

IV. CONCLUSION

An Intresting organic 4-Formylnitrobenzene crystal were successfully grown using slow evaporation solution growth technique, using CCl4 as a growth medium. The solubility of 4-Formylnitrobenzene in CCl4 at different temperature was determined which shows that the percentage solubility increases with temperature.

The FT-IR spectral report confirms the purity and functional group of the crystal. The UV-Visible spectrum proves the transparent nature of the crystal between 500 and 2500nm. Nuclear magnetic resonance spectrum of solution grown 4-Formylnitrobenzene also confirms purity by the presence of the peaks, which are characteristic, and in accordance with the molecular structure of 4-Formylnitrobenzene. The TGA and DTA traces confirm that the crystal is thermally stable at 103°C. Vickers Microhardness studies shows that the hardness values increases up to load of 50g. The optical transparency, thermal stability, and crystalline nature of 4-Formylnitrobenzene may be useful in deciding the application oriented properties.

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