

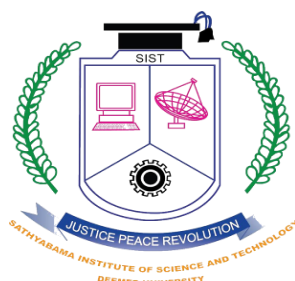
GROWTH AND CHARACTERIZATION OF CADMIUMNITRATE PHTHALATE OF NLO FAMILY CRYSTALS

Submitted in partial fulfillment of the requirements for the award of
Master of Science degree in Physics

By

V. AUXILIA

(41590002)



**DEPARTMENT OF PHYSICS
SCHOOL OF SCIENCE AND HUMANITIES**

SATHYABAMA

**INSTITUTE OF SCIENCE AND TECHNOLOGY
(DEEMED TO BE UNIVERSITY)**

**Accredited with "A" grade by NAAC I 12B Status by
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BONAFIDE CERTIFICATE

This is to certify that this Project Report is the bonafide work of A. Auxilia (41590002) who carried out the project entitled "GROWTH AND CHARACTERIZATION OF CADMIUM NITRATE PHTHALATE OF NLO FAMILY CRYSTALS" under my supervision from November 2022 to May 2023.

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DATE: 09-05-2023



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ABSTRACT

By slow evaporation technique the semi organic single crystal of cadmium nitrate phthalate was successfully grown. Single crystal X-ray diffraction revealed that the crystal structure was monoclinic with a space group. The functional groups have been identified by using Fourier transform infrared spectral analysis. TGA analysis revealed the decomposition steps, compound stability, and melting point of the crystal. The UV-VIS spectrum measures the optical transparency and band gap of grown crystals. The crystal structure is identified by the powdered X-ray diffraction. The second harmonic generation NLO behavior is determined by Kurtz Perry Powder technique. It is analyzed by using SEM. The research on crystal growth is briefly reviewed in this article, which shows how looking into the specific mechanics of crystal growth helps us understand how various different crystal morphologies form.

KEYWORDS: TGA, XRD, FTIR, UV-VIS AND SEM

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CHAPTER 1

INTRODUCTION FOR GROWTH OF CRYSTALS

1. Classification of crystals

A crystal is an infinite lattice of atoms that are arranged in patterns that repeat in all three dimension at repeated distances .In general ,a crystal may be a periodic array of atoms arranged in a three dimensional structure with uniform spacing in a particular direction. Sometimes over the course of hundreds or thousands of years, natural crystals have been produced through crystallization from solutions at relatively low temperature. For a very long period, numerous attempts have been undertaken to create good crystal of the required substance .At this time, experts in crystal growth have moved from the margins to the Center of materials based technologies.

1.1 Methods for Growing crystals

Crystal growth refers to the process of growing crystals from a supersaturated solution or a melt. There are various methods used for crystal growth, some of which include:

1. Solution growth: This is the most common method for crystal growth. In this method, the substance to be crystallized is dissolved in a solvent to form a supersaturated solution. The solution is then allowed to cool slowly, and crystals grow from the supersaturated solution as the solvent evaporates.
2. Bridgman-Stock method: This method involves slowly pulling a container of molten material through a temperature gradient. This causes the crystal to grow in a specific direction.
3. Czochralski method: This method involves melting a substance in a crucible and then slowly pulling a seed crystal through the molten material. The crystal grows as the seed is slowly pulled up and out of the melt.

4. Hydrothermal method: This method involves growing crystals in an aqueous solution at high temperatures and pressures [6]. The solution is sealed in a pressure vessel, and heat is applied to the vessel to promote crystal growth[7].
5. Vapor deposition: In this method, a vapor of the substance to be crystallized is deposited onto a substrate. The vapor is created by heating the substance in a vacuum chamber.
6. Flux method: This method involves dissolving the substance to be crystallized in a flux, such as salt. The solution is then slowly cooled, and the crystals grow from the solution.
7. Electrochemical deposition: In this method, an electric current is passed through a solution containing ions of the substance to be crystallized. The ions are attracted to an electrode, where they deposit and form a crystal.

These are just a few examples of the methods used for crystal growth. The choice of method depends on various factors such as the type of material, the desired crystal structure, and the required crystal size and shape.

1.2 Nonlinear optical crystal

A nonlinear optical crystal is a type of crystal that can modify the polarization or frequency of light passing through it. Unlike linear optical crystals, which simply refract light without changing its fundamental nature, nonlinear optical crystals exhibit a variety of nonlinear optical effects, such as second-harmonic generation, third-harmonic generation, optical parametric amplification, and optical parametric oscillation.

Nonlinear optical crystals are widely used in many areas of modern technology, including telecommunications, laser technology, optical computing, and medical imaging. They are also used in many scientific applications, such as spectroscopy, microscopy, and nonlinear optics research.

Some common examples of nonlinear optical crystals include, potassium dihydrogen phosphate, lithium iodate, beta-barium borate, etc. These crystals have different optical properties that make them suitable for different applications. For example, lithium iodate

is often used in telecommunications, while beta-barium borate is commonly used in laser technology.

1.3 Theory of Nonlinear Optics

Nonlinear optics is the study of how light interacts with matter in a nonlinear way, leading to a range of interesting and useful effects. The theory of nonlinear optics is based on the idea that the response of a material to light is not necessarily linear, but can be highly dependent on the intensity of the light.

The electric dipole approximation, polarizability approximation, and the nonlinear susceptibility model are the different theoretical model which describes the nonlinear optical response of a material. These models take into account the various ways in which light can interact with the electrons and atoms within a material, and predict the resulting nonlinear optical effects.

One of the most important effects of nonlinear optics is second harmonic generation, which occurs when a material generates a new beam of light with twice the frequency of the incident beam. This effect is widely used in laser technology and telecommunications to generate high-intensity, high-frequency light beams.

Another important effect of nonlinear optics is third harmonic generation, which generates a new beam of light with three times the frequency of the incident beam. Other effects include four-wave mixing, where two or more input beams generate a new output beam, and optical parametric amplification, here with different frequencies the input beam generates two output beams.

Nonlinear optics has a wide range of applications, including in optical communications, laser technology, materials science, and bio photonics. In advance this technologies the refinement of theoretical model and development of new nonlinear optical material place an important role.

1.4 Nonlinear optical phenomena for second harmonic generation

Second harmonic generation (SHG) is a nonlinear optical phenomenon in which a material generates a new beam of light with twice the frequency of the incident beam.

This effect is based on the nonlinear response of the material to the incident electromagnetic field.

There are several physical mechanisms that can contribute to second harmonic generation, including:

1. Electric dipole approximation: The nonlinear polarization of the material is proportional to the square of the electric field strength in electric dipole approximation. This leads to a second harmonic signal that is proportional to the square of the incident light intensity.
2. Polarizability approximation: This model assumes that the nonlinear polarization of the material is proportional to the cube of the electric field strength. This leads to a second harmonic signal that is proportional to the cube of the incident light intensity .
3. Nonlinear susceptibility model: This model takes into account the anisotropic properties of the material, which can lead to a directional dependence of the second harmonic signal.

The strength of the second harmonic signal depends on a number of factors, including the intensity and polarization of the incident beam, the nonlinear susceptibility of the material, and the crystal symmetry of the material.

Second harmonic generation is used in a wide range of applications, including in laser technology, telecommunications, and materials science. Without the need for staining or without the need for labelling, second harmonic generation microscopy can provide high resolution images of biological samples.

1.5 Nonlinear optical material

A nonlinear optical material is a material that exhibits a nonlinear response to electromagnetic radiation, such as light. Unlike linear materials, which exhibit a linear response to the electric field of the incident light, nonlinear materials modifies the frequency or polarization of the incident light and generates new frequencies of light.

Nonlinear optical materials are widely used in various fields, including telecommunications, laser technology, and materials science. Some common examples of nonlinear optical materials include:

1. Lithium niobate (LiNbO_3): This is one of the most widely used nonlinear optical materials, particularly in the telecommunications industry. It has a high nonlinear coefficient and a relatively low dispersion, making it ideal for frequency doubling and mixing applications.
2. Potassium dihydrogen phosphate (KDP): KDP is another widely used nonlinear optical material, particularly in the field of laser technology. It has a high nonlinear coefficient and a relatively low absorption, making it ideal for harmonic generation and optical parametric oscillation.
3. Beta-barium borate (BBO): BBO is a popular nonlinear optical material that is used in a wide range of applications, including frequency conversion, optical parametric oscillation, and quantum information processing.
4. Lithium triborate (LiB_3O_5 or LBO): LBO is another important nonlinear optical material, particularly in the field of frequency conversion. It has a high damage threshold and a wide range of transparency, making it suitable for high-power applications.

Other examples of nonlinear optical materials include gallium arsenide (GaAs), indium phosphide (INP), and periodically poled lithium niobate (PPLN). The choice of nonlinear optical material depends on the specific application and the desired nonlinear optical effect.

1.6 Organic nonlinear optical single crystal

Organic nonlinear optical single crystals are a type of nonlinear optical material made up of organic molecules arranged in a crystalline lattice^[8]. These materials have attracted significant attention due to their high nonlinear optical coefficients, which make them promising candidates for various photonic applications.

Organic nonlinear optical single crystals exhibit a range of nonlinear optical effects, including second harmonic generation, sum and difference frequency generation, and

optical parametric oscillation. Some common examples of organic nonlinear optical single crystals include:

1. Beta-barium borate (BBO): For growing nonlinear optical crystal BBO is used as a template even though BBO is not strictly an organic material. By growing organic molecules on the surface of BBO, it is possible to create high-quality organic single crystals with controlled size and orientation.
2. 4-Nitroaniline (4-NA): 4-NA is a common organic nonlinear optical material that has been used for second harmonic generation and sum frequency generation. It has a high nonlinear optical coefficient and is relatively easy to grow into single crystals.
3. P-Nitro Phenol (PNP): PNP is another organic nonlinear optical material that has been used for second harmonic generation and sum frequency generation. It has a high nonlinear optical coefficient and can be grown into single crystals using various methods^[9].
4. N-(4-nitrophenyl)-L-Prolinol (NPP): NPP is a chiral organic nonlinear optical material that has been used for second harmonic generation and optical parametric amplification. It has a high nonlinear optical coefficient and exhibits strong circular dichroism.

Other examples of organic nonlinear optical single crystals include urea, thio-urea, and organic nonlinear optical polymers^[10]. The choice of organic nonlinear optical material depends on the specific application and the desired nonlinear optical effect^[11].

1.7 Crystal characterization techniques

By using crystal characterization technique we can study the chemical and physical properties of crystals. Here are some common techniques used for crystal characterization:

1. X-ray diffraction (XRD): This technique is used to determine the atomic and molecular structure of crystals. X-rays are passed through a crystal, and the diffraction pattern produced is used to calculate the position and arrangement of atoms in the crystal lattice^[12].

2. Scanning electron microscopy (SEM): SEM is a technique used to study the surface morphology and composition of crystals^[13]. A beam of electrons is directed at the crystal surface, and the electrons that are scattered or emitted are detected to generate a high-resolution image.
3. Transmission electron microscopy (TEM): TEM is a technique that can be used to study the crystal structure and defects at the atomic scale. A thin slice of the crystal is bombarded with a beam of electrons, and the transmitted electrons are used to create an image of the crystal.
4. Fourier transform infrared (FTIR) spectroscopy: FTIR is a technique used to study the vibrational modes of molecules in crystals. The crystal is irradiated with infrared radiation, and the resulting absorption or transmission spectrum is used to identify the chemical composition of the crystal.
5. Raman spectroscopy: Raman spectroscopy is a technique that uses the scattering of light to study the vibrational modes of molecules in crystals. A laser is directed at the crystal, and the scattered light is analyzed to determine the chemical composition and crystal structure.
6. Thermo gravimetric analysis (TGA): TGA is a technique used to study the thermal stability and decomposition of crystals. The crystal is heated in a controlled environment, and the weight loss is measured as a function of temperature.
7. Differential scanning calorimetric (DSC): DSC is a technique used to study the thermal properties of crystals. The crystal is heated or cooled, and the heat flow is measured as a function of temperature^[14].

There are many other techniques used for crystal characterization, including optical microscopy, X-ray photoelectron spectroscopy (XPS), and atomic force microscopy (AFM). The choice of technique depends on the specific properties of the crystal being studied and the information required for the application.

1.8 Powder XRD

The crystal structure of powder material can be determined using powder x-ray diffraction (XRD). It works by directing a beam of X-rays at the powdered sample, and measuring the diffraction pattern produced when the X-rays are scattered by the atoms in the crystal lattice.

The X-ray beam is produced by an X-ray generator, and is passed through a monochromator to select a specific wavelength of X-rays. The beam is then directed onto the powdered sample, which is mounted on a flat surface called a sample holder.

The diffraction pattern produced by the scattered X-rays is detected by a detector, such as a photographic film or an electronic detector. The pattern is then analyzed to determine the positions of the atoms in the crystal lattice, as well as the lattice parameters such as the unit cell dimensions, crystal symmetry, and crystal orientation.

Powder XRD is a powerful tool for identifying and characterizing crystalline materials, including minerals, pharmaceuticals, and ceramics. It can be used to identify unknown materials by comparing their diffraction patterns to known standards in a database. Due to changes in temperature, pressure or chemical environment XRD can also be used to study changes in crystal structure.

One advantage of powder XRD is that it can be used to study samples that are not single crystals, such as polycrystalline or partially amorphous materials. Another advantage is that it is a non-destructive technique that requires very small amounts of sample, making it useful for analyzing precious or rare materials. (Fig.1) shows the picture of

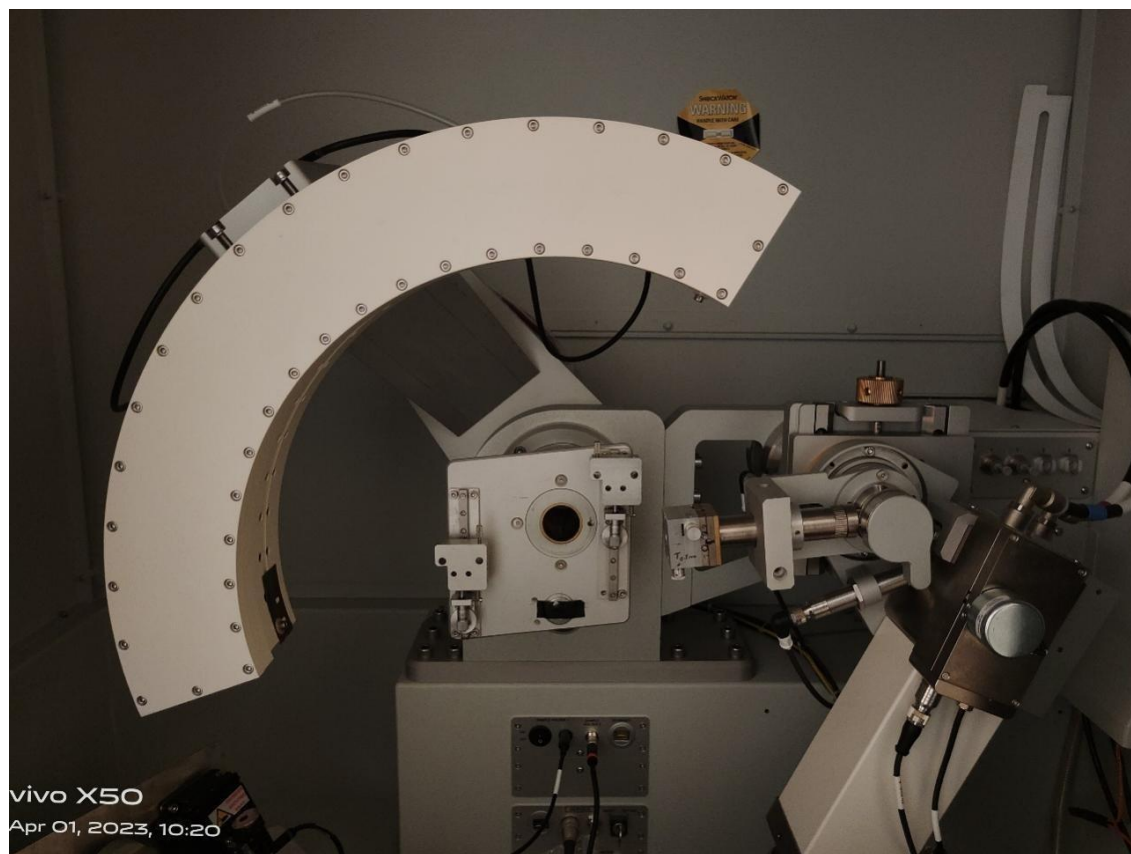


Fig .1 Powdered XRD Instrument

1.9. FTIR Spectroscopy

FTIR spectroscopy stands for Fourier Transform Infrared Spectroscopy. It is a powerful analytical technique that measures the interaction of infrared radiation with matter, providing information about the molecular vibrations and structures of the sample being analyzed.

In FTIR spectroscopy, a sample is exposed to a range of infrared frequencies, and the resulting absorption or transmission of the radiation is measured. This data is then converted into a spectrum, which shows the characteristic absorption or transmission peaks that correspond to the different molecular vibrations of the sample.

FTIR spectroscopy is widely used in a variety of fields, including chemistry, materials science, pharmaceuticals, and forensics. It is particularly useful in identifying unknown compounds and determining the chemical composition and structure of a sample. FTIR

can also be used to study chemical reactions and monitor chemical processes in real time.

FTIR spectroscopy, or Fourier Transform Infrared Spectroscopy, is a technique used to analyze the chemical composition of materials. It measures the absorption or transmission of infrared radiation through a sample, which can provide information about the functional groups and chemical bonds within the material.

The technique works by passing an infrared beam through the sample, which interacts with the sample's chemical bonds and causes them to vibrate. The resulting absorption or transmission spectrum is then analyzed to identify the chemical components of the sample.

FTIR spectroscopy is widely used in a variety of fields, including chemistry, materials science, pharmaceuticals, and environmental science. It can be used to identify unknown materials, determine the quality of a product, and monitor chemical reactions.

One advantage of FTIR spectroscopy is its non-destructive nature, meaning that the sample is not altered or destroyed during analysis. It is also a relatively fast and easy technique to use, requiring minimal sample preparation.

However, FTIR spectroscopy does have limitations. It may not be able to detect certain chemical groups, and it can be affected by the presence of water or other interfering substances in the sample. Additionally, interpreting the results requires a certain level of expertise in spectroscopy and chemistry .(Fig .2) shows the picture of

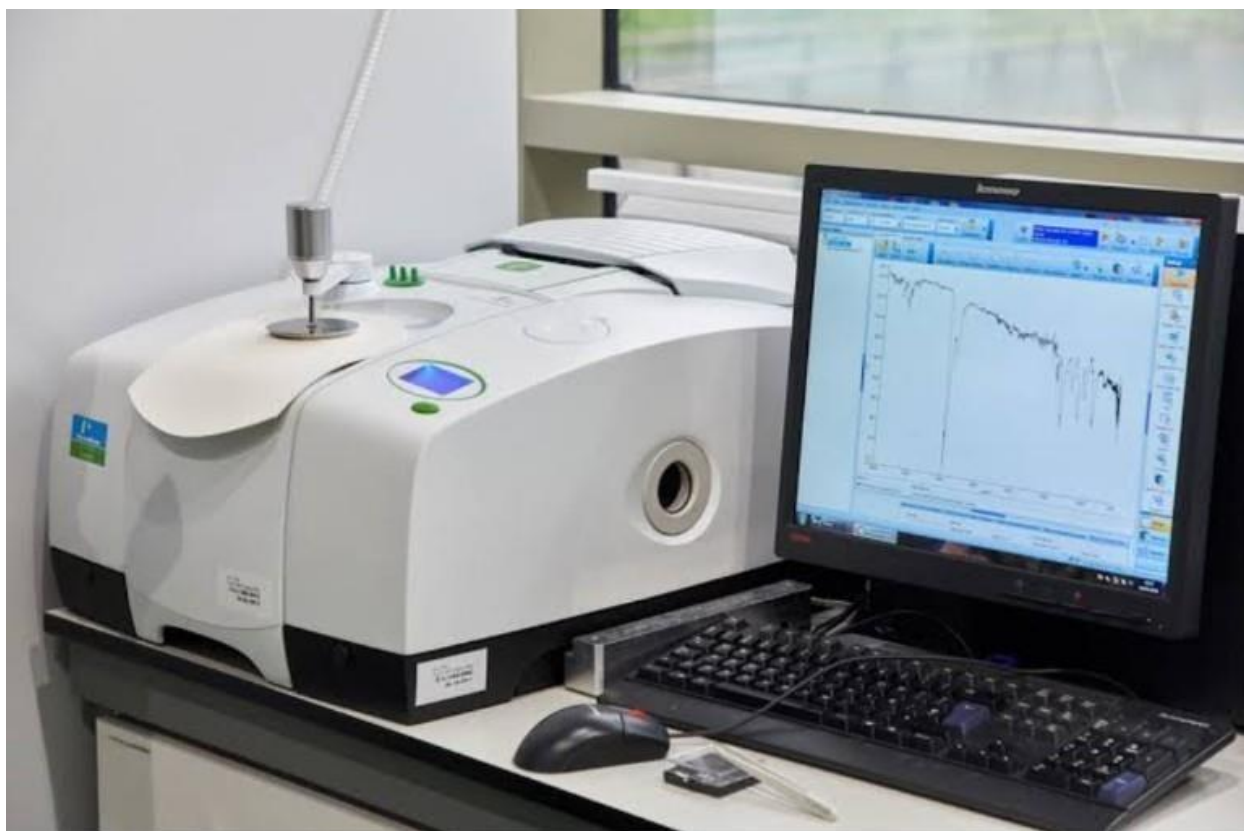


Fig. 2 FTIR Instrument

1.10 .UV-visible near infrared spectroscopy

UV-visible near infrared spectroscopy (UV-Vis-NIR spectroscopy) is a technique used to study the absorption and reflection of light by a material over a range of wavelengths, typically from the ultraviolet (UV) through visible to near-infrared (NIR) regions of the electromagnetic spectrum.

In UV-Vis-NIR spectroscopy, a sample is exposed to a broad spectrum of light, and the amount of light that is absorbed or reflected at each wavelength is measured. The resulting spectrum can provide information about the electronic and molecular structure of the sample.

UV-Vis-NIR spectroscopy is widely used in many fields, including chemistry, materials science, biology, and environmental science. It can be used to determine the concentration of a substance in a solution, identify unknown compounds, and monitor

chemical reactions. It is also commonly used in the study of optical properties of materials, including semiconductors, nanomaterial, and biomolecules.

One of the advantages of UV-Vis-NIR spectroscopy is its versatility, as it can be used to analyze a wide range of samples, from gases and liquids to solids. It is also a relatively simple and quick technique to use, requiring minimal sample preparation.

However, like other spectroscopic techniques, UV-Vis-NIR spectroscopy also has limitations. It may not be able to detect low concentrations of certain compounds, and interference from other substances can affect the accuracy of measurements. Additionally, interpretation of the results may require a certain level of expertise in spectroscopy and chemistry.

However, there are limitations to the technique. UV-Vis-NIR spectroscopy is not able to provide detailed structural information, and can be affected by the presence of impurities or other substances in the sample. Additionally, interpreting the results requires a certain level of expertise in spectroscopy and chemistry.

UV-Vis-NIR spectroscopy is a technique that measures the absorption or transmission of light in the ultraviolet, visible, and near-infrared regions of the electromagnetic spectrum. It is commonly used to determine the concentration of a substance in a solution or to study electronic transitions in molecules.

In UV-Vis-NIR spectroscopy, a beam of light is directed through a sample, and the intensity of the light that passes through the sample is measured. The amount of light absorbed by the sample is related to the concentration of the absorbing species in the solution. The resulting spectrum can be used to identify the presence of certain chemical groups or to quantify the concentration of a particular compound.

UV-Vis-NIR spectroscopy is a widely used technique in many fields, including chemistry, biochemistry, and materials science. It is relatively fast and requires minimal sample preparation, making it a convenient and non-destructive method for analyzing samples.

One limitation of UV-Vis-NIR spectroscopy is that it cannot provide detailed structural information about a compound, such as the positions of specific functional groups.

Additionally, the technique is not very sensitive to compounds with low molar absorptivities, which can limit its usefulness in certain applications.

Overall, UV-Vis-NIR spectroscopy is a powerful analytical tool that provides valuable information about the composition and properties of materials. Its versatility and ease of use make it a popular choice for researchers and analysts in a wide range of fields. (Fig.3) shown in below.



Fig .3 UV-VIS Analytical Instrument

(UV-Vis-NIR) spectroscopy is a technique used to measure the absorption, transmission, or reflection of light by a material across a range of wavelengths from ultraviolet (UV) to

near-infrared (NIR). It is commonly used to determine the concentration of chromophores or analyses in a sample or to study the electronic and vibrational properties of molecules.

The technique involves shining a beam of light onto a sample and measuring the intensity of the transmitted or reflected light as a function of wavelength. The resulting spectrum provides information about the electronic structure and composition of the sample, including the presence of chromophores such as conjugated double bonds, which absorb light in the UV-Vis range.

UV-Vis-NIR spectroscopy is widely used in a range of fields, including chemistry, biochemistry, environmental science, and materials science. It can be used for qualitative and quantitative analysis, and is particularly useful for analyzing complex mixtures or for measuring the concentration of a specific component in a sample.

One of the advantages of UV-Vis-NIR spectroscopy is its versatility, as it can be used to analyze a wide range of samples, from liquids to solids and gases. It is also a relatively simple and non-destructive technique, requiring minimal sample preparation.

1.11 THERMO GRAVIMETRY ANALYSIS (TGA)

Thermo gravimetric analysis (TGA) is a powerful analytical tool used to measure the changes in mass of a sample when exposed to a controlled atmosphere. It is used to study the thermal decomposition of materials, evaluate their thermal stability and assess the physical properties of the sample. TGA can be used to measure the changes in mass of solids, liquids, and gels as a function of temperature or time.

TGA is a technique that is used to analyze the thermal properties of a sample, such as the decomposition temperature and the thermal stability of the sample. A typical TGA setup consists of a sample holder, a thermo balance, and gas flow systems. The sample is heated in a controlled atmosphere and the changes in mass can be monitored as a function of temperature or time.

TGA is an excellent tool for characterizing the thermal stability of a material. It is also useful for characterizing the thermal decomposition of a material, such as the

decomposition temperature of a polymer. The information obtained from TGA is also useful for comparing different materials and selecting the most suitable material for a specific application. Additionally, TGA can be used to determine the thermal stability of a material, and to evaluate the physical properties of a sample, such as the melting point and the glass transition temperature.

TGA is a versatile tool for characterizing the physical and thermal properties of a material. It is a powerful analytical technique that can be used to determine the thermal stability and decomposition temperature of a material. Additionally, TGA can be used to evaluate the physical properties of the sample, such as the melting point and the glass transition temperature.

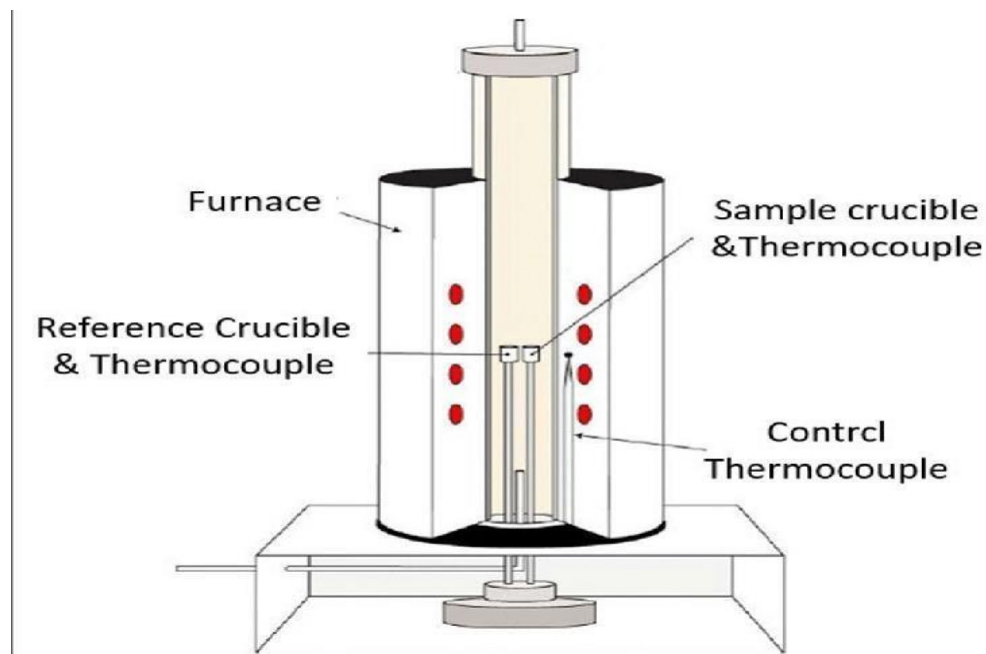


Fig 4 TGA INSTRUMENT

LITERATURE SURVEY:

1. Synthesis, growth and characterization of a new nonlinear optical crystal sodium acid phthalate

According to R. Bairava Ganesha, V. Kannana Sodium acid phthalate (NaAP), a new semi-organic nonlinear optical material, has been synthesized and single crystals were grown from aqueous solution. The FTIR spectrum confirms the compound formation. Single crystals of NaAP have been grown by slow evaporation of solvent at room temperature up to dimensions of 10 mm 5 mm 3 mm. Powder X-ray diffraction study on grown crystals shows that they belong to an orthorhombic system. The UV–Vis–NIR transmission spectrum has been recorded in the range 200–1100 nm. The second harmonic generation conversion efficiency of NaAP was determined using Kurtz powder technique; it was observed that it has double the efficiency of KAP crystals. The laser damage threshold value was determined using a Q-switched Nd: YAG laser operating at 1064 nm and with 65 ns pulses in single shot mode.

2. Studies on structural, thermal and optical properties of novel NLO crystal bis L-glutamine sodium nitrate

According to Redrothu Hanumantharao, S. Kalainathan Single crystals of novel semi-organic novel nonlinear optical material bis L-glutamine sodium nitrate (BGSN) were grown by slow evaporation technique. Single crystal XRD revealed that material crystallized in orthorhombic crystal system with non-Centro symmetric space group P212121. Molecular mass of BGSN has been measured by using mass spectroscopic analysis. Surface morphology of the grown crystal was studied by scanning electron microscopy (SEM). The presence of sodium in the compound and composition of grown crystals was confirmed on the basis of energy dispersive analysis of X-ray (EDAX). Photoluminescence study was carried out using a Xe-lamp source in the range of 250–450 nm. The thermal stability of the crystal is investigated using thermo-gravimetric analysis studies. Second harmonic generation (SHG) efficiency of the grown crystal was measured by Kurtz powder method.

3. Synthesis and crystal growth of cadmium naphthoate crystal for second order non-linear optics and cytotoxic activity

According to Natarajan Arunadevi, Ponnusamy Kanchana A new organometallic crystal diaquo-di (2-hydroxy-1-naphthoate)-cadmium (II) was synthesized by slow evaporation method and characterized for optical, spectral, thermal and biological applications. Single crystal X-ray diffraction revealed that the crystal structure was monoclinic with C2 space group. The crystal perfection and atomic packing of the grown crystal were also analyzed. The presence of functional groups and different vibrational modes were studied from IR spectra. Various decomposition stages and thermal stability of the grown crystal was studied from TG-DTA data. Molar conductance and dielectric of the complex were determined. Energy Band gap, refractive index, skin depth and extinction coefficient of the crystal were calculated from UV-Visible absorption studies and by using Kurtz powder method, the second harmonic generation (SHG) efficiency was examined. In addition, the crystal was investigated for its anticancer activity on lung cancer and breast cancer cells. Using AutoDock Vina software, the binding pattern of the crystal was investigated toward target proteins Leu 862, Glu 793, Trp 796, Gln 858, Tyr 857 and Lys 861.

4. Synthesis and Characterization of New Anhydrous Cadmium Iodate zeta polymorph $\{\text{-Cd}(\text{IO}_3)_2\text{Z}$.

According to Z. Hebboul, D. Benbertal Two methods of preparation with different routes to obtain a new anhydrous cadmium iodate $\{\text{-Cd}(\text{IO}_3)_2\}$ are presented in this paper. This polymorph has been isolated and characterized by powder XRD device, indexed in monoclinic system, with unit cell $a=8.4672 \text{ \AA}$, $b=11.4365 \text{ \AA}$, $c=14.4432 \text{ \AA}$, $\beta=91.12^\circ$.

5. Studies on the growth and characterization of L-Arginine potassium iodide crystals by slow evaporation solution growth method

According to B. Ravindran, R.S. Abina Shiny A new NLO crystal of L-Arginine doped with potassium iodide were harvested from solution growth technique with slow evaporation method. Powder X-ray diffraction analysis exposes the structure. Presence of vibration

functional groups in the grown crystal was confirmed by FTIR spectroscopy. Using the spectrum of UV–Vis-NIR the optical transmission and absorption spectra were studied. TGA-DSC studies reveal the decomposition and thermal stabilities of the grown crystals. Kurtz Perry Powder technique determines the second harmonic generation NLO behavior.

6. Synthesis, growth, structural, optical, thermal and mechanical studies on cadmium nitrate p-nitrophenol: A semi-organic nonlinear optical application

According to E. Chinnasamy, N. Indumathi The semi-organic single crystal of Cadmium nitrate p-nitrophenol (CNPN) was successfully grown by the slow evaporation technique. The functional groups have identified by using Fourier transform infrared (FT-IR) spectral analysis. The optical transparency and band gap of grown crystals were measured from UV–Vis spectrum. The thermal stability of grown crystal has been analyzed by TG/DTA studies. Dielectric studies were measured for the crystal from 50 Hz to 5 MHz at room temperature. The second harmonic generation efficiency of 1.30 times that of KDP was measured using Kurtz Perry powder method.

7. Growth and Characterization of L-Alanine Doped Thiourea Cadmium Chloride Single Crystal

According to M.Selvapandiyan L-alanine thiourea cadmium chloride single crystal has been grown by the slow evaporation method. They are identified as useful crystals for nonlinear optical applications in optical communication, optical switching, optical data storage, frequency conversion and electro- optical modulation. In this work, the grown crystal was characterized powder XRD analysis, FT- IR analysis and UV-vis-NIR analysis. From the XRD analysis the crystalline nature of the material is confirmed. The addition of L-alanine changes the crystal structure. The presence of various functional groups present into (LATCC) crystals has been confirmed by FT-IR analysis. The UV- Visible spectrum shows the cut off wavelength and forbidden gap of the crystal.

8. Influence of different solvents on the growth, thermal and dielectric properties of Phthalic acid single crystals

According to M. Suresh Kumar, G. V. Vijayaraghavan Good quality Phthalic acid single crystals were grown in aqueous solutions of ammonium oxalate and ammonium bromide by slow evaporation solution growth method. Single crystal X-ray diffraction of PA1 and PA2 was analyzed in this article. The functional groups of crystals were identified and confirmed by FT-IR analysis. UV-Vis absorption spectrum was analyzed, which shows that least absorbance in the entire visible region and the value of an optical band gap was determined. The second harmonic generation efficiency of the crystals was determined by Kurtz's powder method, which confirms non-linear optical property of the title material. The thermal stability and melting point of PA1 and PA2 crystals were found by the TGA and DTA thermal analysis. The kinetic parameters were calculated using Coats and Redfern method. The dielectric study was carried out by determining dielectric constant, dielectric loss and ac conductivity using LCR metre and the variation of these parameters with frequency and temperature was analyzed.

9. Growth, optical and thermal studies on novel nonlinear optical crystal: Glycine–phthalic acid (GPA)

According to M. Suresh Kumar, S. Krishnan Optically good and transparent glycine–phthalic acid (GPA) single crystals were grown by slow evaporation solution growth technique at room temperature. Single crystal X-ray diffraction analysis confirms that the grown crystal exhibits orthorhombic structure. The presence of functional groups was determined by FTIR analysis. Optical absorption studies reveal very low absorption in the entire visible region. The thermal stability of the title crystal was found by the TG/DTA analysis. Kurtz powder technique confirms that the SHG efficiency of the grown crystal is 0.55 times than that of KDP crystal.

10. Crystal growth, spectral, structural, optical and thermal properties of semi-organic single crystal: Tetrakis (thiourea) cadmium (II) nitrate

According to A.P. Arthi, M. Sumithra Devi The semi-organic third-order nonlinear optical tetrakis (thiourea) cadmium (II) nitrate (TTCN) single crystal was grown by solution growth method. The grown crystal was found to be in monoclinic crystal system by using single

crystal X-ray diffraction study. The spectral studies of infrared, ^1H -NMR, ^{13}C -NMR studies were used to confirm the functional groups. UV-visible spectral study was recorded in the range of 190-900 nm and found good optical transmittance in the visible region. Z-scan technique was employed to determine the nonlinear refractive index, nonlinear optical absorption and third-order nonlinear susceptibility of the grown crystal using He-Ne laser. TG-DTA analysis revealed that the decomposition steps, compound stability and melting point of the crystal.

CHAPTER 3

OBJECTIVE AND SCOPE

The objective of crystal growth is to produce large, single crystals of high purity and quality. Crystal growth is a complex process that involves the formation of a crystal from a pre-existing seed crystal or from a solution or melt. The scope of crystal growth includes the study of the mechanisms and conditions for producing crystals of good quality and the study of the properties of the crystals themselves.

In order to achieve the desired crystal quality, various parameters such as temperature, pressure, and composition of the solution must be carefully controlled. Additionally, the environment in which the crystal is grown must be carefully controlled. For example, the temperature, humidity, and air flow must be monitored to ensure an optimal crystal growth environment.

The current study focuses on

- Synthesizing the selected materials for single crystal development
- Determining the material's solubility
- Synthesis single crystal
- Determining the crystal structure by single crystal X-ray diffraction analysis.
- To determine each chemical group predominant vibrational mode by FTIR
- UV-VIS is used to identify the absorption
- TGA is used to determine the melting point
- Analyze the shape and size by using scanning electron microscopy

CHAPTER 4

STUDIES OF SYNTHESIS GROWTH AND CHARACTERIZATION OF CADMIUM NITRATE PHTHALATE NLO FAMILY OF CRYSTALS

MATERIAL SELECTION AND EXPERIMENTAL METHODS

4.1. Material selection

In order to determine the proper material selection and experimental methods for cadmium nitrate phthalate, it is important to understand the properties of the material. Cadmium nitrate phthalate is a crystalline salt composed of cadmium, nitrate, and phthalate ions. It is a white, odorless powder that has a melting point of 371.4°C. It is highly soluble in water, with a solubility of 6.2g/1000ml at 25°C. The most important property of cadmium nitrate phthalate is its solubility in water. Therefore, when selecting materials used in experiments involving cadmium nitrate phthalate, it is important to choose materials that have the same solubility as the materials themselves. This will ensure that the experiment is conducted in a manner that allows for accurate results. Additionally, it is important to be used in the experiment, as these can affect the accuracy of the results.

When conducting experiments with cadmium nitrate phthalate, it is important to use methods that are designed to accurately measure the properties of the material. This includes using spectroscopy and chromatography to measure the chemical and physical properties of the material. Additionally, it is important to consider the effects of temperature and pressure on the material when conducting experiments.

Finally, it is important to consider the safety of the experiment when working with cadmium nitrate phthalate. Safety protocols should be followed in order to ensure that the experiment is conducted in a safe and responsible manner. Additionally, it is important to consider the environmental impact of the experiment and how it could potentially affect the surrounding area. By following these guidelines, it is possible to select the proper materials and methods for conducting experiments with cadmium nitrate phthalate (CNP) in a safe and accurate manner.

EXPERIMENTAL SYNTHESIS

4.2 Synthesis

Cadmium nitrate and Phthalic acid were dissolved Millipore or distilled water in a 1:1 ratio at 30°C and mixed thoroughly using a magnetic stirrer (Fig 5). Repeated recrystallization significantly boosted the purity of the synthetic crystal. The optimum solvent for growing a single crystal through gradual evaporation was discovered to be water.



Fig 5 synthesis of CNP using magnetic stirrer

4.3 Growth

Cadmium nitrate phthalate (CNP) single crystals were used in a 1:1 ratio to create the starting material. The entire solution was then thoroughly mixed by continuously swirling it for 4 hours with a magnetic stirrer to create a homogenous mixture. The fully dissolved solution was filtered using whatmann paper to eliminate any suspended contaminants before being slowly evaporated at room temperature and on crystallizing the crystal's process. Finally, using $4 \times 3 \times 2 \text{ mm}^3$, a single crystal of well-defined cadmium nitrate phthalate was produced. The photograph of the grown crystal is shown in (Fig 6).



Fig .6 As Grown crystal of CNP

RESULT AND DISCUSSION

5.1 Powdered XRD Studies :

X-ray powder diffraction is a quick analytical method for determining a crystalline material phase and providing data on unit dimensions. The analyzed material is homogenized and finely powdered, and average bulk composition is calculated as a result. Synchrotron based microcrystal diffraction techniques were employed in this study. The characteristic features of the XRD spectrum, razor-sharp peaks at specific 2θ values, reveals the sample's crystalline composition^[16]. A steep peak appears at the values of 2θ , which are 8.9534, 21.3429, 27.1865, 28.9420, 33.0300, 40.7797 and 56.8308. CNP crystal's powder XRD pattern as shown in (Fig.7).

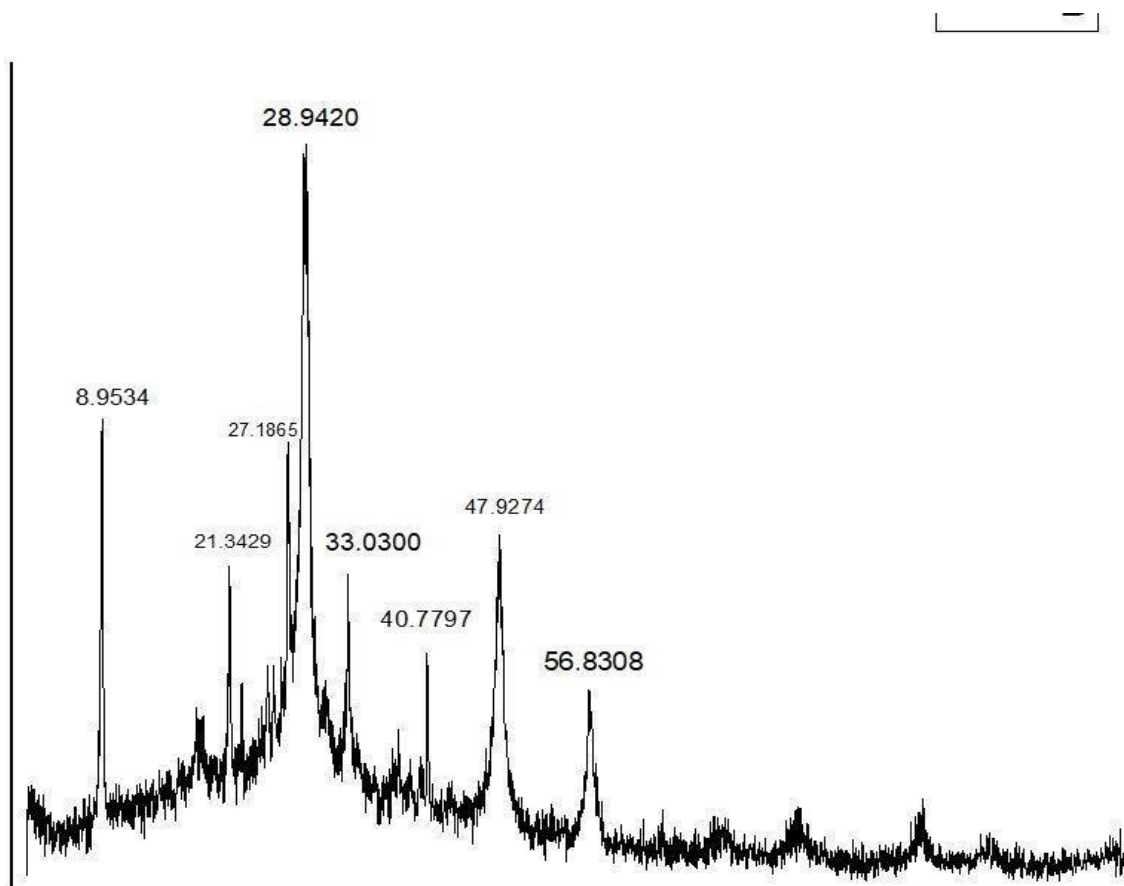


FIG. 7 XRD Pattern of CNP Crystal

TABLE: 1

Powdered XRD of Peak value and Functional group

PEAK VALUE	FUNCTIONAL GROUP
8.9534	
21.3429	
27.1865	
33.0300	
40.7797	
47.9274	

5.3 Fourier Transform Infrared Spectral Studies (FTIR):

The Fourier transform infrared spectral analysis can locate the crystal functional group. On a Perkin Elmer FTIR spectrophotometer , the cadmium nitrate phthalate crystal was captured in the wave number range 500cm^{-1} and 4000cm^{-1} . The C-O bond stretch at 1045cm^{-1} . C-O carbohydrate at 1086cm^{-1} . The O-H stretch carbohydrate proteins and polyphenols at 3346cm^{-1} ^[18]. Bending of C=O inorganic carbonate at 823cm^{-1} . C-O-H stretch at 1310cm^{-1} . The O-H stretching at 3204cm^{-1} (Fig.9) are the reported absorption peaks of cadmium nitrate phthalate crystal.

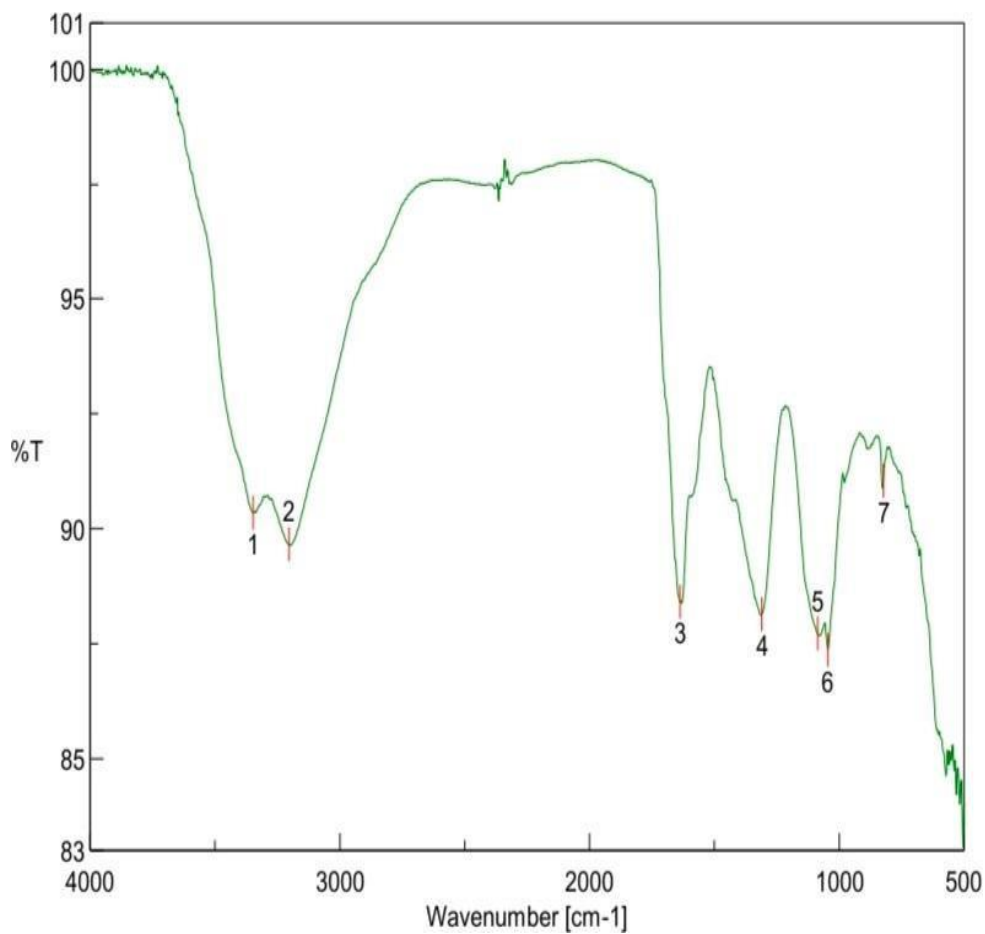


Fig 9 FTIR Spectrum of CNP Crystal

TABLE:

PEAK VALUE	FUNCTIONAL GROUP
1045	C-O BOND
1086	C-O CARBOHYDRATE
3346	OH CARBOHYDRATE PROTEINS AND POLYPHENOLS
823	BENDING C=O INORGANIC CARBONATE

1310	C-O-H STRETCH
3204	O-H STRETCHING

Thermo Gravimetry Analysis Studies (TGA):

The TGA analysis of the CNP crystal visually. TGA of grown crystal maintained at a temperature range between 0°C and 800°C. The TGA traces of the generated cadmium nitrate phthalate crystal are displayed in the figure. The TGA curve give the melting point of the sample which is 210 C^[19]. On crystallization, it was discovered that there was no physically absorbed water on the surface.

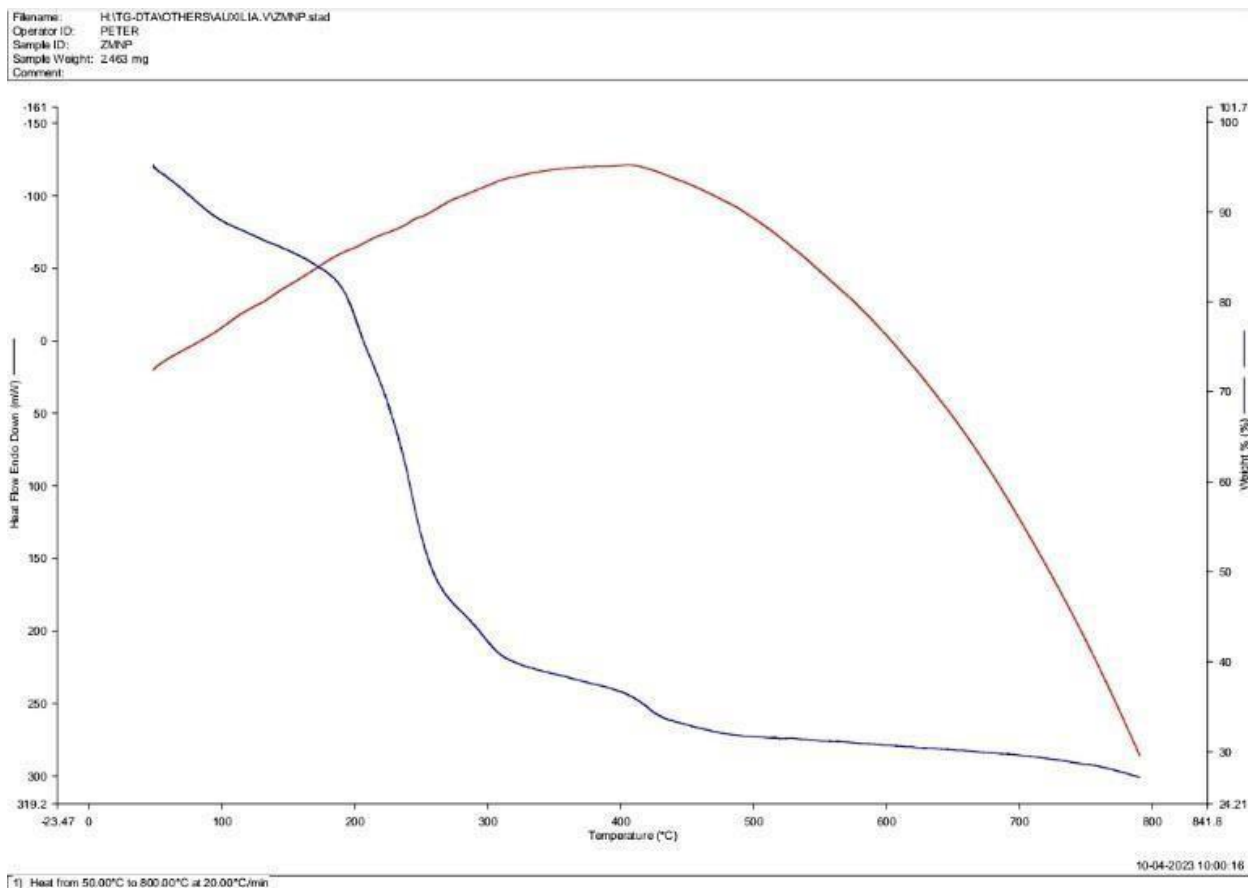
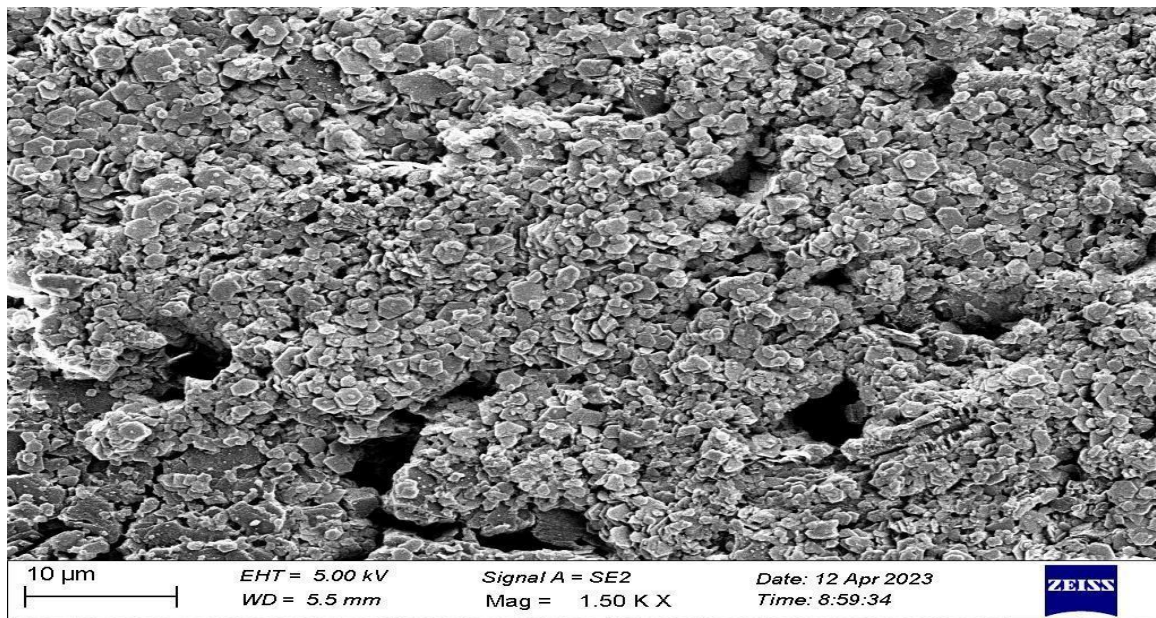


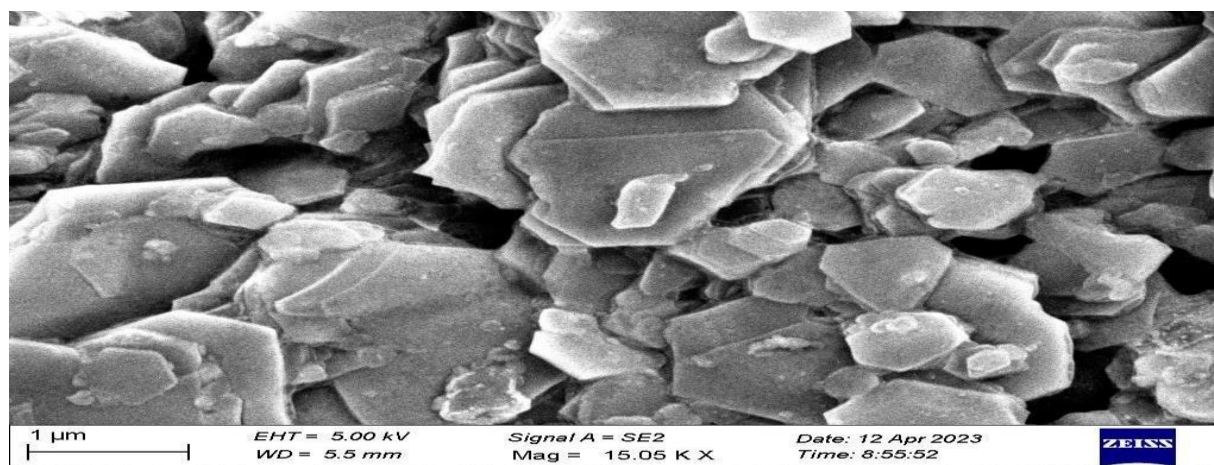
Fig. 10 TGA Graph of CNP Crystal

Non Linear Optics:

The second harmonic generation NLO test for the produced cadmium nitrate phthalate crystal takes into account the Kurtz-Perry method. Using a mortar and pestle, the grown crystal is ground into a powder, which is then loaded into a glass capillary tube with a 1.5 mm diameter. This tube is then placed into a holder, where it is made to expose the path of a very intense laser beam with a wavelength of 1064nm, and an input beam energy of 1.6mJ/pulse. The formed crystal's non-linear optical property is confirmed by the emission of green light with a wavelength and the same output efficiency as assessed in comparison to the standard KDP and found to be the efficiency on mv.

Scanning Electron Microscope(SEM):





CHAPTER 6

CONCLUSION :

By using a slow-evaporation solution growth approach, semi-organic crystals are created. The material crystal structure is discovered using powdered XRD analysis. Within the visible spectrum, the crystal is transparent. FTIR measurements provide evidence that there are many functional groups present in the sample. UV-VIS spectroscopy was used to record the optical characteristics of Cadmium nitrate phthalate in generated crystal. SEM is used to analyze the shape and size. Melting point is determined to analyze by TGA. In XRD a sharp peak at the value of 2θ which is 28.9420° . On Perkin Elmer FTIR spectrophotometer, the FTIR spectrum of Cadmium nitrate phthalate crystal was recorded in the wave number range of 500cm^{-1} to 4000cm^{-1} . FTIR spectra at wavelength

of 3346cm^{-1} is stretch OH - carbohydrate proteins and polyphenols and 3204cm^{-1} is an OH- stretching. The optical absorption investigation demonstrates that UV cut-off wavelength is 316nm. The entire visible shows that there is no significant absorption.

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