*Sujata B. Bade, International Journal of Engineering Research and Applications*
*www.ijera.com*
*ISSN: 2248-9622, Vol. 15, Issue 7, July 2025, pp 69-83*

RESEARCH ARTICLE OPEN ACCESSC

# Study of Metal-Doped Organic Nonlinear Optical Crystals in view of Synthesis, Characterization and Application's: A Review

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**ABSTRACT**
This comprehensive review paper studies the recent advances in the field of metal-doped organic nonlinear optical (NLO) crystals. Metal doping has emerged as a promising strategy for enhancing the optical properties of organic crystals, making them crucial components in modern photonics. The paper highlights the synthesis methodologies employed for producing metal-doped NLO crystals, elucidating the effects of metal incorporation on the crystal lattice, electronic structure, and charge transfer dynamics. By presenting a detailed characterization of these materials using advanced techniques, the review underscores the correlation between metal doping and improved nonlinear optical coefficients. Moreover, the review explores the multifaceted applications of metal-doped organic NLO crystals in cutting-edge technologies. It discusses their potential in laser sources, frequency conversion, optical switching, and telecommunications. The paper also sheds light on the challenges and future directions in this field, including the optimization of doping levels, exploration of new metal-dopant combinations, and tailoring crystal growth techniques to achieve desired properties.
***Keywords -***Crystal Growth, single crystal, Synthesis, Organometallic, FT-IR, Nonlinear Optical, Pl, Z-Scan.

Date of Submission: 15-07-2025 Date of acceptance: 30-07-2025

## I. INTRODUCTION

Organic materials play a crucial role in the development and optimization of nonlinear optical (NLO) crystals. These materials contribute significantly to the overall nonlinear optical response and determine the crystal's properties and potential applications. Here's how organic materials play an important role in NLO crystals. Nonlinear Optical Susceptibility: The nonlinear optical response of a crystal is determined by its nonlinear optical susceptibility, which arises from the electronic structure of the constituent molecules. Organic molecules often have large hyperpolarizabilities, making them ideal candidates for inducing strong NLO effects [1]. Centrosymmetric Breaking: Organic molecules can introduce non-centrosymmetric environments within the crystal lattice. This is essential for generating second harmonic generation (SHG) and other nonlinear effects. Organic NLO molecules disrupt the centrosymmetric of the crystal, allowing for efficient frequency conversion [2]. Electron Donor-Acceptor Systems: Many organic NLO molecules consist of electron donor and acceptor groups. This charge transfer leads to polarizations that contribute to the overall NLO response. These systems can be tailored to specific applications and desired NLO wavelengths [3]. Metals play a significant role in enhancing the nonlinear optical (NLO) properties of crystals when they are doped into the crystal lattice. The introduction of metal ions can lead to various changes in the crystal's electronic structure, symmetry, and local environment, resulting in improved NLO effects. Here are some ways in which metals play an important role in NLO crystals [4].

Electronic Structure Modification: Metal ions introduce additional energy levels within the crystal's band structure. These energy levels can interact with photons of certain frequencies, promoting transitions between energy states and enhancing the crystal's nonlinear optical response. This can lead to efficient frequency conversion processes like second harmonic generation (SHG) and third harmonic generation (THG) [5]. Applications: Metal-doped NLO crystals find applications in laser systems, frequency conversion, optical switching, telecommunications, and sensors. Their enhanced NLO responses make them valuable in various photonics technologies [6]. Phonon Effects: The presence of metal ions can influence

lattice vibrations and phonon modes within the crystal. These phonon interactions can affect the relaxation times of excited states, impacting the efficiency of NLO processes.

Overall, metals introduce new degrees of freedom to the crystal's structure and electronic properties, enabling the creation of materials with enhanced nonlinear optical effects. The synergy between metal ions and the host crystal's organic components can lead to tailored and optimized NLO materials for a wide range of applications in photonics and optoelectronics [7]. Wavelength Flexibility: Organic materials can be modified to exhibit NLO responses over a wide range of wavelengths. This flexibility is important for various applications, such as telecommunications and laser systems [8].

## II. APPLICATIONS

Organic NLO crystals find applications in various fields, including telecommunications, laser systems, optical switches, and sensors. Their unique properties make them versatile candidates for manipulating light in photonic devices. Thermal and Mechanical Properties: Organic materials often exhibit good thermal and mechanical properties, making them suitable for NLO applications where stability and durability are important. In summary, organic materials are the heart of NLO crystals. Their unique electronic structure, non-centrosymmetric properties, and ability to be tailored for specific applications make them essential components for creating efficient and effective nonlinear optical materials-doped organic nonlinear optical (NLO) crystals combine the properties of organic materials with the introduction of metal ions, leading to enhanced nonlinear optical effects. Incorporating metal ions can modify the electronic structure and properties of the organic molecules, resulting in improved NLO responses. Here's a general overview of metal-doped organic NLO crystals [9]. Metal-doped organic NLO crystals find applications in various fields like,

**Laser Systems:** These crystals can be used for frequency doubling and laser sources in telecommunication and laser machining. Optical Modulators: Metal-doped organic NLO materials can be employed in electro-optic modulators for controlling light signals in optical communication systems.

**Photonic Devices:** These materials are valuable for designing photonic devices like switches, modulators, and amplifiers.

**Sensors:** Metal-doped organic NLO crystals can be used in sensors for detecting specific wavelengths of light or changes in optical properties due to external factors.

The field of metal-doped organic NLO crystals is a promising area of research, as it combines the advantages of both organic and inorganic materials, allowing for tailored and improved nonlinear optical properties for a range of technological applications.

## III. ADVANTAGES OF METAL-DOPED ORGANIC NLO CRYSTALS

Enhanced Nonlinear Optical Response: The presence of metal ions can alter the electron distribution in the crystal, leading to enhanced nonlinear optical effects such as second harmonic generation (SHG) and electro-optic modulation. Tailored Properties: Different metal ions can be chosen to tailor the crystal's properties, including its NLO coefficients, phase-matching conditions, and response to specific wavelengths of light. Metalloporphyrin: Porphyrins are organic macrocycles with a conjugated ring system. Metalloporphyrin’s are formed when metal ions are incorporated into the porphyrin structure. These compounds have been explored for their NLO properties due to their strong electronic transitions and rich coordination chemistry. For example, zinc porphyrin derivatives doped with metal ions like copper or nickel have been investigated for their NLO responses. Metal-Doped Organic Polymers: Conjugated organic polymers doped with metal ions can exhibit enhanced NLO effects. Metal-doped polyacetylene derivatives and other π-conjugated polymers have been studied for their potential in nonlinear optical applications. Metal-Doped Organic Crystals: Organic crystals can be doped with metal ions to improve their NLO properties. For instance, metal ions like chromium, iron, or manganese have been incorporated into organic host crystals to enhance their SHG efficiency and other nonlinear optical responses. Metal-Doped Organic Nanomaterials: Nanomaterials such as metal-doped nanoparticles, nanorods, and nanowires have been explored for their enhanced NLO effects. Metal-doped nanocrystals can exhibit strong localized plasmon resonances interacting with incident light to enhance nonlinear responses [10].

## IV. EXPERIMENTAL DETAILS

Certainly, here are some detailed experimental techniques that are commonly used when studying metal-doped nonlinear optical (NLO) crystals. These techniques help researchers for characterize the crystal's structure, optical properties, and nonlinear optical responses:

**Crystal Growth:**

Metal-doped NLO crystals are typically grown using techniques like solution growth, Bridgman-Stock Barger method, or the Kochanski method.

The concentration of the metal dopant is carefully controlled during the growth process.
Growth parameters such as temperature, cooling rate, and growth direction are optimized to obtain single crystals with the desired properties.

**Single Crystal X-ray Diffraction (SCXRD):**
SCXRD is used to determine the crystal structure and confirm the presence of metal ions in the lattice.
X-ray diffraction data is collected from a single crystal, and the resulting electron density map is used to determine atomic positions and crystal symmetry.
The crystallographic information obtained helps understand how metal ions are incorporated into the crystal lattice.

**Optical Characterization:**
**UV-Vis Absorption Spectroscopy:** This technique measures the crystal's absorption of light at different wavelengths. The presence of metal ions can lead to shifts or changes in absorption bands.
**Photoluminescence Spectroscopy:** This technique measures the light emitted by the crystal upon excitation with photons. It can provide insights into electronic transitions and energy levels.
**Nonlinear Optical Measurements:**
**Second Harmonic Generation (SHG):** A focused laser beam is incident on the crystal, and the second harmonic light (twice the frequency) generated is measured. This quantifies the crystal's nonlinear response.
**Z-Scan Technique:** A focused laser beam is scanned through the crystal along the z-axis. The change in transmission or reflection provides information about the nonlinear absorption and refraction properties.
**Raman Spectroscopy:**
Raman spectroscopy measures the scattering of light by molecular vibrations. It can confirm the presence of metal ions and provide information about crystal symmetry and lattice vibrations.

**Fourier Transform Infrared Spectroscopy (FTIR):**
FTIR measures the absorption of infrared light by molecular vibrations. It can reveal functional groups and molecular interactions within the crystal.
**Temperature-Dependent Studies:**
Nonlinear optical measurements, such as SHG, can be performed at different temperatures to understand how the crystal's NLO properties change with temperature.
Thermal stability and phase transitions can also be investigated using techniques like differential scanning calorimetry (DSC).

**Microscopy and Imaging:**
**Optical Microscopy:** Optical microscopy provides images of the crystal's morphology, growth patterns, and defects.
**Scanning Electron Microscopy (SEM):** SEM offers high-resolution images of the crystal's surface, revealing its topography and features.
**Stability and Durability Tests:**
The crystal's stability and durability can be tested by subjecting it to varying environmental conditions and monitoring changes in its properties.

## V. MATERIAL CHARACTERIZATION

Techniques like X-ray diffraction (XRD) and energy-dispersive X-ray spectroscopy (EDX) can be used to verify the crystal's phase, composition, and presence of metal dopants. These experimental techniques, along with proper sample preparation and characterization, provide insights into the structural, optical, and nonlinear optical properties of metal-doped NLO crystals. Researchers combine data from these techniques to understand how the incorporation of metal ions influences the crystal's behavior and its potential for nonlinear optical applications.

**Table 1.** Reported crystals

| Compound Name | Crystal growth method | Growth duration in days | Harvested crystal size | Reference |
|---|---|---|---|---|
| l-prolinium picrate | Slow evaporation | 6 | $22*4*3mm^3$ | 1 |
| l-cystindoped zinc thiourea chloride | Slow evaporation | 15 | - | 2 |
| Ammonium malate | Slow evaporation | 20 | $38*35*27mm^3$ | |
| 3-amino propanoic acid cadmium (II) | Slow evaporation | 30 | $10*8*3mm^3$ | 8 |
| Organometallic calcium bis thiourea | Slow evaporation | 20 | $25*9*10mm^3$ | 9 |
| 2-Amino5-nitropyridinium | SR method | 60 | $85*10mm^3$ | 11 |
| Imidazolium l- tartrate | Slow evaporation | 15 | $20*10*5mm^3$ | 12 |
| Bis thiourea nickel nitrate | Slow evaporation | 14 | $42*14*7mm^3$ | 13 |
| Bis thiourea zinc chloride | Slow evaporation | 12 | $11*10*4mm^3$ | 15 |

| | | | | |
|---|---|---|---|---|
| Potassium chloride doped bis thiourea cadmium acetate | Slow evaporation | 21 | - | 16 |
| Zn 2+ doped potassium dihydrogen phosphate | Slow evaporation | 20 | - | 17 |
| Ammonium dihydrogen phosphate (ADP)with Zn2+ | Slow evaporation | 20 | 10*7*5mm$^3$ | 18 |
| Zn2+ KH2 po4 | Slow evaporation | 20 | 10*7*5mm$^3$ | 19 |
| Pristine&cd2+ potassium acid phthalate | Slow evaporation | - | - | 20 |
| Cadmium thiourea acetate doped with oxalic acid | Slow evaporation | - | - | 21 |
| KDP additive salicylic acid | Slow evaporation | 21 | 15*10*7mm$^3$ | 22 |
| l-arginine 4-nitrophenolate 4-nitro phenol | SR | 30 | 45*15mm | 23 |
| Carboxylic acid in KDP | Slow evaporation | 32 | - | 24 |
| Nd3+ on zinc tris thiourea | Slow evaporation | 29 | - | 25 |
| L-valine doped ADP | Slow evaporation | - | - | 26 |
| Carboxylic acids in KDP | Slow evaporation | 10 | - | 27 |
| Bis-thiourea zinc chloride doped KDP | Slow evaporation | - | - | 28 |
| Glycine-doped bis-thiourea cadmium formate | Slow evaporation | - | - | 29 |
| l-arginine doped zinc thiourea chloride | Slow evaporation | - | - | 30 |
| Formic acid doped KDP | Slow evaporation | 35 | 21*15*9mm$^3$ | 32 |
| Glycine doped ADP | Slow evaporation | 10 | - | 33 |
| KDP doped cadmium thiourea acetate | Slow evaporation | - | - | 34 |
| Amino acid doped ADP | Slow evaporation | 7 | - | 35 |
| l-alanine doped bis-thiourea | Slow evaporation | 16 | - | 36 |
| Glycine doped malic acid | Slow evaporation | - | - | 37 |
| Pure& doped bis-thiourea cadmium formate | Slow evaporation | 25 | - | 38 |
| Guanidinium 4-nitrobenzoate | Slow evaporation | - | - | 39 |
| Cinnamoyl proline | Slow evaporation | - | 0.21*0.20*0.18mm$^3$ | 40 |
| 4-methoxy aniline | Slow evaporation | 10 | 11*6*2mm$^3$ | 41 |
| l-ornithinium | Slow evaporation | - | - | 42 |
| Succinate salt of 8-hydroxy quinoline | Slow evaporation | - | - | 43 |
| Organic NLO OHB-T | Slow evaporation | - | 10*4*4*mm$^3$ | 44 |
| L-asparaginium oxalate | Slow evaporation | 20 | 10*5*5mm$^3$ | 45 |
| Anilinium l-tartrate | Slow evaporation | 7 | - | 46 |
| L-threonine formate | Slow evaporation | - | 15*9*3mm$^3$ | 47 |
| Three thiophenyl chalcone | Slow evaporation | - | - | 48 |
| Creatininium benzene sulfonate | Slow evaporation | - | - | 49 |
| Toluidine tartrate | Slow evaporation | - | - | 50 |
| l-prolinium picrate | Slow evaporation | - | 22*4*3mm$^3$ | 51 |
| Glycine nitrate | Slow evaporation | - | 15*11*5mm$^3$ | 52 |
| Bis 2-aminopyridinium malate | Slow evaporation | 55 | - | 53 |
| Benzaldehyde thiosemicarbazone monohydrate | Slow evaporation | 25 | 10*10*3mm$^3$ | 54 |
| 4-fluro 4-nitrostilbene | Slow evaporation | 15 | - | 55 |
| N, N'-diphenyl guanidinium L-tartrate | Slow evaporation | 90 | - | 56 |
| Pyrrolidinium p-hydroxy benzoate | Slow evaporation | 12 | 23*12*5mm$^3$ | 57 |
| 2-naphthalenol | Slow evaporation | - | - | 58 |
| l-proline doped imidazolinium l-tartrate | Slow evaporation | - | - | 59 |

**5.1 Single crystal XRD**

Single Crystal X-ray Diffraction (SCXRD) plays a crucial role in the study of metal-doped organic nonlinear optical (NLO) crystals. It provides valuable insights into the crystal structure, atomic arrangement, and bonding environment, which are essential for understanding the influence of metal dopants on the crystal's nonlinear optical properties. Here are the important roles of SCXRD in the characterization of metal-doped organic NLO crystals:

Crystal Structure Determination: SCXRD allows researchers to determine the precise three-dimensional arrangement of atoms within the crystal lattice. This is vital for understanding how metal ions are incorporated into the organic crystal matrix and how they interact with neighboring molecules. The crystal structure provides information about bond lengths, angles, and coordination environments of the metal dopants.

Confirmation of Metal Incorporation: SCXRD confirms the successful incorporation of metal ions into the crystal lattice. It helps verify the specific sites where metal ions are located and their interactions with adjacent organic molecules. This confirmation is critical for understanding how the metal dopants affect the crystal's electronic structure and properties.

Identification of Structural Changes: SCXRD can reveal any changes in the crystal's unit cell dimensions, symmetry, or orientation of molecular groups due to the presence of metal dopants. Changes in the crystal structure can affect the crystal's nonlinear optical responses.

Centro symmetry: Metal-doped organic NLO crystals often require a non-centrosymmetric arrangement for efficient nonlinear optical effects like second harmonic generation (SHG). SCXRD helps identify regions of the crystal that exhibit non-Centro symmetry due to the presence of metal ions, enabling SHG and other NLO processes.

Local Environment of Metal Ions: SCXRD provides information about the local coordination environment of the metal ions. This information is crucial for understanding how the metal dopants interact with surrounding organic molecules and contribute to the overall nonlinear optical response.

Crystal Packing and Interactions: SCXRD reveals the arrangement of molecules within the crystal lattice and their intermolecular interactions. This information helps explain how metal ions affect crystal packing and influence the crystal's optical and NLO properties.

Prediction of NLO Properties: The crystal structure obtained from SCXRD can be used to predict the crystal's nonlinear optical properties using quantum mechanical calculations. This allows researchers to understand the underlying mechanisms responsible for the observed NLO effects.

Guidance for Material Design: SCXRD data guides the design of new metal-doped organic NLO crystals by providing insights into the coordination geometries, crystal packing, and intermolecular interactions. This information is essential for tailoring crystal structures to achieve specific NLO responses.

**Table 2.** Crystal structure of reported crystals

| Compound name | Crystal structure | Unit cell parameter | Cell volume | Reference |
|---|---|---|---|---|
| Formic acid in KDP | Tetragonal | a=b=7.49, c=6.99 | 392 | 3 |
| Potassium acid phthalate | Orthorhombic | a=9.609<br>b=13.852<br>c=6.466 | - | 4 |
| L-glutamic acid hydrochloride | Orthorhombic | a=5.151<br>b=11.79<br>c=13.35 | - | 5 |
| 3-aminopropanoic acid | Monoclinic | a=9.0740<br>b=14.1239<br>c=8.0972 | 1006.90 | 8 |
| Ammonium malate | - | a=8.6378<br>b=13.7280<br>c=7.3463 | - | 7 |
| 2-Amino 5-nitropyridinium | Orthorhombic | a=25.370<br>b=6.207<br>c=5.675 | 926.58 | 11 |
| L-tartrate | Monoclinic | - | - | 12 |
| Bis-thiourea nickel | Tetragonal | a=b=7.49 | 393 | 13 |

| | | | | |
|---|---|---|---|---|
| nitrate | | c=7.00 | | |
| Potassium tetra thiourea chloride in KDP | Tetragonal | a=b=7.48<br>c=7.00 | 391 | 14 |
| KH2PO4 doped Zn2+ | Orthorhombic | a=9.54<br>b=13.873<br>c=6.470 | 861.14 | 20 |
| L-arginine 4-nitrophenolate 4-nitrophenoldihydrate | Monoclinic | a=7.869<br>b=10.376<br>c=13.832 | 1129 | 24 |
| Carboxylic acid in KDP | Tetragonal | a=b=7.45<br>c=6.99 | 388 | 25 |
| Zinc tris thiourea sulphate | Orthorhombic | a=11.142<br>b=7.749<br>c=15.619 | 1348.10 | 26 |
| L-valine doped ADP | - | a=b=7.497<br>C=7.534 | - | 27 |
| L-arginine doped zinc thiourea chloride | Orthorhombic | a=5.92<br>b=12.82<br>c=13.11 | 995 | 31 |
| Formic acid doped KDP | Tetragonal | a=b=7.48<br>c=6.59 | 389.97 | 32 |
| Glycine doped ADP | Tetragonal | a=b=7.5<br>c=7.55 | 424 | 33 |
| Bisthiourea in zinc chloride doped KDP | Tetragonal | a=b=7.47<br>C=6.98 | 389 | 29 |
| KDP doped CTA | Orthorhombic | a=7.57<br>b=11.78<br>c=15.44 | - | 34 |
| L-alanine doped BTC acetate | Orthorhombic | a=5.76<br>b=6<br>c=12.3 | 425 | 36 |
| Cinnamoyl proline | Trigonal | a=b=15.997<br>C=13.4251 | 2975.5 | 32 |
| 4-methoxyaniline | Orthorhombic | a=14.595 b=7.516<br>C=6.150 | - | 41 |
| L-asparaginium oxalate | Triclinic | a=6.34<br>b=7.27<br>c=10.56 | - | 42 |
| Anilinium | Triclinic | a=6.1496<br>b=7.4156<br>c=12.9784 | 545.9 | 47 |
| L-threonine formate | Tetragonal | a=13.61<br>b=7.73<br>c=5.25 | 552.32 | 48 |
| Toluidine tartrate | Triclinic | a=6.0956<br>b=7.4342<br>c=14.3591 | 607.01 | 50 |
| L-prolinium picrate | Monoclinic | a=10.91<br>b=5.351<br>c=12.466 | 687 | 52 |
| Glycine nitrate | Orthorhombic | a=5.601<br>b=6.090<br>c=16.373 | - | 54 |
| Bis (2-aminopyridinium | Orthorhombic | a=21.797<br>b=23.556 | 2887.8 | 55 |

| | | | | |
|---|---|---|---|---|
| maleate | | c=5.624 | | |
| Benzaldehyde thiosemicarbazone monohydrate | Orthorhombic | a=6.240<br>b=7.5820<br>c=21.129 | 21.129 | 56 |
| 4-fluoro 4-nitrostilbene | Monoclinic | a=9.494<br>b=9.864<br>c=19.501 | 1793 | 57 |
| L-tartrate monohydrate | Orthorhombic | a=7.05<br>b=14.72<br>c=19.501 | 1889 | 58 |
| Pyrrolidinium p-hydroxy benzoate | Monoclinic | a=5.98<br>b=17.94<br>c=10.22 | - | 59 |
| 2-naphthalenol | Monoclinic | a=32.054<br>b=5.887<br>c=8.178 | 151355 | 60 |
| l-proline doped imidazolinium L-tartrate | Monoclinic | a=6.68<br>b=6.92<br>c=9.67 | 465.02 | 59 |

**5.2 FTIR**

Fourier Transform Infrared Spectroscopy (FTIR) plays a significant role in the characterization of metal-doped nonlinear optical (NLO) crystals. FTIR is a powerful technique used to study the vibrational modes of molecules, providing insights into molecular structure, bonding, and interactions. In the context of metal-doped NLO crystals, FTIR can offer valuable information about how the presence of metal ions influences the crystal's vibrational properties and its potential impact on nonlinear optical responses.

Here's how FTIR plays a role in the characterization of metal-doped NLO crystals:

**Identifying Functional Groups:** FTIR spectra provide characteristic absorption bands associated with specific functional groups within the crystal's organic molecules. By analyzing these absorption bands, researchers can identify the presence of various functional groups that are crucial for nonlinear optical properties.

**Confirming Chemical Bonds:** FTIR can confirm the presence of chemical bonds within the crystal lattice. It can reveal changes in bond lengths and strengths that may result from the incorporation of metal ions, indicating potential interactions between metal dopants and organic molecules.

**Detection of Metal-Ligand Vibrations:** Metal ions often form coordination bonds with surrounding ligands. FTIR can detect vibrations associated with these metal-ligand interactions. Shifts or changes in metal-ligand vibrational modes can indicate the coordination and binding of metal ions within the crystal structure.

**Probing Crystal Symmetry and Phase Transitions:** FTIR can reveal changes in crystal symmetry and phase transitions that may occur due to the introduction of metal dopants. Such changes can have implications for the crystal's nonlinear optical properties.

**Quantifying Hydrogen Bonding:** Hydrogen bonding within the crystal lattice can affect its nonlinear optical responses. FTIR can provide information about the strength and nature of hydrogen bonding interactions, giving insights into the crystal's stability and electronic structure.

**Monitoring Molecular Conformations:** Metal dopants can influence the conformation of organic molecules within the crystal lattice. FTIR can detect changes in molecular conformations, which can impact the crystal's optical and NLO properties.

**Studying Intermolecular Interactions:** FTIR spectra can reveal interactions between neighboring molecules within the crystal lattice. These interactions can affect crystal packing and influence the crystal's nonlinear optical behavior.

**Quantifying Disorder:** The presence of metal ions can introduce disorder or distortions in the crystal lattice. FTIR can help identify and quantify these structural irregularities.

**Characterizing Polymorphism:** Different crystal forms (polymorphs) may arise due to the presence of metal dopants. FTIR can aid in distinguishing between different crystal forms and studying their respective vibrational modes.

In summary, FTIR spectroscopy is a versatile technique for investigating the vibrational properties and molecular interactions within metal-doped NLO crystals. It helps researchers understand how the incorporation of metal ions affects the crystal's structure, bonding, and vibrational behavior, all of which can have implications for the crystal's nonlinear optical responses. FTIR data complements other characterization techniques like X-ray

diffraction and optical measurements, providing a comprehensive picture of the crystal's properties.

**5.3 UV**

Ultraviolet-visible (UV-Vis) spectroscopy plays a crucial role in the characterization of metal-doped nonlinear optical (NLO) crystals. This technique provides insights into the electronic transitions and absorption properties of the crystals, especially when metal ions are introduced. Here's why UV-Vis spectroscopy is important in the study of metal-doped NLO crystals:

**Electronic Transitions and Energy Levels:** UV-Vis spectroscopy reveals the electronic transitions that occur within the crystal's molecular structure. The absorption spectrum provides information about the energy levels of electrons and the wavelengths of light that are absorbed by the crystal.

**Confirmation of Metal Dopants:** When metal ions are doped into the crystal lattice, they can introduce new electronic transitions and absorption bands. UV-Vis spectra can confirm the presence of metal dopants by identifying shifts or new peaks in the absorption spectrum that correspond to the metal ions' characteristic absorption.

**Identification of Metal-Ligand Complexes:** Metal-doped NLO crystals often involve coordination complexes between metal ions and surrounding ligands. UV-Vis spectroscopy can reveal the formation of these complexes by showing changes in absorption bands related to metal-ligand interactions.

**Quantification of Metal Concentration:** The intensity of absorption bands in the UV-Vis spectrum can be correlated with the concentration of metal dopants. This helps researchers optimize the doping levels for desired NLO effects.

**Assessment of Optical Bandgap:** The absorption edge in the UV region of the spectrum corresponds to the crystal's optical bandgap. Changes in the bandgap due to metal doping can provide insights into how metal ions influence the crystal's electronic structure.

**Resonance Enhancement:** UV-Vis spectroscopy helps identify resonant electronic transitions that are important for enhancing nonlinear optical responses. Resonance occurs when the energy of the incident light matches the energy of electronic transitions in the crystal.

**Evaluation of Conjugation and Delocalization:** Organic molecules in NLO crystals often exhibit conjugated systems and delocalized π-electrons. UV-Vis spectroscopy can show changes in the extent of conjugation or delocalization due to metal doping.

**Absorption Shifts and Splitting:** Metal dopants can induce shifts or splitting of absorption bands in the UV-Vis spectrum. These changes provide insights into how the electronic structure of the crystal is modified by the presence of metal ions.

**Photoluminescence Properties:** UV-Vis spectroscopy can also be coupled with photoluminescence measurements to study the crystal's emission properties. This is particularly useful when investigating luminescent metal-doped NLO materials.

In summary, UV-Vis spectroscopy is a valuable tool for understanding the electronic properties, energy levels, and absorption characteristics of metal-doped NLO crystals. It helps researchers assess the influence of metal dopants on the crystal's optical behavior and provides crucial information for designing and optimizing NLO materials for various photonic applications.

**5.4 SHG**

Second Harmonic Generation (SHG) is a nonlinear optical process in which two photons of the same frequency are combined within a material to generate a photon with double the frequency and half the wavelength. The efficiency of SHG in metal-doped nonlinear optical (NLO) crystals is influenced by various factors related to both the metal dopants and the host organic material. Here's how different factors play an important role in enhancing SHG in metal-doped NLO crystals:

**Centro symmetry Breaking**: Efficient SHG requires a non-centrosymmetric crystal structure. Metal dopants can induce local asymmetry, disrupting the Centro symmetry of the crystal and enabling SHG to occur. This non-centrosymmetric environment is essential for the generation of the second harmonic signal.

**Enhanced Hyperpolarizability:** Metal dopants can contribute to the overall hyperpolarizability of the crystal. The hyperpolarizability is a measure of the crystal's ability to respond nonlinearly to an applied electric field. Metal ions with large nonlinear optical coefficients can significantly enhance the SHG efficiency.

**Transition Metal Complexes:** Some metal ions, particularly transition metals, have partially filled d or f orbitals that can contribute to enhanced nonlinear optical responses. Transition metal complexes can exhibit strong electronic transitions that resonate with the incident light, leading to efficient SHG.

**Resonance Enhancement:** The presence of metal ions can lead to resonant enhancement of certain electronic transitions. When the energy levels of these transitions match the incident light's energy, the SHG efficiency can be significantly boosted.

**Local Field Effects:** Metal-doped NLO crystals can exhibit local field effects due to the interaction

between metal ions and incident light. This interaction can result in stronger polarization of electrons and enhanced SHG responses.

**Charge Transfer Transitions:** Metal-ligand charge transfer transitions can lead to large changes in dipole moments, resulting in enhanced nonlinear responses. These transitions often involve electron transfers between the metal ion and the surrounding ligands.

**Conjugation and Delocalization:** The presence of metal ions can influence the degree of conjugation and electron delocalization within the crystal. Enhanced conjugation and delocalization can lead to larger changes in electron distributions and stronger NLO effects.

**Crystal Symmetry:** Metal dopants can modify the crystal's symmetry, introducing regions of non-Centro symmetry that are crucial for SHG. These local symmetry changes allow for efficient phase matching of the SHG process.

**Phase Matching:** Achieving phase matching conditions is important for efficient SHG. Metal dopants can affect the refractive indices of the crystal, which in turn impacts the phase-matching conditions.

**Material Homogeneity:** The distribution of metal dopants within the crystal affects the homogeneity of the nonlinear response. The uniform distribution of metal ions is important for consistent and efficient SHG.

In summary, metal dopants play a critical role in enhancing SHG in NLO crystals by introducing non-Centro symmetry, modifying electronic properties, and creating an environment conducive to efficient frequency doubling. The specific effects of metal dopants depend on factors such as the type of metal, its electronic structure, coordination environment, and interactions with the host organic material. This interplay between metal dopants and organic molecules allows for the design and optimization of metal-doped NLO crystals with enhanced SHG efficiency.

**Table 3**. Comparison of SHG efficiency and fundamental UV cut-off wavelength of organometallic NLO crystals

| Crystal | UV-Vis-cut off wavelength | SHG efficiency | Reference |
|---|---|---|---|
| L-cysteine doped zinc thiourea chloride | 306 nm | 1.96 times greater than pure ZTC | 2 |
| Formic acid in KDP | Lower than 2191nm | 1.13times than of pure KDP | 3 |
| Calcium bis thiourea chloride | 1064nm | - | 9 |
| 3-aminopropanoic acid cadmium (II) | 248 | - | 8 |
| Bis-thiourea nickel nitrate of KDP | 250 | 1.98 times KDP | 13 |
| Potassium tetra thiourea chloride | 300 | 1.09 times of KDP | 14 |
| KCL doped (BTCA) | 632 | 1.27times of KDP | 16 |
| Zn2+ is doped in ADP | - | 3.15 times KDP | 19 |
| Nd3+ on zinc tris thiourea | 270 | 1.4 times ZTS | 26 |
| L-valine doped ADP | 280 | - | 27 |
| L-arginine doped zinc thiourea chloride | 235 | - | 31 |
| Glycine-doped bis-thiourea cadmium formate | 232 | 2.15 times of KDP | 30 |
| Formic acid doped potassium dihydrogen phosphate | 396 | - | 32 |
| L-Alanine doped Bis thiourea cadmium acetate | 240 | 4 times of pure BTCA | 36 |
| Glycine doped malic acid | 361 | - | 37 |
| cinnamoyl proline | 326 | 1,08 times that of KDP | 41 |

**5.5 TGA/DTA**

Thermogravimetric Analysis (TGA) and Differential Thermal Analysis (DTA) are thermal analysis techniques that are used to study the thermal behavior and stability of materials, including metal-doped nonlinear optical (NLO) crystals. These techniques provide information about weight changes, decomposition processes, phase transitions, and thermal stability. Here's how TGA and DTA play a role in the characterization of metal-doped NLO crystals:

**Thermogravimetric Analysis (TGA):** TGA measures the change in mass of a material as it is heated (or cooled) under controlled conditions. In the context of metal-doped NLO crystals:
Determination of Thermal Stability: TGA can reveal the temperature at which the crystal begins to lose weight due to decomposition or other thermal processes. This information is important for assessing the crystal's thermal stability and suitability for practical applications.
Identification of Decomposition Temperatures: TGA provides data on the temperature range at which different decomposition processes occur. This can help identify the thermal stability of both the metal dopants and the host organic material.
Quantification of Mass Changes: TGA quantifies the mass loss or gain as a function of temperature. This can be used to estimate the composition of the crystal, including the content of metal dopants.
Detection of Volatile Components: TGA can detect and quantify volatile components that may be released during heating. This is particularly relevant when studying the effects of metal dopants on crystal decomposition.
**Differential Thermal Analysis (DTA):** DTA measures the temperature difference between a sample and a reference material as they are both subjected to the same heating profile. The resulting DTA curve provides information about phase transitions, reactions, and energy changes within the sample.
Detection of Phase Transitions: DTA can identify endothermic and exothermic phase transitions, such as melting, crystallization, and phase changes related to the incorporation of metal dopants.

**Table 4.** Thermal study of reported crystals

| Compound name | % of weight loss calculated | % of weight loss observed | TGA temp range | DTA peak temp | Reference |
|---|---|---|---|---|---|
| l-cysteine doped ZTC | 11.25 | 5 | 100-400 | Endothermic 150 | 2 |
| l-leucine zinc thiourea sulphate | No loss weight | No loss weight | 100 | - | 7 |
| Aqua diiodo 3-aminopropanoic acid II | No loss weight | No loss weight | 35-600 | Endothermic 104 | 8 |
| 2-Amino-5nitropyridinium dihydrogen phosphate | 52.1 | 52 | 179-294 | Both endothermic& exothermic | 11 |
| Imidazolium L-tartrate (IMLT) | 20.8 | 20.8 | 205-250 | Endothermic 200 | |
| Bisthiourea nickel nitrate of KDP | 9.140 | - | 218-340 | Endothermic 233 | 13 |
| L-arginine 4-nitrophenolate 4-nitro phenol dihydrate | 7.3 | 7 | 30-500 | 153 | 24 |
| Carboxylic acid traits of KDP | - | - | 30-450 | Endothermic 100 | 25 |
| L-arginine doped zinc thiourea chloride | - | - | 30-400 | Endothermic 225 | 31 |
| Cinnamoyl proline | - | 4.5 | 420-504 | Endothermic 375.7 | 41 |
| 4-methoxyaniline | - | - | 100-175 | Exothermic peak at 61 | 42 |
| L-ornithinium dipicrate | - | - | 205-244 | Endothermic at 211 | |
| 8-hydroxy quinoline | - | - | 145-200 | Endothermic at 45 | 44 |
| OHB-T | - | - | 250-400 | Endothermic at 264 | 45 |
| L-asparaginium oxalate | - | - | 35-500 | Endothermic at 111 | 46 |
| L-prolinium picrate | 6.36 | 6.13 | 30-1000 | Endothermic at 250 | 53 |
| Glycine nitrate | 20.62 | 20.31 | 146-200 | Endothermic at 145 | 54 |
| Bis(2-aminopyridinium) | - | - | 105-210 | Endothermic at 137 | 55 |
| Benzaldehyde thiosemicarbazone | 1.745 | 1.50 | 220-400 | Endothermic at 153 | 56 |

Characterization of Heat Capacity: The areas under the DTA peaks provide information about the heat capacity changes associated with different thermal events. This can reveal insights

into the stability and interactions of metal-doped NLO crystals.

Assessment of Crystal Purity: DTA can detect impurities or changes in crystal purity based on shifts in phase transition temperatures or the appearance of additional peaks.

Verification of Crystallographic Changes: DTA can help confirm if the crystal lattice undergoes structural changes, as these changes can manifest as distinct thermal events.

Role in Metal-Doped NLO Crystals: TGA and DTA provide critical information about the thermal stability, decomposition behavior, and phase transitions of metal-doped NLO crystals. This information is essential for understanding how metal dopants influence the crystal's thermal properties and stability. Researchers can use TGA and DTA data to optimize crystal growth conditions, assess the effects of metal doping on crystal decomposition, and ensure the suitability of metal-doped NLO crystals for specific applications that involve varying temperature ranges.

By combining TGA, DTA, and other characterization techniques like X-ray diffraction, UV-Vis spectroscopy, and nonlinear optical measurements, researchers can develop a comprehensive understanding of the structural, optical, and thermal properties of metal-doped NLO crystals.

**5.6 PL**

Photoluminescence (PL) is a spectroscopic technique that involves the emission of light (photons) from a material following the absorption of photons. In the context of metal-doped nonlinear optical (NLO) crystals, photoluminescence plays a crucial role in understanding the electronic transitions, energy levels, and luminescent properties of the material. Here's how PL plays an important role in the study of metal-doped NLO crystals:

Photoluminescence in Metal-Doped NLO Crystals:

**Emission Spectra:** Photoluminescence spectra provide information about the energy levels and electronic transitions within the crystal's molecular structure. The emitted light can originate from various electronic states, and the PL spectrum reflects the energy differences between these states.

**Electronic Structure:** Metal dopants can introduce new energy levels and electronic transitions within the crystal. PL spectra can help identify the contributions of metal-related electronic transitions and assess their influence on the crystal's overall optical properties.

**Confirmation of Metal Incorporation:** Metal-doped NLO crystals may exhibit distinct emission peaks associated with metal-related electronic transitions. The presence of these peaks in the PL spectrum confirms the successful incorporation of metal ions into the crystal lattice.

**Luminescent Centers:** Metal dopants can act as luminescent centers, contributing to the emission of light. PL can reveal the energy levels and transitions associated with these luminescent centers.

**Charge Transfer States:** Metal-ligand interactions in metal-doped NLO crystals can lead to charge transfer states with unique emission properties. PL spectra can provide insights into the nature of these charge transfer processes.

**Localized and Delocalized States:** Metal dopants can influence the distribution of electronic states within the crystal. PL spectra can help distinguish between localized and delocalized electronic states and provide information about their contributions to emission.

**Importance of PL in Metal-Doped NLO Crystals:**

Electronic Structure Investigation: PL spectroscopy allows researchers to investigate the electronic structure of metal-doped NLO crystals, providing insights into the energy levels and transitions that are relevant for nonlinear optical processes.

Characterization of Metal-Ligand Interactions: PL can help identify interactions between metal ions and surrounding ligands. Changes in PL emission can provide information about changes in coordination environments or bonding interactions due to metal doping.

Quantitative Analysis: PL intensity can be correlated with the concentration of luminescent species, which may include metal dopants. This allows for the quantification of metal content within the crystal.

Optimization of Crystal Properties: PL data can guide the optimization of metal dopant concentrations, crystal growth conditions, and post-processing treatments to enhance the crystal's luminescent properties and overall nonlinear optical behavior.

Monitoring Material Quality: PL can serve as a diagnostic tool to monitor the quality and homogeneity of metal-doped NLO crystals. Variations in emission characteristics can indicate defects or inconsistencies in crystal growth.

Applications in Photonic Devices: Understanding the luminescent properties of metal-doped NLO crystals is crucial for designing photonic devices that involve light emission, such as lasers, light-emitting diodes (LEDs), and optical amplifiers.

In summary, photoluminescence spectroscopy is a valuable technique for investigating the electronic structure, energy levels, and luminescent properties of metal-doped NLO crystals. It offers insights into how metal dopants influence the crystal's optical behavior and provides

essential information for designing and optimizing materials for nonlinear optical and luminescent applications.

**5.7 Z-scan**

The Z-scan technique is a powerful experimental method used to characterize the nonlinear optical (NLO) properties of materials, including metal-doped organic NLO crystals. It provides valuable information about the material's response to intense light, especially when metal dopants are introduced. The Z-scan technique helps researchers understand how the material interacts with different light intensities and provides insights into its NLO behavior. Here's how the Z-scan technique plays an important role in studying metal-doped organic NLO crystals. The Z-scan technique involves scanning a sample through the focus of a laser beam while measuring the resulting transmitted or reflected light intensity. The sample's nonlinear response is characterized by changes in light intensity due to nonlinear processes such as self-focusing, self-defocusing, and nonlinear absorption. The Z-scan can be performed in both open-aperture and closed-aperture configurations.

Role in Metal-Doped Organic NLO Crystals:

Nonlinear Absorption: Metal-doped NLO crystals can exhibit enhanced nonlinear absorption due to the presence of metal ions. The Z-scan technique allows researchers to quantify the nonlinear absorption coefficient and study how metal dopants influence this behavior.

Two-Photon Absorption (TPA): Metal dopants can contribute to enhanced two-photon absorption, a nonlinear process where two photons are simultaneously absorbed to induce a single transition. The Z-scan can provide information about TPA cross-sections and their dependence on metal dopant concentration.

Determination of Nonlinear Refractive Index: The Z-scan technique allows researchers to determine the material's nonlinear refractive index. Metal dopants can modify the refractive index due to their influence on the electronic structure. This information is crucial for designing devices such as optical switches and modulators.

Study of Self-Phase Modulation: The Z-scan technique can reveal self-phase modulation effects, where the refractive index changes with light intensity. This effect can be influenced by metal dopants and is relevant for ultrafast nonlinear optical processes.

Observation of Beam Defocusing: The Z-scan technique can detect self-defocusing behavior caused by nonlinear effects. The presence of metal dopants can modify the extent of self-defocusing, leading to altered NLO responses.

Nonlinear Focusing and Saturation: Metal dopants can lead to changes in nonlinear focusing behavior and saturation intensities. The Z-scan technique provides insights into how metal dopants impact these aspects of nonlinear optics.

Importance of Z-Scan in Metal-Doped Organic NLO Crystals:

Quantitative NLO Characterization: The Z-scan technique provides quantitative measurements of NLO parameters, such as nonlinear absorption coefficients and nonlinear refractive indices. These parameters are critical for assessing the material's potential for NLO applications.

Optimization of Metal Dopants: The Z-scan results can guide the optimization of metal dopant concentrations, crystal growth conditions, and post-processing steps to achieve desired NLO effects.

Material Comparison: The Z-scan technique allows for the comparison of different metal-doped organic NLO crystals, helping researchers identify materials with superior nonlinear optical properties.

Device Design: Understanding the Z-scan behavior of metal-doped organic NLO crystals is essential for designing photonic devices that utilize their nonlinear properties, such as frequency converters, optical limiters, and modulators.

Table 5. Optical third order nonlinearity data of crystals

| Crystal | $n^2$ | β | Im ($X^3$) | Re($X^3$) | $(X)^3$ | Reference |
|---|---|---|---|---|---|---|
| Formic acid KDP | 0.11 | $1.5*10^{-7}$ | $10^{-2}$ | $10^{-4}$ | $10^{-7}$ | 3 |
| 3-aminopropaboic acid (cadmium (II)) | $2.4*10^{-11}$ | $1.004*10^{-4}$ | 3.24*10-8 | $1.5*10^{-9}$ | 3.24*10-8 | 8 |
| 2-Amino-5-nitro pyridinium dihydrogen phosphate | $9.74*10^{-8}$ | $0.33*10^{-4}$ | $0.04*10^{-6}$ | $2.07*10^{-6}$ | $5.52*10^{-6}$ | 11 |
| Bis-thiourea nickel nitrate | $1.51*10^{-9}$ | $5.57*10^{-6}$ | $10^{-2}$ | $10^{-4}$ | $1.77*10^{-5}$ | 6 |
| Potassium tetra thiourea chloride on KDP | $2.25*10^{-5}$ | $8.99*10^{-8}$ | $1.32*10^{-5}$ | $1.32*10^{-5}$ | $2.13*10^{-7}$ | 14 |

| | | | | | | |
|---|---|---|---|---|---|---|
| Zn2+ in ADP | $5.44*10^{-12}$ | $4.5*10^{-7}$ | - | - | $1.14*10^{-6}$ | 19 |
| L-arginine doped ZTC | $4.45*10^{-9}$ | $8.2*10^{-6}$ | - | - | $2.43*10^{-3}$ | 22 |
| L-valine doped ADP | $4.5*10^{-12}$ | $5.79*10^{-6}$ | - | - | $2.249*10^{-5}$ | 27 |
| Glycine doped ADP | $-4.55*10^{-13}$ | $5.26*10^{-7}$ | - | - | $4.84*10^{-6}$ | 33 |
| Carboxylic acid on KDP | $7.92*10^{-5}$ | $2.13*10^{-7}$ | - | - | $1.90*10^{-7}$ | 28 |
| Malic acid cinnamoyl proline | - | $3.307*10^{-30}$ | - | - | $3.45*10^{-9}$ | 41 |

In summary, the Z-scan technique plays a vital role in characterizing the nonlinear optical responses of metal-doped organic NLO crystals. It helps researchers understand how metal dopants influence the crystal's nonlinear behavior and provides essential data for designing and optimizing materials for various photonic applications.

## VI. CONCLUSIONS

In this research, all the crystal was analyzed were grown by the slow evaporation and SR method. It is observed that metal doping has emerged as a promising strategy for enhancing all the optical properties. In this paper detailed characterization study was done for the group of amino acid and organic crystals. The metal doping enhances the nonlinear optical coefficients. Moreover, the review explores the multifaceted applications of metal-doped organic NLO crystals in cutting-edge technologies. It discusses their potential in laser sources, frequency conversion, optical switching, and telecommunications. Overall, this review paper serves as a comprehensive resource for researchers, scientists, and engineers. It provides an overview on synthesis, characterization and applications, highlighting key developments and offering insights into the potential for these materials to revolutionize the realm of nonlinear optics.

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