



STUDIES ON SYNTHESIS AND CHARACTERIZATION OF METAL COMPLEXES

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ABSTRACT

Diphenylamine metal complexes were synthesized using ligand diphenylamine, Zn (II) sulphate and Cinnamic acid (1) Glycine (2) resulted in the form of solids characterized by FTIR Spectroscopy and UV-Visible spectral analysis. FTIR Spectral studies indicated that the ligands coordinate through nitrogen and oxygen. The spectroscopic data of complexes confirmed an octahedral geometry around the central metal ion with water molecules occupying the coordination sphere. Optical window width is also used to characterize the material.

KEYWORDS: Ligands, synthesis, Spectral characterization.

INTRODUCTION

A crystal is a solid material whose constituent atoms, molecules, or ions are arranged in an orderly repeating pattern extending in all three spatial dimensions. Crystal growth is a major stage of a crystallization process, and consists in the addition of new atoms, ions, or polymer strings into the characteristic arrangement of a crystalline Bravais lattice.

The regular surface geometry and the shiny and often colourful appearance have made crystals from the mineral kingdom fascinating objects for everybody. Natural crystals have often been formed at relatively low temperatures by crystallisation from solutions, sometimes in the course of hundreds and thousands of years.

Nowadays, crystals are produced artificially to satisfy the needs of science, technology and jewellery. The ability to grow high quality crystals has become an essential criterion for the competitiveness of nations. Crystal growth specialists have been moved from the periphery to the center of the materials-based technology.

An interdisciplinary crystal growth science has developed with scientific journals, conventions and societies. International networks of crystal growth laboratories and materials science centres have been formed. Crystal laboratories operate in large numbers to satisfy the needs of research and technology for high-quality, tailor-made crystals of all kinds.

Most solids consist of many single crystals of different orientations which stick together at "grain boundaries".

Binding forces are usually weaker at grain boundaries. Therefore, chemical reactions and evaporation processes proceed more easily at these boundaries which make them visible to the naked eye. Usually, single crystals not only contain point defects but also extended defects, namely dislocations and dislocation networks.

In research and technology many artificial crystals are required with chemical compositions from all parts of the periodic system with high chemical and - in special cases - even isotopic purity. Roughly speaking, the artificial crystal kingdom can be divided into three sectors.

Technical crystals belong to one of the two big sectors of the single crystal market. They are widely present, often in hidden form. We eat crystals (salt, sugar), we use crystals as clocks in watches and computers (quartz), for information processing and storage (silicon), for switching TV-sets (gallium arsenide), for telecommunication (gallium arsenide) and for transport (turbine blades from nickel-aluminium compounds). Huge salt crystals (CaF₂) are used as UV-light lenses in the submicron structuring during electronic device fabrication.

Jewellery forms the second big sector of the single crystal market. Verneuil-rubies have been the first artificial crystals which have been grown on an industrial scale to be used in making jewellery and as bearings in mechanical watches "Falkenberg".

Natural crystals are usually much more expensive than artificial crystals of the same kind. They often can be distinguished only by sophisticated characterisation

methods, not obvious for the naked eye. The excessively high costs of certain natural crystals has been an enormous incentive for clever crystal growers to adjust their growth procedures until artificial crystals cannot be distinguished from natural ones in every detail of their microstructure.

The market of research crystals is relatively small but extremely diversified. Artificial research crystals of high quality are the basis of solid state research activities. Natural crystals are normally not sufficiently qualified for research purposes. Crystals are also required for modern light and particle scattering and diffraction instruments as monochromators an essential prerequisite for success in crystal growth is the availability of material of the highest purity. Solute and solvents of high purity are required, since impurity may be incorporated into the crystal lattice resulting in the formation of flaws and defects. Sometimes impurities may slow down the crystallization process by being adsorbed on the growing face of the crystal, which changes the crystal habit. A careful repetitive use of standard purification methods of recrystallization followed by filtration of the solution would increase the level of purity. Some materials introduces water of crystallization to others which may be desired in the anhydrous form. Therefore, for the growth of crystals of a compound from solution, the selection of a solvent is critical. For example, crystals of nonpolar organic compounds can be grown easily from nonpolar organic solvents Chemical and detectors. A broad range of geometrically well prepared crystals is required for thin film, catalysis and electrochemical studies.

Zn (II) ion plays a variety of roles like enzyme function, gene expression, and hormone receptors and in storage of protein in the human body.

MATERIALS AND METHODS

Material

Diphenylamine, cinnamic acid, glycine, zinc sulphate were procured and used without further purification. All organic solvents were purchased from Merck and dried before use, IR spectra in the range 400-4000 cm^{-1} were obtained on a Perkin Elmer spectrum version 10.4.2 FTIR spectrophotometer using KBrpelletting technique.

Methods

1. Synthesis of zinc chelate of Cinnamic acid

Synthesis

All the reagents used were of BDH reagents and were dried and purified before use Zinc sulphate crystals and Diphenylamine and cinnamic acid crystals were mixed in 1:1:1 ratio. The mixture is heated for one hour in water bath. Then it is cooled and the beaker is kept without any disturbance. The mixture undergoes slow evaporation to form the complex in the form of crystals.

Synthesis of zinc chelate of Glycine

Synthesis

Zinc sulphate crystals and diphenylamine and glycine crystals were mixed in 1:1:1 molar ratio. The mixture is heated for one hour in water bath. Then it is cooled and the beaker is kept without any disturbance. The mixture undergoes slow evaporation to form the complex in the form of crystals.

RESULT AND DISCUSSION

The crystal were harvested and subjected to various characterization studies viz, UV and FTIR spectral studies shows the functional groups present in mixed crystal.

Spectral characterization

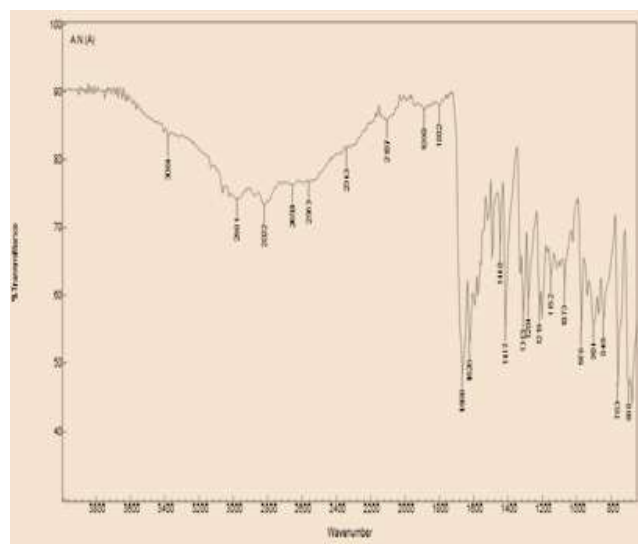
Characterization is a tool for the measurement of physical and chemical properties of materials. Characterization provides a basis for understanding and improving the characteristics of material for specific applications.

** IR Study

The IR Spectrashowed characteristic bands at 1629 cm^{-1} for compound 1, 1661 cm^{-1} for compound 2, 1414 cm^{-1} .

FTIR spectrum of Zinc chelate of cinnamic acid

In the IR spectra of free ligands NH stretching bands appear at 3434 cm^{-1} . On complexation this vibrations of ligand shifts in the zinc complex and observed at 3384 cm^{-1} . The appearance of band at 1629 cm^{-1} suggests the presence of C=C stretching vibrations of cinnamic acid. The bands at 1577 cm^{-1} and 1495 cm^{-1} indicate the presence of aromatic C=C group.



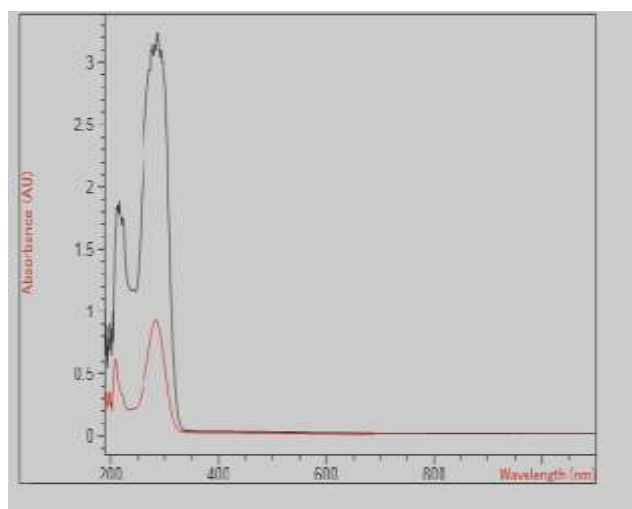
FTIR spectrum of mixed crystals of zinc chelate of glycine

The absorption frequency at 3534 cm^{-1} shows the presence of O-H group in the compound. In the IR spectra of free ligands N-H stretching bands appear at 3434 cm^{-1} . On complexation this vibration of ligand shifts in the zinc complex and observed at 3384 cm^{-1} .

The carbonyl band prepared in the spectrum of the ligand (1680cm^{-1}) disappeared in that of metal complex and was replaced by 2 bands at about 1580cm^{-1} & 1480cm^{-1} assigned to asymmetric & symmetric carboxylate stretching vibration with the difference in wave number suggested unidentate bonding. The peak at 1414cm^{-1} shows the presence of C=O stretching, the peak at 1154cm^{-1} shows the presence of C-O stretching.

UV spectrum analysis

The UV- Visible spectral analysis of was The UV-Visible spectral studies for mixed crystal were carried out in VARIAN CARY 500 UV-VIS-NIR double beam spectrometer.



UV spectrum of mixed crystals of zinc chelate of glycine

Zinc chelate of cinnamic acid

The observed λ_{max} at 226 nm is due to aromatic system and the electronic transition of $\pi-\pi^*$ occurs at λ_{max} 285 nm due to the presence of longer chromophore of cinnamic acid and the K band shifts and B band completely enveloped.

Zinc chelate of glycine

λ_{max} 283 nm is due to $n-\pi^*$ transition λ_{max} at 220 nm is due to the presence of aromatic

Optical window width

Optical window width is an important characterization of NLO material. Hence it is necessary that the transmission of electromagnetic wave the UV and NIR range is measured. The UV visible spectroscopy was recorded with a highly transparent single crystal in the range of 190-1100 nm and the spectrum observed that the crystal has good transmission in the entire visible and IR region and the lower cut off of the crystal is at 260 nm.

CONCLUSION

The study revealed the synthesis and characterization of zinc (II) complexes and confirmed an octahedral geometry around the central metal ion with water

molecules occupying the coordination sphere Optical window width is also used to characterize the material.

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