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GEL GROWTH AND CHARACTERIZATION OF NICKELNICOTINATE CRYSTAL

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Abstract

Coordination compounds of nicotinic acid with metals have significant relevance in biological applications. The crystal structures of the metals like manganese,cobalt, cadmium, copper etc with nicotinic acid have been reported. In this work, single crystals of nickelnicotinate were successfully grown by gel method. The powder X ray diffraction technique was carried out to reveal the high crystalline nature of the grown crystal. CHN analysis was done to determine the elemental composition of the synthesized crystal. The grown crystal was subjected to FTIR spectral analysis to confirm the presence of functional groups. The thermal decomposition behavior of the complex was studied from the TGA/DTA data in nitrogen atmosphere from ambient temperature to 800^{0}C.

Keywords : Nicotinic Acid(NA), Nickel nicotinate, gel growth, Powder Xray diffraction,Elemental Analysis,FTIR,Thermal Analysis.

I. INTRODUCTION

New materials are prime requisites of solid state research and device technology. Crystal growth is a prominent area in the scientific and technological researchto meet the demand of materials for different applications. Since the crystal growth has immense applications, it is an interdisciplinary subject covering physics, chemistry, material science, chemical engineering, metallurgy, crystallography, mineralogy etc.

Niacin also known as vitamin B_3 or nicotinic acid is an organic compound with the formula $C_6H_5NO_2$. This colorless, water-soluble solid is * Corresponding author: K. Rajendra Babu, PG Department and

a derivative of pyridine, with a carboxyl group

at the 3-position. Other forms of vitamin B_3 include nicotinamide where the carboxyl

group has been replaced by a carboxamide group ($CONH_2$), as well as more complex amides and a variety of esters. NADP(nicotinamide adenine dinucleotide phosphate)and NAD(nicotinamide adenine dinucleotide) are coenzymes for many dehydrogenases, participating in many hydrogen transfer processes[1]. Niacin is involved in both DNA repair and the production of steroid hormones in the adrenal gland. Insufficient niacin in the diet can cause nausea, skin and mouth lesions, anaemia, headaches, and tiredness. Chronic niacin deficiency leads to a disease called pellagra.

The ligand used here is NA which can produce extended structures. It has two potential donor sites: the pyridine ring nitrogen and the carboxylic oxygen. Multidentate ligands are usually used to bridge between metal centers to form polymeric structures through covalent or hydrogen bonds[2].

Co-ordination compounds of NA with metals like manganese,cobalt, cadmium, etc. are likely to exhibit significant biological effects and their crystal structures have been reported [3,4]. Changes in crystal structure can alter the way in which the drug is absorbed and accessed by the body [5]. The search for new crystal structures of the ligands having biological applications made us to perform this work.Here an attempt is made to grow nickelnicotinate crystal by conventional gel method.

II. EXPERIMENTAL PROCEDURE

The apparatus used for crystallization of single crystals by gel technique consists of borosilicate test tubes of length 20cm and diameter 2.5cm. Silica gel of specific gravity 1.03 to 1.06g/cc was prepared by dissolving sodium metasilicate (SMS) in double distilled water. The pH of sodium metasilicate solution was adjusted by adding acetic acid. About 10ml of this solution was taken in the test tube and 5ml of nicotinic acid was added slowly with continuous stirring to avoid any local ion concentration, which would otherwise cause premature local gelling and make the final solution inhomogeneous. The pH of the final solution was adjusted in the range 4.5-6.5. Test tubes were sealed with sheet of plastic to avoid contamination of the solution. Over the set gel, an aqueous solution of 10 ml nickel sulphate(0.25M) solution was poured carefully along the walls of the test tube to avoid gel breakage. Good quality single crystals of nickel nicotinate (NiN) were obtained by controlled nucleation and convectionless growth offered by gel technique. The growth of crystal continued for three weeks. The most favorable conditions for the growth of NiN crystals are gel density 1.04 g/cc and pH 6.The grown crystals are shown in fig 1.

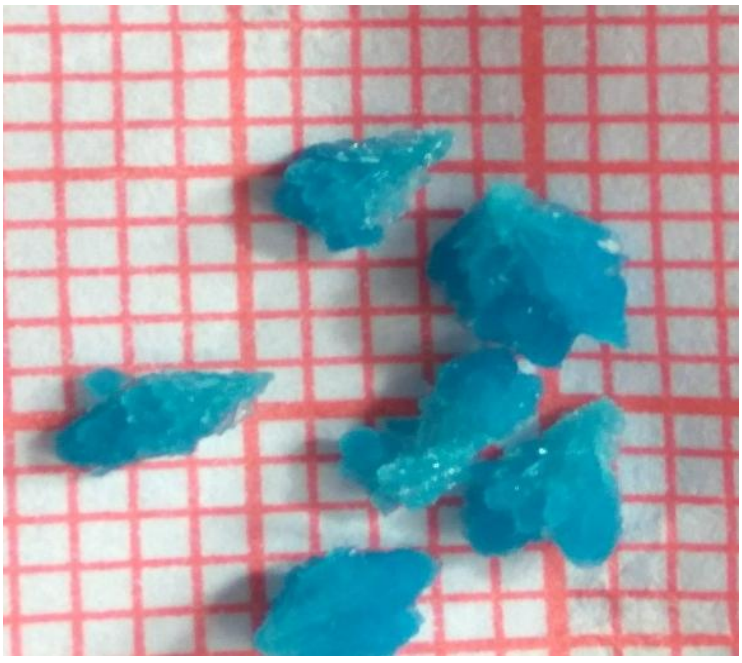

Fig.1. Photograph of Nickel nicotinate crystals

III. RESULTS AND DISCUSSION

3.1 Powder X-RAY Diffraction Analysis

The crystal in the powdered form is subjected to X ray diffraction in the 2θ range of 3°–80° and the PXRD pattern is shown in Fig 2.

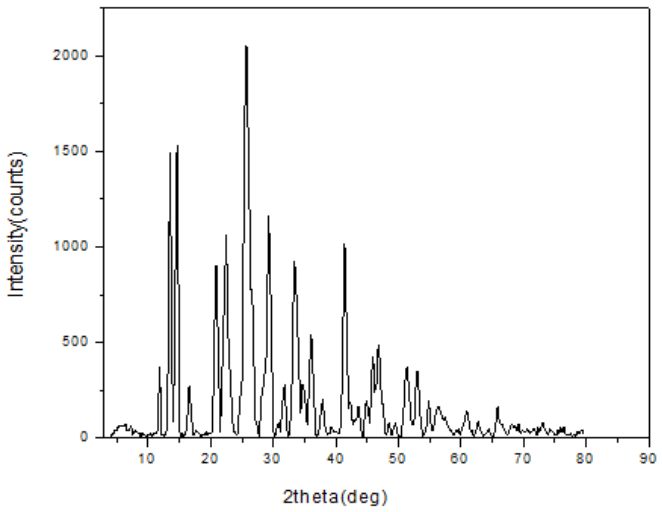

Fig 2. PXRD pattern of the Nickel nicotinate crystals

The sharp and strong peaks at specific 2θ values show that the grown crystal is exhibiting high crystalline nature.

3.2. Elemental Analysis

The percentage of carbon, hydrogen and nitrogen present in the grown crystal can be determined from the elemental analysis. Here the experimental values are compared with the theoretical values of carbon, nitrogen and hydrogen present and is given in table 1. Both the values are found to be in good agreement with each other and the molecular formula is derived as $C_{12}H_8NiN_2O_4$. $4H_2O$.

Table 1. Percentage composition of C, H &N as obtained by CHN analysis

Element	Composition	
	Experimental	Calculated
Carbon	37.52%	38.40%
Nitrogen	7.21%	7.46%
Hydrogen	3.93%	4.30%

3.3 Fourier Transform Infrared Spectroscopy (FTIR)

The FTIR spectrum of the grown crystal is shown in Fig 3. The coordination of NA with metal significantly alters the chemical & physical properties of the compounds by changing electronic charge distribution in the molecule. This might be observed as the changes in molecular vibration [6]. The peak at3232.33 cm^{-1}corresponds to ν(OH) of the coordinated water molecule present in the crystal. The bands observed at 1708 & 1416 cm^{-1} assigned to υ_{asy} and υ_{sym} vibrations of C=O in the ligand are shifted to lower regions 1617 & 1390 cm^{-1}, respectively in the

complex confirming the coordination of the metal through carboxyl oxygen[7]. The difference between the asymmetric and symmetric stretching (Δυ) of COO – group corresponds to different co-ordination modes[3]. The value (Δυ) of present complex is equal to 227 cm^{-1} revealing the bridging mode of COO- group[8].The lower value of υ_{asy}(C=O) absorption frequency of carboxylic acid is due to hydrogen bonding, which lengthens C=O bond[9, 10].The band corresponding to pyridine ring vibrations (C-C+C-N) observed at 1588 cm^{-1} in the ligand spectrum is shifted to lower wavenumber 1563cm^{-1}. This negative shift with respect to the ligand indicates the co-ordination of pyridine nitrogen to the metal atom.The peaks observed at 529$cm^{-1}$and 411cm^{-1}may be due to the stretching vibrations of Ni-O and Ni-N respectively.

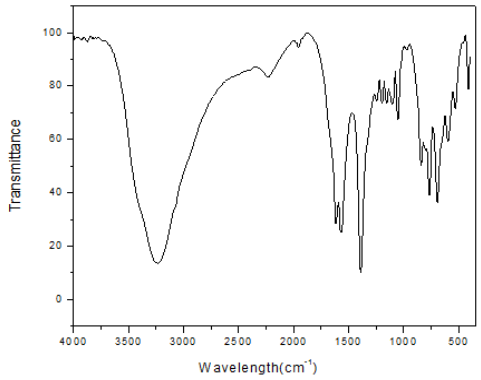

Fig 3. FTIR Spectrum of the Nickel nicotinate crystals

3.4 Thermal Analysis

The thermal properties of the complex has been studied from the TGA/DTA curve shown in fig.4.The graph shows two endothermic peaks corresponding to two decomposition stages.The crystal is stable up to 179.23°C. The first decomposition is due to the loss of 4 coordinated water molecules with a mass loss of 19%(Cal.19.2%). The endothermic peak at 427.16^{0}C corresponds to the degradation of ligand with a mass loss of 49% (Cal 49.1%) leading to the formation of $NiCO_3$.

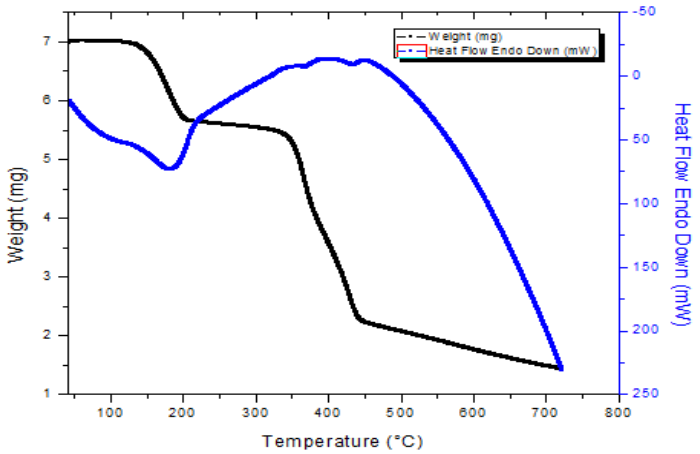

Fig 4. TGA/DTA curves of the Nickel nicotinate crystals

VI. CONCLUSION

Nickel nicotinate crystals have been successfully grown by gel diffusion method. The optimum conditions for the growth of these crystals are gel density 1.04 and pH 6. Crystalline nature of grown crystal was confirmed by powder XRD studies. The chemical formula was derived as $C_{12}H_8NiN_2O_4$. $4H_2O$ from the elemental analysis.The involvement of two potential donor sites - pyridine nitrogen and carboxylic oxygen in the coordination is established by FTIR. TGA/DTA provides the thermal decomposition behavior of the complex.The crystal is stable up to179.23°C.

VII. ACKNOWLEDGEMENT

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