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Raman Study of Fe doped Potassium Oxalate Monohydrate crystals grown by Slow evaporation technique

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Abstract: *Raman spectroscopy study of potassium oxalate monohydrate $K_2C_2O_4.H_2O$ (POM) its doped varieties with 5%,10%,15%,20% Ferrous concentration investigated . Fe-doped POM crystals were grown by slow evaporation technique at laboratory temperature. Powder x-ray diffraction (PXRD) and Raman spectroscopy has been used to characterize the crystal material. The results inform that the doping concentration had a significant impact on the structural and spectral properties of the material. From XRD data analysis of the crystal suggest that this material forming $K_3Fe(C_2O_4)_3.3H_2O$ compound and is belong to $P2_{1/c}$ space group with the monoclinic structure.*

Keywords: *Potassium Oxalate Monohydrate, Fe-Doped, Raman Spectroscopy.*

1. INTRODUCTION:

Study of the doped crystalline material is more interested in Solid State field for a long time. How the external impurities affect in the lattice structure with neighbouring molecule. We select the potassium oxalate monohydrate (POM) to study the external impurities affected in bonding pattern of POM crystal [1]. In crystal structure determination , the study of x-ray and neutron diffraction is important[2-4]. Eriksson and Nielson presented the Raman spectroscopy study for powder and crystalline potassium oxalate monohydrate[5]. Infrared (IR) studies of pore POM was reported by Fukushima [6]. Vanadyl ions in doped POM , the lattice forming $VO(C_2O_4).H_2O$ by replacing the two number of potassium ion in the oxalate group with H_2O-C_2O_4-H_2O chain[7]. Fe doped POM crystals have many application in actinometry, photochemical studies, magnetic material and sensors, etc[8,9]. Bonding arrangement, lattice structure, vibrational and rotational motion is affected by differ doping group in POM. The vanadyl doping in POM is changes the relative intensities in some Raman bands. It form the $VO(C_2O_4).H_2O$ complex by change in the C_2O_4-H_2O-C_2O_4-H_2O chains[10]. In the microscopic level Raman spectroscopy studies suggest the direct proof of structural changes with the lattice modes. In this paper we are representing the results of Raman study for 5%, 10%, 15%, 20% varieties for ferrous doped POM crystal.

2. MATERIALS AND METHOD :

Crystals of POM doped with ferrous sulphate were grown by saturated solution using slow evaporation technique at laboratory temperature. The preparation of saturated solution by dissolving a.r. grade POM along with 5%, 10%,15% and 20% ferrous sulphate by weight in distilled water. This solution was stirred for 3 hours and boiled for 10 min. After this it filtered at cold condition to remove any unreacted residue . Further this green colour solution was kept for slow evaporation at lab temperature for crystallization. Nearly about 15 days green crystals were obtained[11].

3. RESULT AND DISCUSSION:

(a) (b) (c) (d) (e)

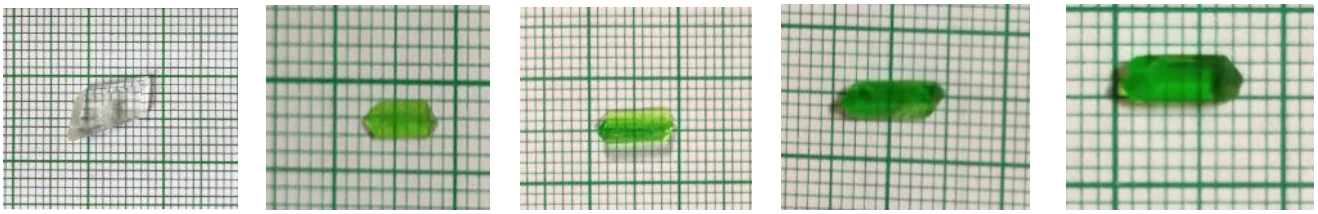

Fig.1 Photographic image of (a) POM (b) POM:Fe(5%) (c) POM:Fe(10%) (d) POM:Fe(15%)(e) POM:Fe(20%) crystals

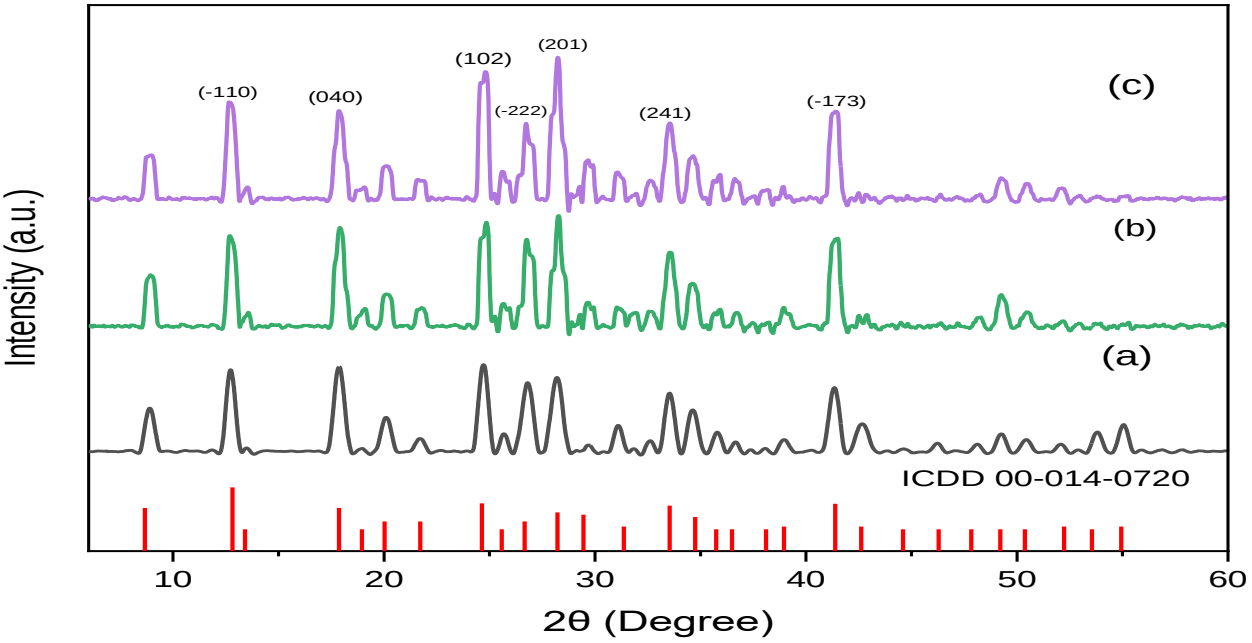

Fig.2 Powder XRD Pattern of Fe-doped POM crystal (a) POM:Fe(20%) (b) POM:Fe(15%) (c) POM:Fe(10%)

Pure and Fe-doped crystals photographic images shows in the fig.1. In the fig.2 (a,b,c,) are the xrd-pattern for 20%, 15% and 10% Fe-doped POM crystal. This xrd-pattern are match with the ICDD Card No. 00-014-0720. X-ray analysis of the crystal revels that this compound forming $K_3Fe(C_2O_4)_3.3H_2O$ and is belong to the monoclinic structure with $P2_{1/c}$ space group and make a three-dimensional chain form [12,13].

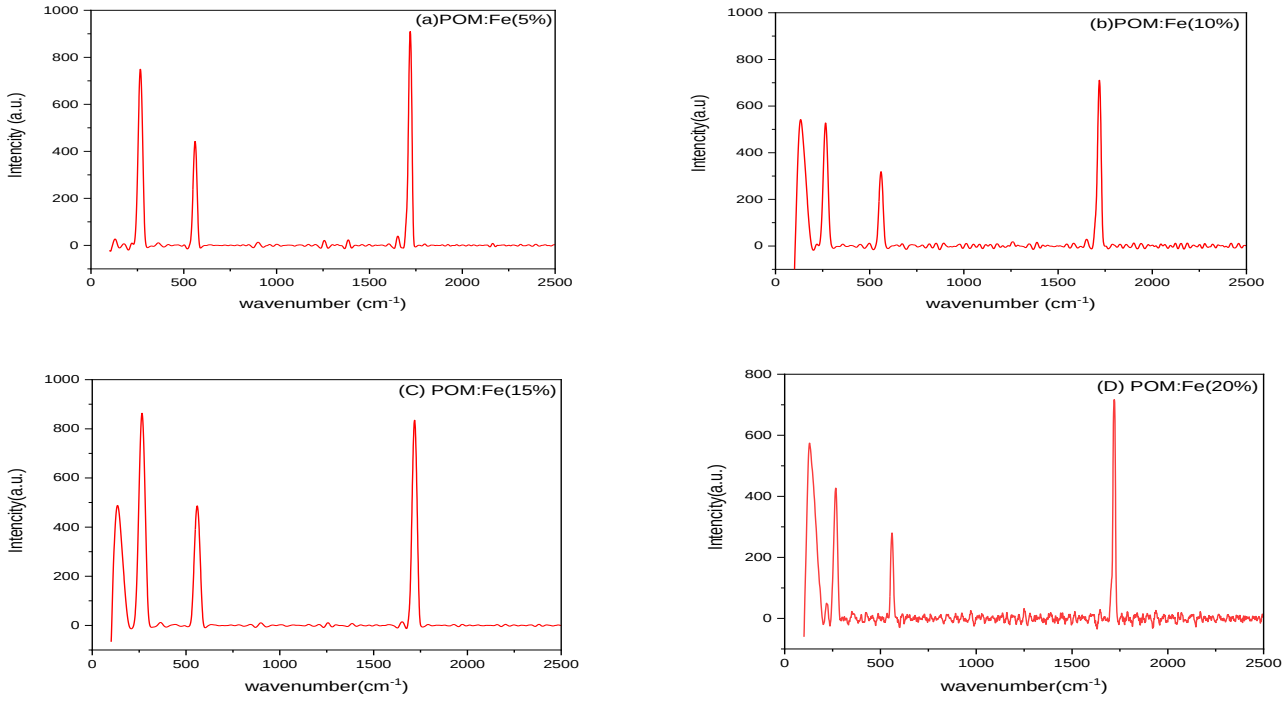

Fig.3 Raman Spectrum of Fe-doped POM crystal (a) POM:Fe(5%) (b) POM:Fe(10%) (c) POM:Fe(15%) (d) POM:Fe(20%)

The Raman spectrum of Fe-doped POM crystal is shown in fig.3. From the earlier Raman spectrum studies of similar metal oxalate compound the band assignments are made[11,14-16].The band observed at 133.77, 132.63, 128.07 and 266.88, 267.02 (cm^{-1}) are assigned to υ(Fe-O)/ρ(H2O). The observed band at 562.06, 560.04, 562.19 and 585.96 (cm^{-1}) are assigned as δ(O-C-O) oxalate ion mode. The υ(C-O) mode are exhibited at 1720, 1719.57, 1716.84 and 1721.39 (cm^{-1}). This assignment are show in table.1 .

Table:1 Raman data for Fe-doped POM crystal (a) POM:Fe(5%) (b) POM:Fe(10%) (c) POM:Fe(15%) (d) POM:Fe(20%)

Sample	POM:Fe(5%)	POM:Fe(10%)	POM:Fe(15%)	POM:Fe(20%)	Assignment
Raman Shift (cm^{-1})		133.77	132.63	128.07	υ(Fe-O) /ρ(H2O)
	266.884	267.02	267.02	267.02	
	562.06	560.04	562.19	585.96	δ(O-C-O)
	1720	1719.57	1716.84	1721.39	υ(C-O)

4. CONCLUSION:

Fe-Doped (5,10,15 & 20%) POM crystals were synthesized and grown successfully using slow evaporation technique at laboratory temperature. The XRD data suggest that the samples are in good crystalline form. And compound forming the $K_3Fe(C_2O_4)_3.3H_2O$ structure with monoclinic group. The Raman data interpretations are align with reported assignments.

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