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Crystal Growth And Characterization Of Cadmium Manganese Oxalate Crystals Grown By Agar -Agar Gel Method

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Abstract: Single crystal of cadmium manganese oxalate crystals was grown by using agar – agar gel method at room temperature. Effect of various parameters on the growth of cadmium manganese crystals has been studied and reported. X- ray Diffraction analysis was done to determine the structure of grown crystal. Orthorhombic crystal structure was found of grown crystal. The chemical analysis has been performed by FTIR to denote the functional group of grown crystal. Scanning Electron Microscope images highlights that crystals were grown by layer deposition. Elemental analysis carried out by Energy Dispersive X-ray Analysis (EDAX). Thermal behaviour of grown crystal studied by using Differential Scanning Calorimeter (DSC).

Keywords **-** Single diffusion, XRD, FTIR, SEM, DSC, EDAX.

1. INTRODUCTION

Crystal growth is currently a rapidly growing field of research because of the huge demand for crystal in many applications. In many researchers a series of pure and mixed crystal have been studied to find new materials for various purpose. Crystal has long been admired by mankind for their symmetrical structural simplicity and purity. Crystal growth has been widely used in the fields of ceramics, physics, chemistry, metallurgy, mineralogy, medicine, engineering,[1] amplifiers, semiconductor, optics, piezoelectric, photosensitive material, infrared, nonlinear optics, microelectronic and microprocessor are great demand for crystal growth [2]. There are various techniques for growing crystal-like melt growth; vapour phase solution growth [3]. The gel method is one of the best for grown crystals. It is a very simple and easy method that allowed perfect and stress-free crystal growth due to the principle involved in the method is very simple. There are a variety of crystals that can be grown simply and inexpensively method. Insoluble or sparingly soluble crystals were synthesizing by the gel method. In the gel techniques, a solution of two suitable compounds, which gives rise to required insoluble crystalline material by simple reaction, allow to chemically react in the set gel medium. The gel method of crystal growth is simple and most versatile technique compared to other methods of crystal growth under the room temperature condition [4]. Crystal growth in gels is an excellent simple technique for growing single crystals for metal oxalate and for transition metal oxalates due to their solubility in water [5-7]. Many people grown crystal by using silica gel [8-14] and very few researches grown crystal by using agar-agar gel method [15-18]. The Study of cadmium manganese oxalate crystal growth in gel method had very rarely observed [19]. Cadmium manganese oxalate is a very interesting compound because it has good applications. Therefore, during the present investigation it has been decided to synthesize and characterized cadmium manganese oxalate crystal by using agar-agar gel method. Characteristics of cadmium manganese oxalate crystals grown by various techniques are reported.

2. MATERIALS AND METHODS

In the present work, the single diffusion agar-agar gel method was used to grow the cadmium manganese oxalate crystals at ambient temperature. Cadmium chloride ($CdCl_2$), manganese chloride ($MnCl_2$), oxalic acid ($H_2C_2O_4$) and agar- agar powder were used as chemical compound. All the chemical compounds were AR grade. The all the experimental solutions were prepared using distilled water. A single glass tube having length 15cm and

diameter 2.5cm and 250mL glass beaker were used as crystallizing vessel. In single diffusion, the test tube was filled by first reactance as mixture with same ratio of cadmium chloride and manganese chloride of desired volume and morality. Gel prepared by mixing (0.5g – 2.5g) agar powder in 100mL double distilled water at boiling temperature, hot agar-agar gel was poured in the test tube and kept for setting. After setting and aging the gel the second reactant which is oxalic acid solution of desired volume and morality was added over the set gel. The open end of tube was closing with cotton plugs and kept undisturbed at normal temperature. After 6-7 days some nucleation had seen on the interface of test tube, then various shape of grown crystals observed in the test tube was separated from the gel. As the grown crystal were collected and observed. The reaction which command to the growth of cadmium manganese oxalate crystal were expressed as,

$$CdMnCl_2 + H_2C_2O_4 \longrightarrow CdMnC_2O_4 + 2HCl$$

3. RESULTS AND DISCUSSION

Single diffuse technique using sodium meta silicate is widely used for crystal growth. There are few reporters to use agar-agar gel for crystal growth; here we report the growth of cadmium manganese oxalate crystal in agar-agar gel by using single diffusion method. The optimum condition for growing good shine cadmium manganese oxalate crystals is shown in table 1. Different parameter like concentration of first and second reactants, gel percentage, aging time significantly affected the growth rate. Different percentage of gel poured into test tube while other concentration kept constant, it was found that gel percentage increased, size and shape of grown crystals were decreased. The amount of concentration of reactants varies 0.5M to 2M, it was observed that quality of grown crystals decreased with increased morality of reactant. Aging period has significantly affected the growth rate; gel is poured into test tube; gel takes more time to set. The rate growth was fast at 40-42 hours. The use of reversing reactants did not see to cause any changes in the quality and size of crystals having rhombus, cubic, quadrilateral, kite like, diamond shape was obtained. In the present work figure 1 shows growth of cadmium manganese oxalate in the test tube and figure 2 shows different morphology of grown cadmium manganese oxalate crystals.

Condition	Single diffusion
Concentration of first reactants	1M
Concentration of second reactants	1M
Percentage of gel	1%
Volume of cadmium chloride	5ml
Volume of oxalic acid	5ml
Gel aging period	2-3 days
Temperature	Room temperature
Quality	white, rhombus, kite like, diamond shape

Table 1. Optimum condition for growth of cadmium manganese oxalate

Fig1. Growth of cadmium manganese oxalate crystal in test tube

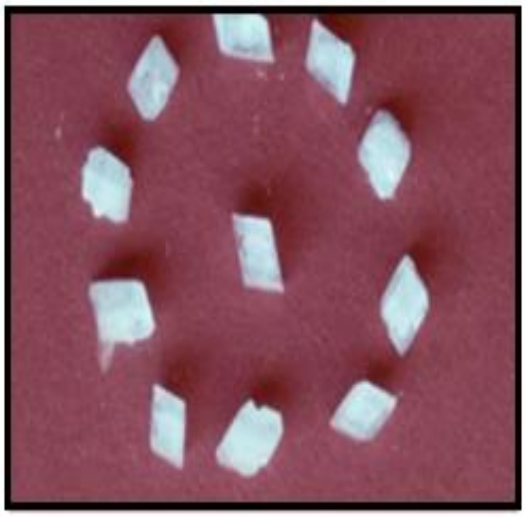

Fig 2. rhombus, cubic, diamond shapes of grown cadmium manganese oxalate crystals

4.CHARACTERIZATION

4.1. X-RAY DIFFRACTOMETER (XRD)

The X-ray diffractometer carried out at CRYSTA PEAK SOLUTION LAB, Pune. In the present work, The XRD pattern of cadmium manganese oxalate crystals grown by single diffusion techniques as shown in figure 3.

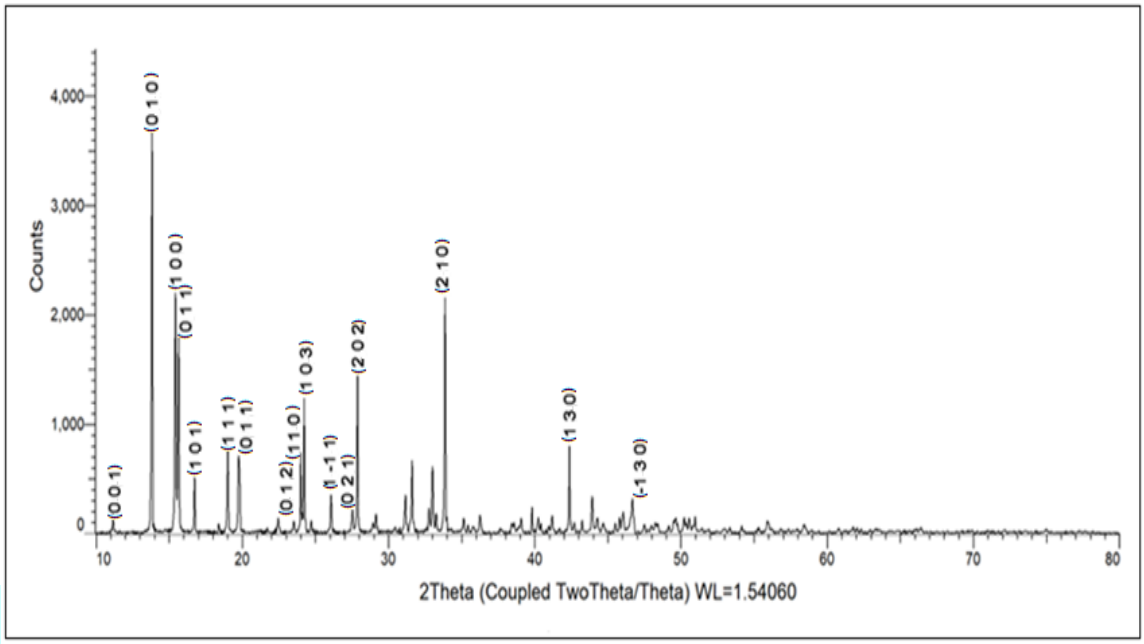

Fig 3. XRD pattern of the grown cadmium manganese oxalate crystals

The sample scanned in 2θ value from 11^0 to 50^0. The characteristic peak appears at 13.83 (2θ) for cadmium manganese oxalate crystal. The XRD pattern reveals that sample is crystalline in nature having orthorhombic system. The 2θ Peak observed at 11.17^0, 13.83^0,15.43^0, 15.64, 16.73^0, 18.39^0, 19.76^0, 22.47^0, 24.00^0, 24.72^0, 26.06^0, 27.53^0, 27.88^0, 33.87^0, 42.37^0, Which corresponds to (0 0 1), (0 1 0), (1 0 0), (0 1 1), (1 0 1), (1 1 1), (0 1 1), (0 1 2), (1 1 0), (1 0 3), (1 -1 1), (0 2 1), (2 0 2), (2 1 0), (1 3 0) plane of reflection respectively. These results were found to be good agreement with JCPDS data (card no 32-0647). The 2θ, d value, relative intensity and Millar indices (h k l) of major peak observed in spectra of cadmium manganese oxalate crystal are tabulated in table 2. By using Scherer's formula, the grain size of grown crystal calculated as D = 83.60 nm. Lattice parameter from XRD data as a = 6.265 A^0, b = 6.086 A^0, c = 13.59 A^0 i.e. $a \neq b \neq c$ and volume V = 518.17 A^3. In orthorhombic crystal structure the length of unit cell is different and $\alpha = \beta = \gamma = 90^0$.

2θ	d A^0	Relative Intensity (cts)	Indices
11.17	7.9143	142.988	(0 0 1)
13.83	6.3979	3574.53	(0 1 0)
15.43	5.7374	2120.30	(1 0 0)
15.64	5.6585	1725.53	(0 1 1)
16.73	5.2925	475.240	(1 0 1)
18.39	4.8199	88.5700	(1 1 1)
19.76	4.4892	701.958	(0 1 1)
22.47	3.9534	128.170	(0 1 2)
24.00	3.7039	696.420	(1 1 0)
24.72	3.5977	108.015	(1 0 3)
26.06	3.4153	352.301	(1 -1 1)
27.53	3.2368	210.410	(0 2 1)
27.88	3.1967	1434.15	(2 0 2)
33.87	2.6444	2037.55	(2 1 0)
42.37	2.1311	786.130	(1 3 0)

Table 2. XRD data of grown cadmium manganese oxalate crystal

4.2 FOURIER TRANSFORMS INFRARED SPECTROSCOPY (FTIR)

FTIR spectroscopy is used for identified functional group of cadmium manganese oxalate crystals involved in vibration frequency. The FTIR spectrum of cadmium manganese oxalate crystals are shown in figure 4. The spectrum was obtaining using 400 cm^{-1} to 4000 cm^{-1} at R.C.Patel Pharmacy Research Institute Shirpur. The spectrum shows the peak in the range 500 cm^{-1} to 3500 cm^{-1}. The fundamental FTIR frequencies observed in grown crystal [20]. The FTIR spectrum record for cadmium manganese oxalate crystals with observed band. The spectrum shows the peak in the range 500 cm^{-1} to 3500 cm^{-1}. The bands in the range 3499.54cm^{-1} to 2313.18 cm^{-1} recognized to O-H strong broad starching of water molecule. The presence of C-C stretching group is indicated by occurrence the sharp and intense bond at 1601.92cm^{-1}.C-O bond stretching appear at the range 1368.92, 1313.92cm^{-1}. The peak between 898.63cm^{-1} to 724.00 cm^{-1} can be attributed C-H bending bond. The absorption situated below 668.01 cm^{-1} are due to metal oxygen bond stretching vibration. Table 3 shows the assignment of FTIR spectrum.

Sr. No	Wave Number cm^{-1}	Assignments
1.	3499.54, 3432.52, 3197.68, 2313.28	O-H stretching and water molecule
2.	1601.92	C-C
3.	1368.92, 1313.92	C-O
4.	789.90	C-H
5.	668.01	Metal oxygen bond

Table 3. FTIR data of grown cadmium manganese oxalate crystals

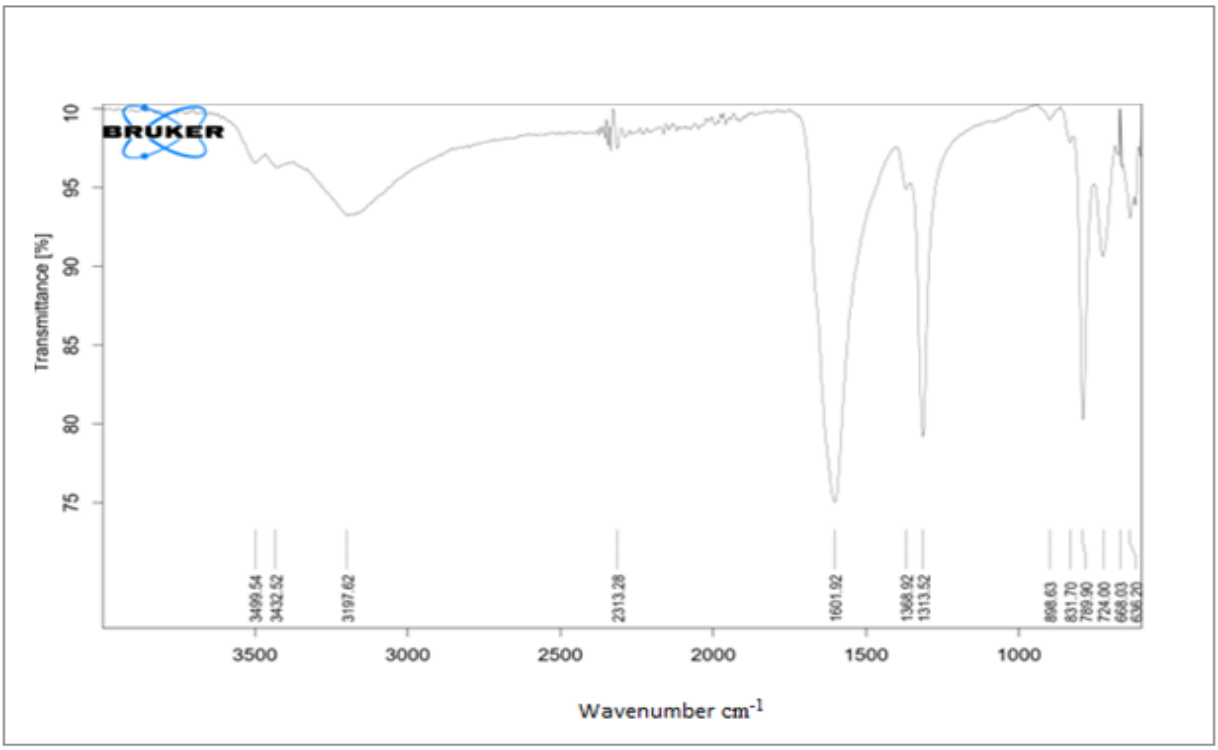

Fig 4. FTIR spectrum of the grown cadmium manganese oxalate crystals

4.3. DIFFERENTIAL SCANNING CALORIMETER (DSC)

Differential scanning calorimeter was studied at KBCNMU Jalgaon. The 9.00 mg weight of powder of cadmium manganese oxalate crystal was taken for DSC analysis. The DSC analysis of grown crystal was recorded between 30^0C to 300^0C. DSC curve for cadmium manganese oxalate crystal as shown in figure 5 from the DSC curve confirmed the existence of endothermic dip peak at 112^0C result in the formation cadmium manganese oxide. Heat of transition ΔH i.e. enthalpy change transition 543J/g observed in temperature range between 100^0C to 150^0C.

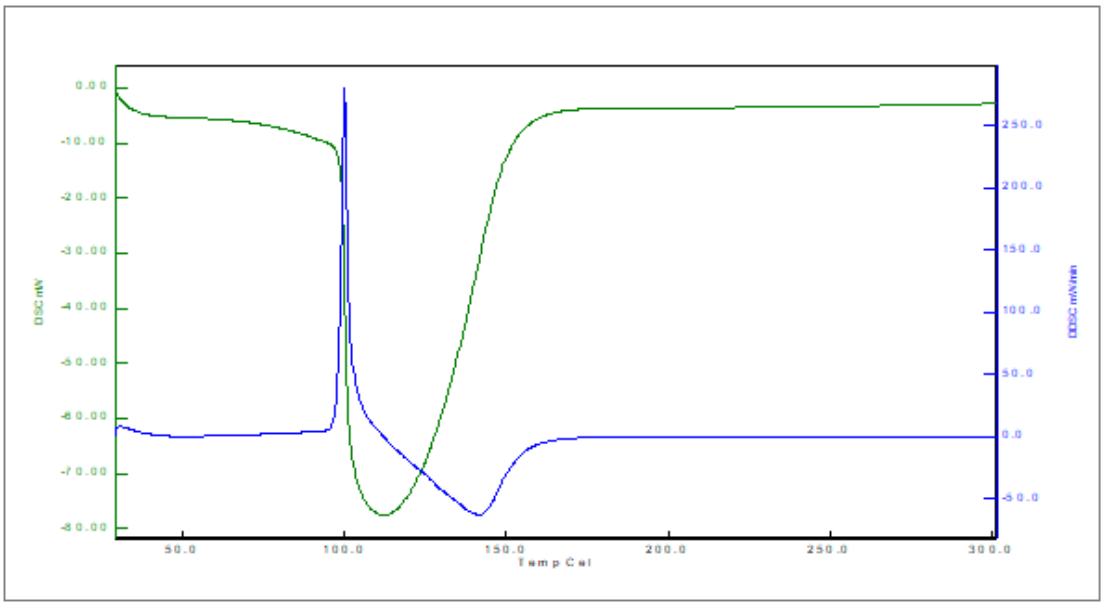

Fig 5. DSC spectrum of the grown cadmium manganese oxalate crystals

4.4 ENERGY DISPERSIVE ANALYSIS BY X-RAY (EDAX)

Energy Dispersive Analysis by X-Ray (EDAX) is used for the qualitative analysis and called as elemental analysis. Elemental analysis of cadmium manganese oxalate crystal carried out at CRYSTA PEAK SOLUTION LAB, Pune which standard less at 10 eV energy show the results. The peak ranging from 3.0 KeV to 6.0 KeV clearly indicates the presence of cadmium and manganese in the sample. The relative concentration of cadmium and manganese is observed as 42.90 %, 06.70% respectively thus EDAX observed that sample crystal show presence of cadmium and manganese. Figure 6 shows spectrum of cadmium manganese elemental analysis and observed elemental analysis data is tabulated in table 4

Sr. No.	Element	Shell	Wt. %	At. %
1	O	K	50.40	86.22
2	Mn	K	06.70	03.34
3	Cd	L	42.90	10.45

Table 4. EDAX data of cadmium manganese oxalate

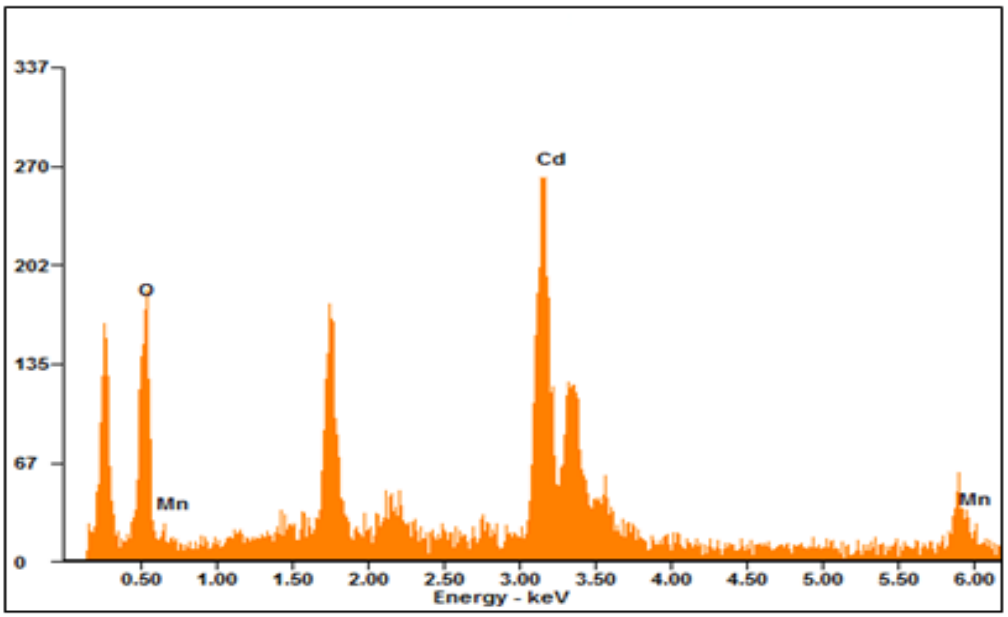

Fig 6. EDAX spectrum of the grown cadmium manganese oxalate crystals

4.5 SCANNING ELECTRON MICROSCOPE (SEM)

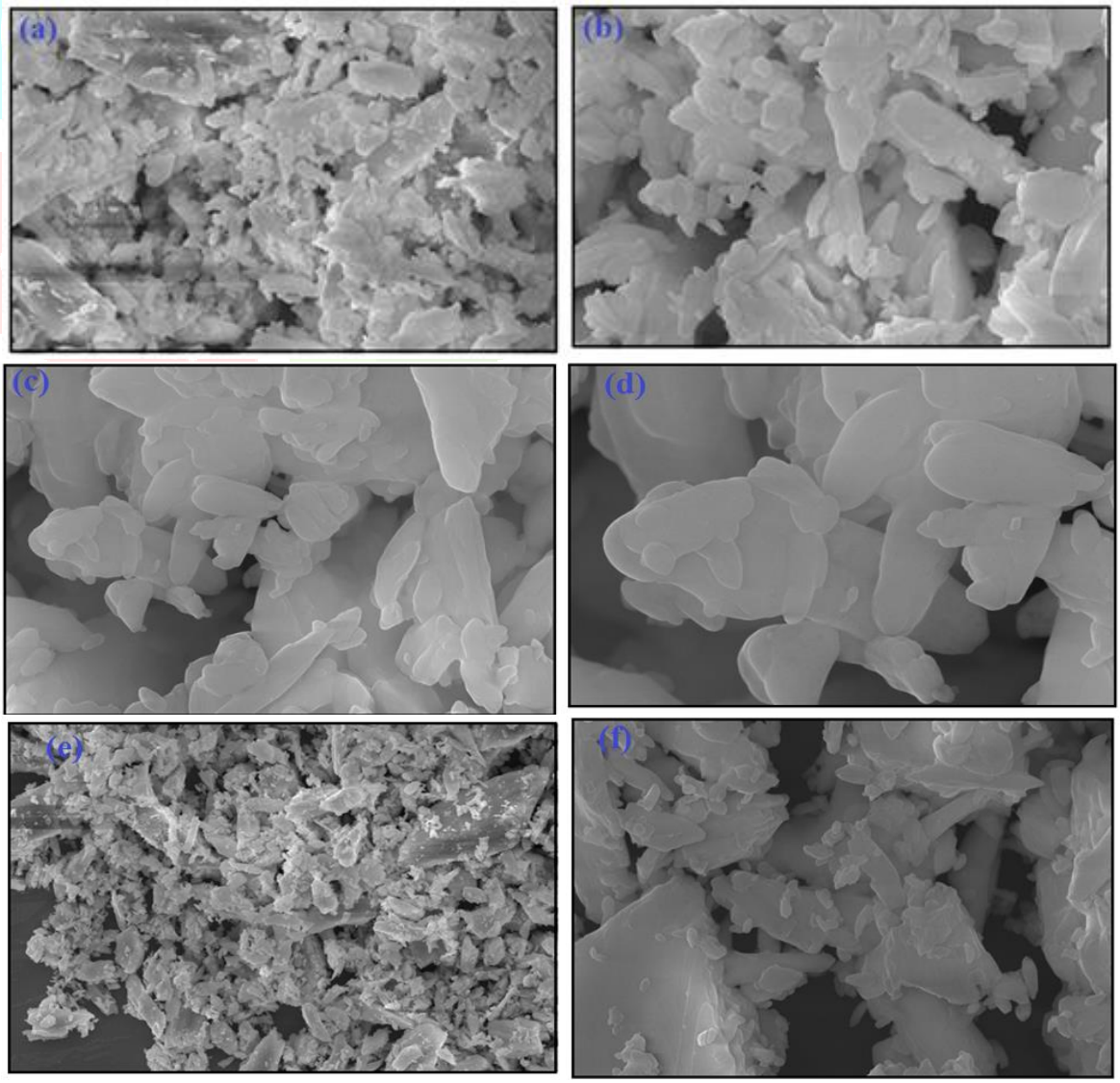

Fig 7. Different SEM image of grown cadmium manganese oxalate crystal

SEM image gives information about the nature and stability for device application. It is also applied to check the presence of imperfections. The morphology of cadmium manganese crystals was studied by scanning electron microscope. SEM analysis was carried out at CRYSTA PEAK SOLUTION LAB, Pune.The different morphology of SEM image of grown crystal as shown in the figure 7. The SEM photographs of cadmium manganese oxalate crystal shows coarse mixture of compound of various particle size. The compound is mainly crystalline in nature observed due to formation of various shapes having regular phase. The certain combination of these faces generates like as needle, pyramidal, oval, quadrilateral, flat, spherical, cuboidal, capsulated, chrysocolla, amber, columnar, ruby, triangular, selenite and cocci etc. shapes are observed in SEM photograph.

5. Conclusion

1. Single crystal of cadmium manganese oxalate crystals was successfully grown by using single diffusion technique in agar -agar gel.
2. White colour with average size of 4 x 3 x 1 mm^3 were obtained during a period of 8 weeks at room temperature by applying different parameter.
3. The XRD pattern shows that grown crystals are in crystalline in nature and having orthorhombic structure.
4. FTIR spectroscopy of cadmium manganese oxalate reveals the presence of oxalate ligands and crystallization water molecule
5. Thermal stability was studied by DSC analysis
6. SEM images suggest that grown crystal has different morphology like as needle, pyramidal, oval, rectangular, capsulated, flat, cuboidal, triangular, spherical, and pyramidal shape.
7. The presence Cd and Mn in the cadmium manganese oxalate crystals has been confirmed by EDAX.

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