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Impact of Zn^{2+} ions on Optical and Mechanical Investigations of Pure LTMA Single Crystal

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Abstract*:*The Pure L-Threonine manganese acetate (LTMA) and Zn^{2+} doped LTMA Single crystals have been grown at 30^{0}C by the slow evaporation method. The grown crystals were subjected to various characterizations as single crystal XRD, Powder XRD, FTIR, UV–Vis, EDAX and Microhardness analysis. The lattice parameters of the grown crystals determined as Orthorhombic by single crystal X-ray analysis and Zn^{2+} ions made the volume increases as 530 Å^3 to 538 Å^3. EDAX analysis confirms the presence of Zn^{2+} ion in the host material. The functional group and optical behavior of the crystals were identified from FTIR. UV-Vis absorption spectrum analysis determines the energy band gap 6.3 eV and transmittance spectrum determines the lower cutoff wavelength of the grown crystal 200nm. Mechanical properties of the crystals studied by Vicker's hardness and hardness load graph plotted.

***Keywords*: EDAX. Microhardness, Powder XRD, Single Crystal XRD**

I. INTRODUCTION

At present lot of the exploration work done on the synthesizing and portrayal of semi natural crystal. Because of their wide optical applications, contrasted with different materials, amino corrosive blended natural crystal was intrigued to orchestrated [1].The search materials has qualifies for the discovery of numerous natural NLO materials with high nonlinear natures and photonic applications [2–4] and [5-11]. Authentically, it can utilize in uses of predominant quality precious stones [12, 13]. Like the semi natural precious stones, amino corrosive based semi natural gems were likewise has acceptable optical and non-direct optical natures. This single crystal can likewise be developed from watery answer for the improved hardness and the warm steadiness. In this reference, unadulterated L-Threonine single gem and L-Threonine based Lithium Chloride (LTLC), Calcium Chloride (LTCC), Cadmium chloride (LTCC), Manganese chloride (LTMC) single precious crystal are developed and its characters likewise examined [14-18]. Afterward, L-Threonine Sulfate crystal like LithiumSulfate (LTLS), PotassiumSulfate (LTKS), ZincSulfate (LTZS) and Copper Sulfate (LTCS) single precious crystal are developed and considered [19-22]. At that point, L-Threonine Cadmium Acetate (LTCA) and L-Threonine Zinc Acetate (LTZA) single precious crystal are developed [23-24]. In proceeds, this work finished with development on unadulterated L-Threonine Manganese Acetate single crystal (LTMA) and the Zinc doped L-Threonine Manganese Acetate (Zn2+ doped LTMA) novel single crystals were effectively developed by moderate vanishing technique. The different portrayals have been done and those properties of both crystals were detailed.

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II. EXPERIMENTAL SECTION

A. Synthesis & Crystal growth

AR grade chemicals of L-Threonine and Manganese acetate tetra hydrate in the ratio of 1:1M taken in 10ml distilled water and 0.2M of (AR grade) Zinc acetate (ZnA) added in 1:0.8 M of L-Threonine and Manganese Acetate solution taken in 10ml distilled water separately. Both prepared saturated solutions was filtered and then housed in a dust free atmosphere. The pure and Zn^{2+}doped LTMA single crystals were successfully grown at room temperature with repeated recrystallization by slow evaporation method. After a period of 20-25 days, harvested single crystals of 10×3×2 mm3 size pure LTMA single crystal and 10×4×2 mm^3 size of Zn^{2+} doped LTMA were grown as shown in Fig.1.

Chemical formula:

i) Pure LTMA single crystal

$C_4H_9NO_3+(CH_3COO)_2Mn.4H_2O$

$$\rightarrow Mn[C_4H_9NO_3]^+ . 2(CH_3COO)^- + 4H_2O \uparrow \quad (1)$$

ii) 0.2M Zn^{2+} doped LTMA single crystal

$C_4H_9NO_3+0.8M(CH_3COO)_2Mn.4H_2O+0.2M(CH_3COO)_2\ Zn$

$$\rightarrow (Mn_{0.8}[C_4H_9NO_3]Zn_{0.2})^+ .2(CH_3COO)^- + 4H_2O \uparrow \quad (2)$$

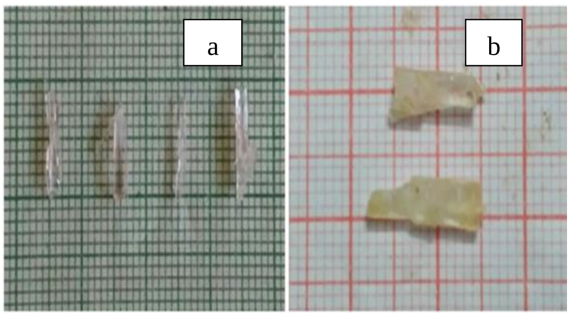

Fig.1.As grown (a) pure LTMA and (b) Zn^{2+}doped LTMA Single Crystal

III. RESULT AND DISCUSSION

A. Single Crystal X – Ray Diffraction Analysis

In order to determine the Crystal system, the grown single crystals of pure LTMA and Zn^{2+} doped LTMA subjected into Single Crystal XRD analysis. SCXRD analysis carried out using Nonius CAD4/MACH3 Single Crystal X-ray diffractometer with MoKα (α= 0.71073 Å) source lies that pure L-Threonine Manganese Acetate (LTMA) and Zn^{2+} doped LTMA become Orthorhombic system with different unit cell parameters as shown in Table I. Previous reports of similar base material L-Threonine (L-T)[14] and Cadmium acetate doped L-Threonine single crystal (LTCA)[24] are compared in this Table I. This result proved that pure LTMA and Zn^{2+} doped LTMA also satisfy the previous same reports in this regard.

TABLE-I: Lattice Parameters of Pure LTMA andZn^{2+}doped LTMA

Parameter	LTMA [Present work]	Cd^{2+}doped LTMA [Present work]
a	5.106(19) Å	5.17 Å
b	7.721(19) Å	7.71 Å
b	13.45(4) Å	13.59 Å
α=β=γ	90°	90°
Volume	530(4) $Å^3$	540 $Å^3$
Space group	P_{212121}	P_{212121}
Crystal System	Orthorhombic	Orthorhombic

B.Powder X – ray diffraction analysis

To study the Powder X-ray diffraction analysis recorded is to determine the lattice point and repeated regular atoms illustrated. This pattern results that different peaks reports the strength of atoms of compositions regarding the reaching the height of peaks are shown in Fig.2. Miller indices estimated by powder V1.0 software along with 2 Theta values of LTMA crystal is given in Table II. The different peaks confirm the Powder XRD pattern L-Threonine Manganese Acetate single crystal. All the peaks differ from the peaks of pure L-Threonine single crystal [14]. The first peak start at 3.93 degree and its plane calculated [111] and second maximum reaches as 345 a.u intensity in angle 22.2 degree, its plane calculated as [411]. The continual peaks reveal at the angles 25.6, 26.6, 36, 38.4 and 44.9 in the respective peaks of plane 422, 431, 444, 552 and 661. Fig.2 proved that the pure LTMA consist, number of different planes with different lattice points. It concluded that no one single element result as different peaks, therefore this material contain greater single element.In this Zn^{2+} doped LTMA single crystal, the difference in peak powder XRD Pattern are determined and its all the different the hkl plane values are calculated and tabulated in the Table III.

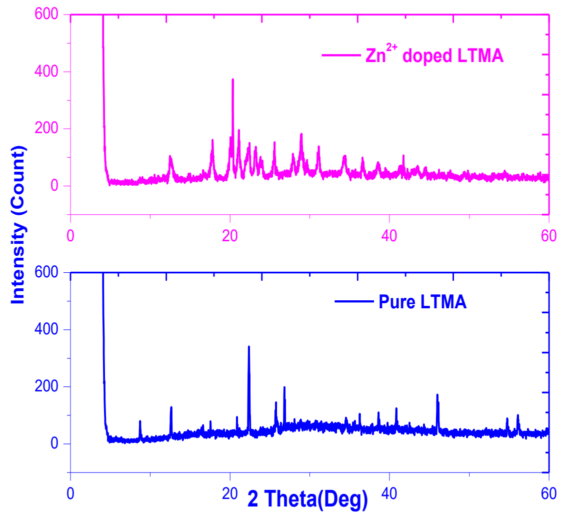

Fig. 2: Powder XRD patterns of pure LTMA and Zn^{2+} doped LTMA

TABLE-II: 2Θ VsIntensityValuesof LTMA Crystal

2θ	Intensity	hkl
3.93	1178	111
12.5	134	211
17.6	100	222
22.2	345	411
25.6	154	422
26.6	203	431
36.0	108	444
38.4	113	552
44.9	176	661

TABLE-III: 2Θ VsIntensityValuesof Zn^{2+}doped LTMA

2θ	Intensity	hkl
3.93	1039	111
9.03	52	222
12.94	235	334
17.12	70	76-5
18.12	238	764
20.5	261	761
21.4	241	763
22.4	194	772
23.7	239	774

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C. Energy Dispersive analysis (EDAX)

Energy dispersive X-ray analysis (EDAX) illustrates the EDAX spectrum of LTMA crystal using JEOL company (JSM-6701 F, SEM). The presence of Carbon, Nitrogen, Oxygen and Manganese in LTMA single crystal was obtained as shown in Fig. 3. Due to the inclusion of acetic acid, Carbon & Oxygen has the maximum peaks. Manganese places are clearly displayed in EDAX Spectrum. The percentage of elements recorded and showed that the compounds contained the elements as: Carbon (C), Nitrogen (N), Oxygen (O) and Manganese (Mn). In Table IV, displays the weight, percentage of compound placed in crystal as experimental report. It proves the purity and exacts of the crystal.

In this Zn^{2+} doped LTMA single crystal, doped composition are clearly indicated in the EDAX Spectrum as shown in Fig.4. In which the peaks of Carbon (C), Nitrogen (N), Oxygen (O), Manganese (Mn) and dopant Zinc (Zn) are well defined. In this determination the weight of the elements are given as experimental report Table V.

The atomic mass percentages of C, N, O, Mn in Pure material and inclusion of Zn also experimentally prove in the composition and grown success of raw material.

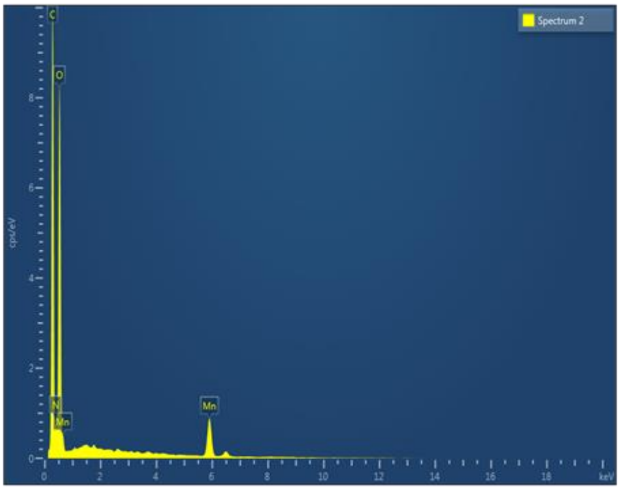

Fig. 3: EDAX Spectrum of LTMA single crystal

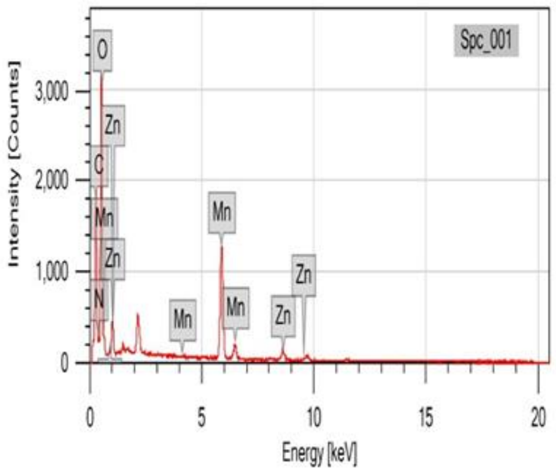

Fig. 4: EDAX Spectrum of Zn^{2+} doped LTMA

TABLE-IV: EDAX ReportofPure LTMA

Element	Line Type	Wt%	Atomic %
C	K series	41.51	49.7
N	K series	6.76	6.94
O	K series	46.79	42.06
Mn	K series	4.94	1.29
Total:		100	100

TABLE-V: EDAX Reportof Zn^{2+}doped LTMA

Element	Line Type	Wt%	Atomic %
C	K series	36.47±0.19	51.73±0.26
N	K series	0.34±0.14	0.41±0.17
O	K series	37.73±0.28 4	40.17±0.30
Mn	K series	21.24±0.22	6.59±0.07
Zn	K series	4.22±0.16	1.10±0.04
Total:		100	100

D. Fourier Transform Infra-Red analysis

The FTIR spectrum of Pure and Zn^{2+} doped LTMA crystals was recorded as shown in Fig. 5. The presence of NH_3^+ is easily identified in the FT-IR spectrum by the broad intense band with the absorption at 3169.25cm^{-1} corresponding to asymmetric stretching mode of NH_3^+. The NH_3^+ symmetric stretching band appears at 3029.80 cm^{-1}. The NH_3^+ symmetric and asymmetric bending mode appears at 1417.94 cm^{-1} and 1629.28 cm^{-1}. The CH symmetric deformation appears in 1346.04cm^{-1} respectively.NH_3^+ Rocking, C-N Rocking, C-C Rocking and C-C-N Rocking appear in 1113.14cm^{-1}.1040.72cm^{-1}, 932.53cm^{-1} and 871.20cm^{-1}. 769.77 cm^{-1}indicates COO^- Bending, 701.35 cm^{-1}and 560.11cm^{-1} indicates the COO^- wagging vibration and COO- Rocking Deformation respectively. NH_3^+ Bending appears at 489.82 cm^{-1}.The OCO rocking frequency found out at 445.18 cm^{-1}.

In the Zn^{2+} doped LTMA single crystal FT-IR spectrum shows small changes in all the peaks with examples of NH_3^+ symmetric stretching and asymmetric bands, CH symmetric deformation and CH_3 group, NH_3^+ and CH_3^+rocking frequencies absorptions and COO^- Bending groups also. Especially at stretched symmetric and asymmetric several peaks are large variations in structure. The OCO rocking frequency found out at 444.79cm^{-1}.Theseare confirming incorporation of the new element such as Zn^{2+} ion in pure LTMA. The compared readings of the Zn^{2+} doped LTMA single crystal tabulated in Table VI.

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TABLE-VI: FTIR Assessments of Pure LTMA andZn^{2+}doped LTMA

Wavenumber			
Pure L-Threonine (14)	**Pure LTMA**	**Zn^{2+} doped LTMA**	**Assignment**
-	3169.25	3168.09	NH_3^+ asymmetric stretching
-	3029.8	3028.32	NH_3^+ symmetric stretching
1625.99	1629.28	1627.65	NH_3^+ asymmetric deformation
1417	1417.94	1417.16	NH_3^+ symmetric deformation
1346.48	1346.04	1345.48	CH symmetric deformation
--	1113.14	1112.73	NH_3^+ Rocking
1040.52	1040.72	1040.89	C-N Rocking
931.5	932.53	932.33	C-C Rocking
871.09	871.2	870.76	C-C-N Rocking
767.9	769.77	769.12	COO^- Bending
701.52	701.35	701.1	COO^- wagging vibration
560.19 C	560.11	559.9	COO^- Rocking Deformation
489.64	489.82	489.3	NH_3^+ Bending
-	445..18	444.79	In plan OCO Rocking

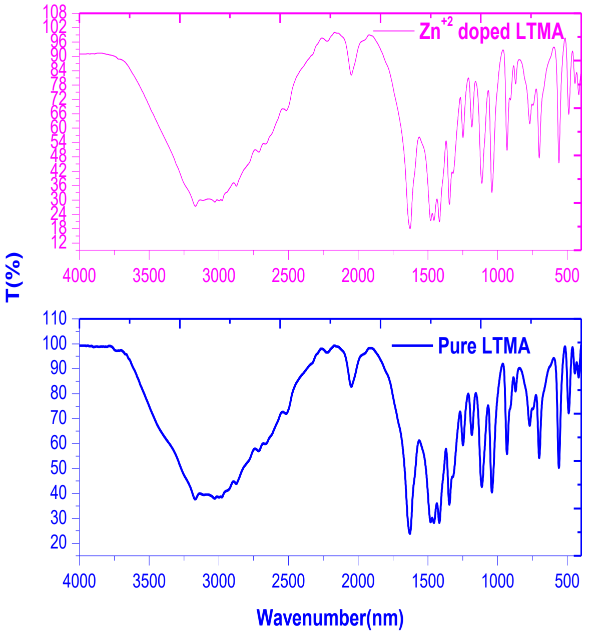

Fig.5: FTIR spectra of pure LTMA and Zn^{2+}doped LTMA

E. Optical Studies

- ***Transmittance Studies***

The transmittance spectra of pure LTMA recorded in the range 190-1200 nm using Lambda 35 spectrometer. The Optical transmittance spectra of LTMA are shown in Fig.6. It reveals that there is no absorption peak in the range of 267 nm to 1100 nm. It can be seen from the transmission curve that below 300nm the transmittance of the grown crystal LTMA slightly decreases. Variation in the transmittance may be due to the presence of manganese acetate. Very low absorbance in the entire visible region would be attributed to the delocalization of electronic cloud through charge transfer. For Zn^{2+} doped LTMA transmittance start to transmit from 200nm to 1100nm. This change in transmittance of pure LTMA and the Zn^{2+} doped LTMA clearly separated reason of Zinc (Zn).

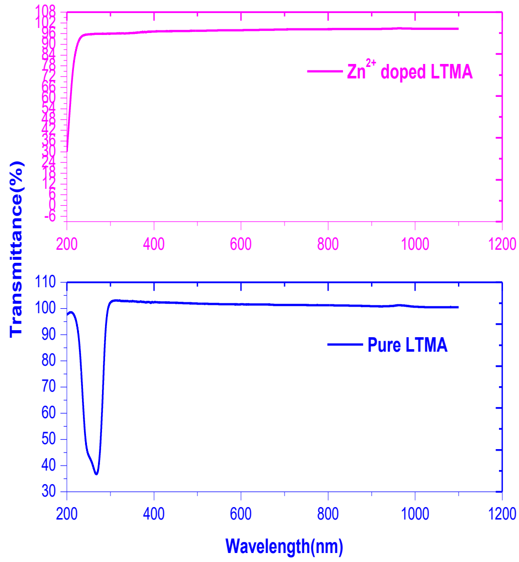

Fig.6: Transmittance of Pure and Zn^{2+}doped LTMA

- ***Absorption Studies***

The absorption spectrum of grown crystal analyzed in the range 190-1200 nm. Fig.7 shown, there is no change from the transmittance spectra. The absence of absorption in the visible region clearly indicates that the grown crystal can be used for photonic applications. The absence of absorption spectrum concluded the good energy band gap value. By knowing optical constants of a material, examine the potential of the material for photonics applications. The optical absorption coefficient (α) calculated by the relation

$$\alpha = (1/t)\ \log(1/T) \qquad (3)$$

Where, t is Thickness of the material and T is Transmittance.

The band gap of the crystal was estimated by Tauc's relation:

$$\alpha h\nu = A(h\nu - E_g)^n \qquad (4)$$

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Where, E_g is the optical band gap of the crystal and A is a constant. So, the energy band gap of grown crystal was determined from the Fig. 7, as 4.3eV and the energy band gap Zn^{2+} doped LTMA is 6.3eV.

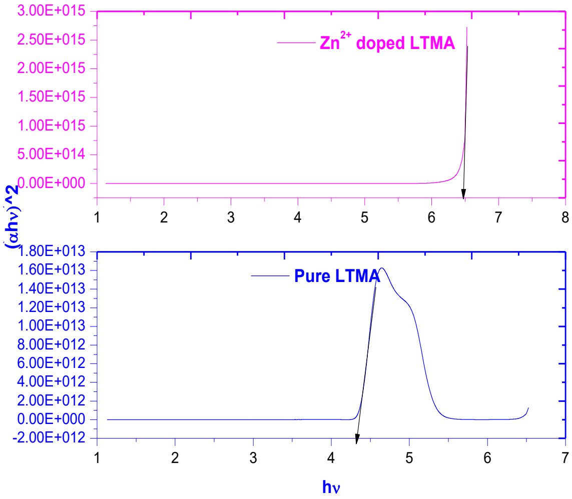

Fig.7: Energy band gapof Pure and Zn^{2+} doped LTMA Single Crystals

3.6 Vicker's Micro Hardness Study (MHD)

The micro hardness study was carried out to determine the mechanical strength of the grown crystals using HMT 2T, SHIMADZU Vickers micro hardness tester. The indentation marks were made on the surface of the both crystals at room temperature by applying load of 25gm, 50gm and 100gm. The H_v was found to increase with the increase in the load from 25m to 100gm and crack occurred at higher loads as shown in Table VII.

The graph (Fig. 7) has been plotted between H_v and applied load P. The Vickers micro hardness number H_v of the crystal was calculated using the relation

$$H_v = 1.8544\ P/d^2\ (kg/mm^2) \qquad (5)$$

Where, H_v is the Vickers hardness number in kg/mm^2, P is the applied load in kg and d is the average diagonal length of the indentation in mm. From the graph it can be observed that the hardness value increased up to 100g and the maximum hardness value of pure and doped crystal was 80.5 $kg/mm^2$and 57.7 kg/mm^2at 100gm.

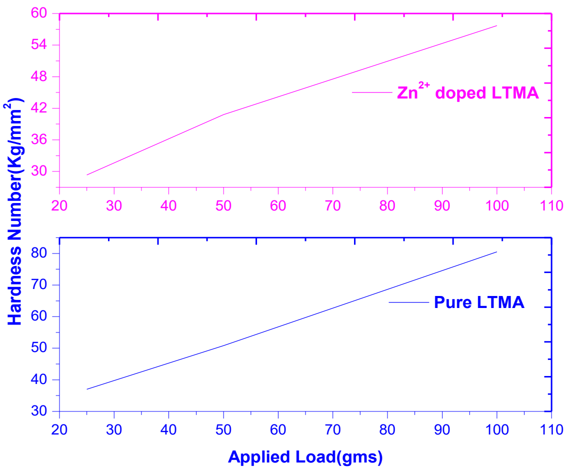

Fig. 8: Graph against Load P Vs Hardness H_v of Pure LTMA &Zn^{2+} doped LTMA

TABLE-VI: Hardness Values Of Pure LTMA and Zn^{2+}doped LTMA

Load P (gm)	Hardness (H_V) Pure LTMA	Zn^{2+} doped LTMA
25	37	29.34
50	50.8	40.8
100	80.5	57.7

IV. CONCLUSION

Novel Single crystals of Pure and Zn^{2+}doped L-Threonine Manganese Acetate were effectively developed by slow evaporation technique. Single precious crystal X-ray diffraction examines found that the gem structure of the two gems becomes Orthorhombic and has bigger volume. The powder X-ray diffraction examination has been done the crystallinity of the contrasts between the unadulterated and Zn^{2+} doped LTMA single crystal. EDAX study affirmed the presence of the dopant Mn in Pure LTMA and Zn in doped Crystal. UV-Vis-NIR considers found that the unadulterated and doped gems have wide straightforwardness in the entire noticeable area and utilizing with their vitality band holes 4.3eV and 6.3eV were resolved. FTIR considers uncover that the nearness of utilitarian gatherings in the gems. The mechanical quality of the both developed crystals was concentrated by Vicker's Micro hardness analyzer.

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