Synthesis and characterization of L-Isoleucine Maleate and L-Isoleucine Oxalate crystals

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***Abstract:** A novel organic single crystal of L-Isoleucine Maleate(LIM) and L-Isoleucine Oxalate(LIO) were grown by slow evaporation solution growth method using water as the solvent at room temperature. X-ray Powder diffraction studies have been carried out in order to calculate the lattice parameter values. The FT-IR spectrum of the materials were recorded on BRUKER IFS 66V FT-IR SPECTROMETER using KBr pellet technique. FT-IR studies revealed the functional groups present in the compounds.*

***Key words:**L-Isoleucine Maleate (LIM), L-Isoleucine Oxalate (LIO), XRD, FT-IR*

1.1 Introduction

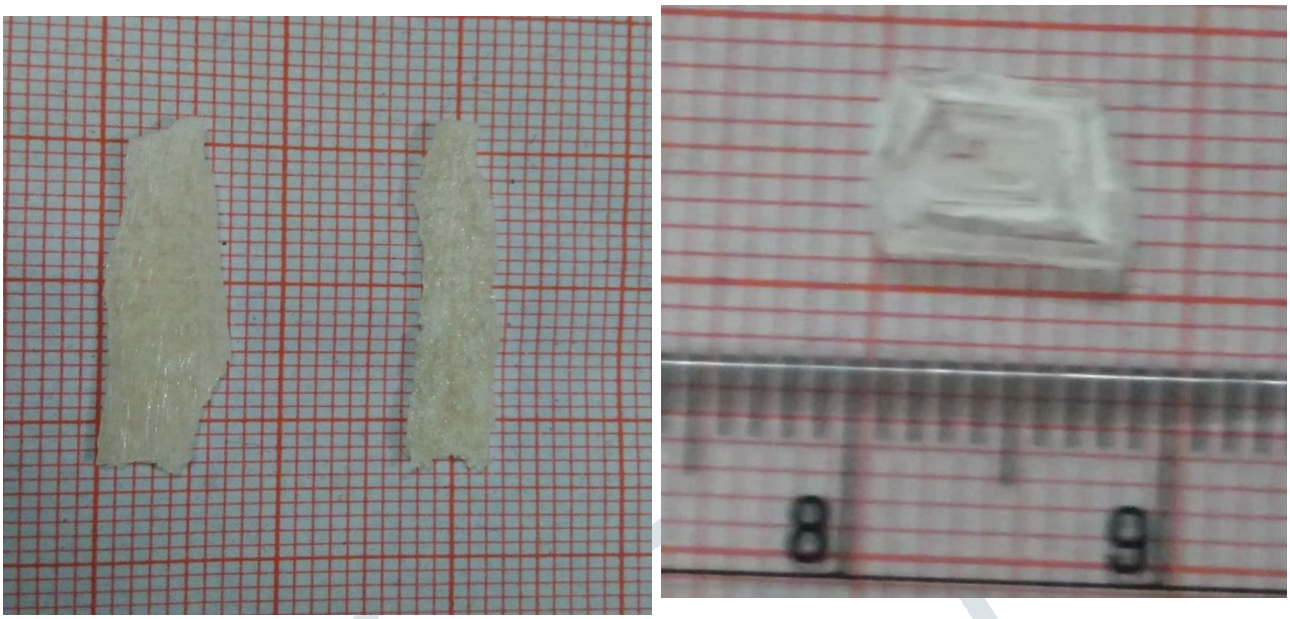
Crystal growth plays an important role in modern technology. A crystal is nothing but a solid in which the constituents atoms molecules or ions are packed in a regular ordered, repeating pattern extending in all three spatial dimensions in the present study. Amino acids are crystalline solids. They are generally soluble in water and insoluble in non-polar organic solvents. The predictable formation of networks or assemblies through intermolecular interactions such as hydrogen bonding or co-ordination bonds in the entire crystal lattice of crystalline materials having desire chemical and physical properties is the main objective of crystal engineering. It is multi disciplinary area and it has implications for materials chemistry, supramolecular chemistry, molecular recognition and biology [1-4]. Among the organic molecules, α-amino acids exhibit specific features of interest such as molecular chirality, absence of strongly conjugated π-bonds, wide transparency window in the entire UV, Visible and NIR regions of the electromagnetic spectrum and zwitter ionic nature as a consequence of internal acid-base reactions[5]. The α-amino acids are the building blocks of poly peptides and proteins and are linked to one another by means of peptide bonds. L-Isoleucine is both glucogenic and ketogenic amino acid . This is one of the amino acids having branched hydro carbon side chains. It is non polar and aliphatic in nature.On the basis of infra red spectroscopic study, the crystal of L-Isoleucine was assumed to belong a rather unusual type in which the molecules two type of conformation[6,7]. In the present paper, the synthesis and single crystal growth of L-Isoleucine organic acids followed by characterization by Powder X-ray diffraction (XRD) and FT-IR have been described.

1.2 Experimental Details

L-Isoleucine Maleate(LIM)was synthesized by the by the reaction between a weak organic maleic acid and the strongly basic amino acid L-Isoleucine (Hi-media) taken in equimolar proportions.

L-Isoleucine Oxalate(LIO) was synthesized by taking L-Isoleucine (Hi-media) and Oxalic acid in equimolar ratio.

The calculated amounts of reactants in each of the reactions were thoroughly dissolved in double distilled water and stirred well for about 6h using a magnetic stirrer to ensure homogenous temperature and concentration over the entire volume of the solutions. The solution was filtered using a whatmann filter paper of pore size eleven μm, transformed to crystal growth vessels and crystallizations were allowed to take place by slow evaporation under room temperature. Transparent colorless LIM and LIO were harvested in a period of 45 days and 60 days, respectively by slow evaporation and are shown in Fig.1(a and b).

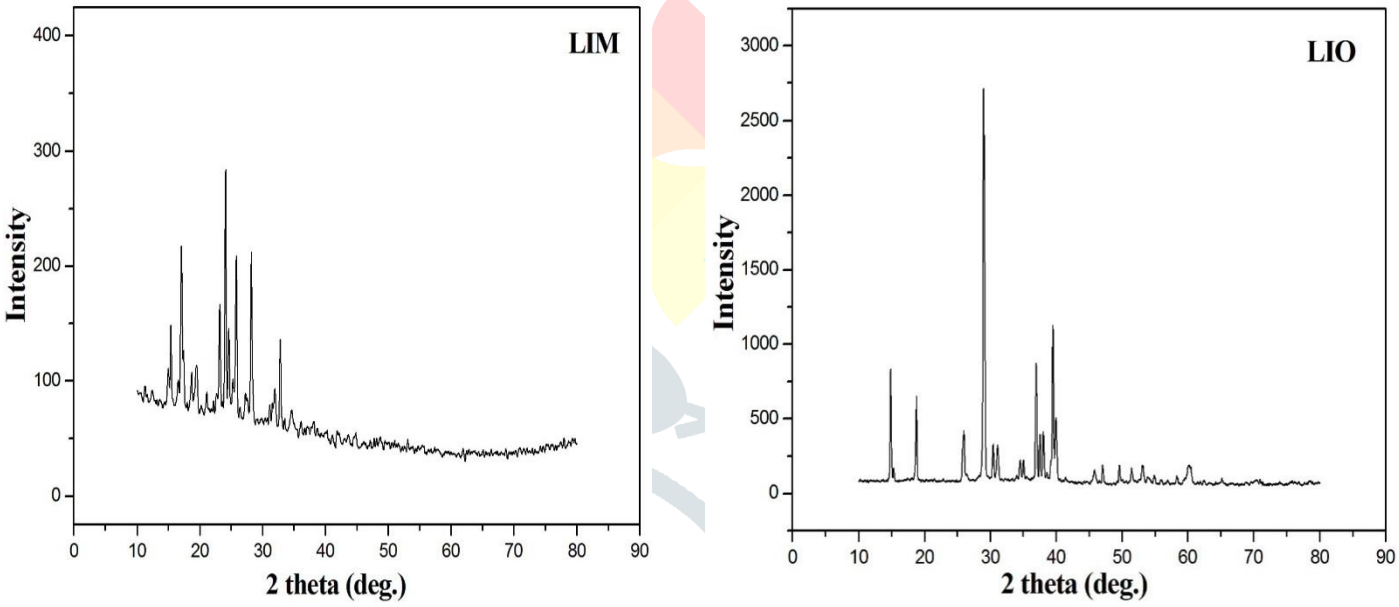


(a) **(b)**

Fig. 1 (a & b) Photograph of LIM and LIO crystals

1.3 XRD Studies

Powder X-Ray diffraction studies of L-Isoleucine Maleate and L-Isoleucine Oxalate crystals are carried out. The samples were scanned for 2θ values from 10^0 to 80^0 at a rate of 2^0per minute. The resulted powder XRD pattern is shown in Fig. 2 (a & b). The sample displays sharp and well resolved diffraction peaks with good crystalline nature.



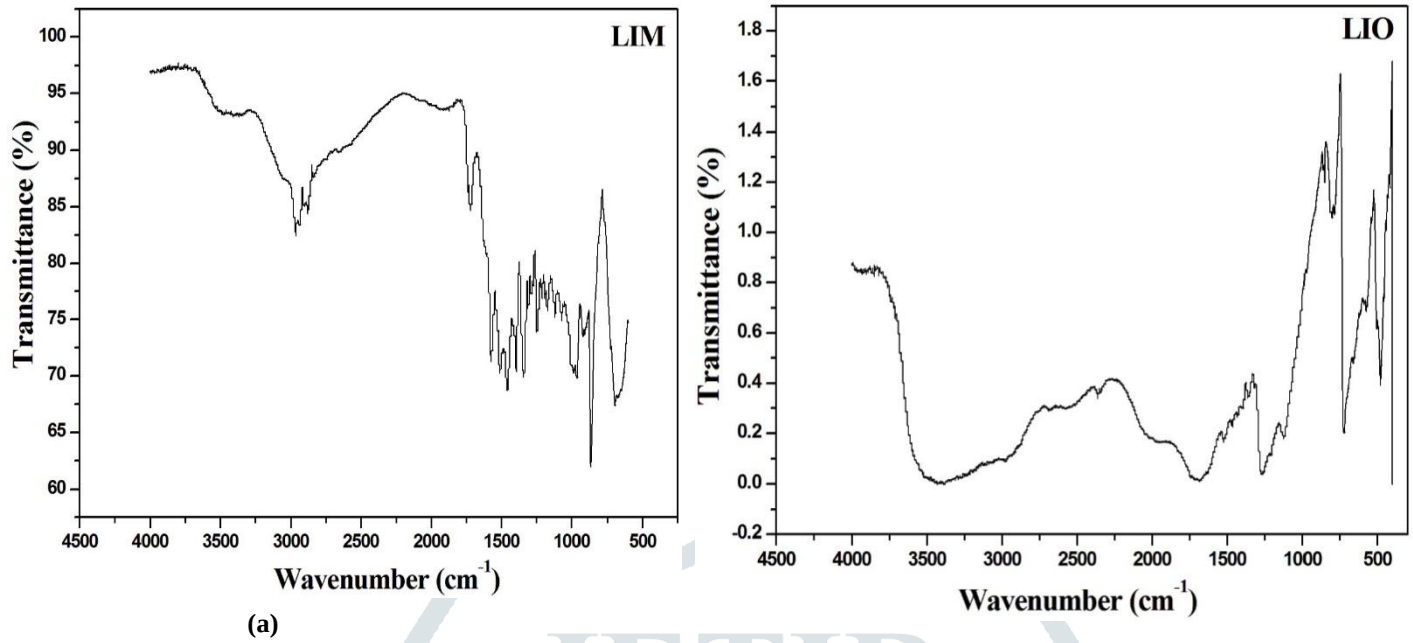
(a) **(b)**

Fig. 2 (a & b) XRD pattern of LIM and LIO crystals

1.4 FT-IR Studies

The FT-IR spectrum of the candidate materials were recorded on BRUKER IFS 667 FT-IR SPETROMETER using KBr pellet technique .The FT-IR analysis of L-Isoleucine Maleate and L-Isoleucine Oxalate crystals have been carried out in the wave number range of 4000 to 450cm^{-1} are shown in fig.3(a and b).In LIM the higher energy region, peak at 2961 cm^{-1} is assigned to C-H stretching vibration [8].The C-O and C=O stretching modes produce peaks at 2878 $cm^{-1}$and 1720 cm^{-1}. Multiple fine structures at the lower energy mode of the envelope indicates the strong hydrogen bonding interaction of NH_3^+ group with strong absorptions of COO^- group at 1568 cm^{-1}[9].The C-O stretching mode is observed at 1307cm^{-1}.The CH_2 wagging and rocking modes produce peaks at 1240 and 866 cm^{-1},respectively. Further medium band observed at about 1069 cm^{-1} is due to C-N stretching. The COO^- scissoring (694 cm^{-1}) vibration is also observed[10].

Investigating the absorption bands of LIO below 1000 cm^{-1} three characteristic bands were identified ,one at 675cm^{-1} (COO^-in plane deformation),one at 580cm^{-1} (COO^- wagging mode) and the third one at 851cm^{-1} (C-C stretching).The band corresponding to NH_3^+asymmetric deformation vibration occurs at 1683cm^{-1} ,COO^- asymmetric stretching at1512 cm^{-1}.High wave number region (3377-2350cm^{-1})contains NH and CH stretching vibration and combination of them. Band at 3377cm^{-1} is due to the presence of water molecules.



(a) **(b)**

Fig. 3 (a & b) FTIR spectrum of LIM and LIO crystals

The observed vibrational frequencies and their tentative assignments of LIM and LIO are listed in Table 1 (a & b)

Table1a. Band Assignments of LIM

Wave number cm^{-1}	Assignments
2961	C-H stretching
2878	C-O stretching
1720	C=O stretching
1568	COO^- asymmetric stretching
1307	C-O stretching
1240	CH_2wagging
1069	C-N stretching
866	CH_2 rocking
694	COO^-scissoring

Table 1b. Band Assignments of LIO

Wave number cm^{-1}	Assignments
3377-2350	NH and C-H stretching vibration
1683	NH_3^+asymmetric deformation
1512	COO^- asymmetric stretching
1262	CH_2 wagging
1080	C-N stretching
851	C-C stretching
675	COO^- plane deformation
580	COO^- wagging mode
478	N-H deformation

1.5 Conclusion

Transparent crystals of **L-Isoleucine Maleate and L-Isoleucine Oxalate were** grown by slow evaporation solution growth method. Grown crystals were characterized by X-ray diffraction. The modes of vibration of the molecules and the presence of functional groups were identified using FT-IR technique.

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