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Synthesis, Growth and Characterization Studies on Butylamine, Ascarbic Acid and Leucine (BAL) Mixed Single Crystal By Slow Evaporation Method

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Abstract:*The Synthesis Of Butylamine , Ascarpic Acid And Leucine Mixed Crystals Done Under Low Temperature Slow Evaporation Method. The Synthesized Crystals Characters Are Studied Using Xrd, Ft-Ir, Sem, And Uv-Visible Processes. Xrd Studies Are Used To Carry Out The Crystallographic Analysis. Ft-Ir Studies Are Used To Observe The Functional Groups Of Butylamine , Ascarpic Acid And Leucine Mixed Crystals. Sem Studies Are Used To Study The Morphological Identification. Uv-Visible Studies Are Used To Study The Optical Absorbance Analysis.*

Key Words: *Growth From Solution, Single Crystal, Xrd, Ft-Ir, Sem, And Uv-Visible.*

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I. Introduction

Crystal Growth Is An Interdiscipilinary Subject Covering Physics, Chemistry, Material Science, Chemical Engineering, Metallurgy, Crystallography, Mineralogy, Etc., At One Time Natural Specimens Were The Only Source Of Large And Well Formed Crystals. Now – A- Days, Large Crystals Are Being Grown Artificially In Laboratories And Being Used In Many Scientific And Technological Fields. In Recent Years, The Need Of Nonlinear Optical Single Crystals Are Very Much Useful In The Field Of Second Harmonic Generation, Fiber Optic Communication, Electro –Optic Modulation, Etc [1, 2].The Formation Of A Single Crystal Structure In Materials Is Affected By Its Atomic Or Ionic Mobility Behaviour. In The Past, There Has Been A Growing Interest On Crystal Growth Processes, Particularly In View Of The Increasing Demand Of Materials For Technological Applications [3-5].The Major Advantages Are The Anisotropy, Uniformity Of Composition And The Absence Of Boundaries Between Individual Grains, Which Are Essentially Present In Polycrystalline Materials. The Strong Influence Of Single Crystals In The Present Day Technology Is Evident From The Recent Developments In The Above Mentioned Fields. Hence, In Order To Achieve Performance From The Device, Good Quality Single Crystals Are Needed. Crysatlline Structure Is More Likely Used In Many Applications Compare To The Non-Crystalline Structure. Amino Acids Are Molecules Containing An Amine Group, A Carboxylic Acid Group And A Side Chain That Varies Between Different Amino Acids. Recent Studies Indicate The Leucine Favorably Forms Several Salts With Organic Or Inorganic Acids. In This Paper, The Method Of Crysatl Growth With Emphasis On Low Temperature Solution Growth Technique Was Described. The Synthesized Crystals Characters Are Studied Using Xrd, Ft-Ir, Sem, And Uv-Visible Processes.

II. Growth Of Crystals

Synthesis Of Butylamine, Ascarpic Acid And Leucine (Bal) Mixed Crystals

The Synthesis Of Mixed Crystals Are Done Under Low Temperature Slow Evaporation Method. 5g Of Each Butylamine , Ascarpic Acid And Leucine Is Weighed Accurately And Dissolved In 50ml Of Double Distilled Water And Stirred It Vigorously At 28^0c For 15minutes. Then, The Saturated Solution Was Decanted Into The 100ml Beaker. The Remaining Solute Was Dissolved In 5ml Of Distilled Water; The Solution Is Stirred Continuously For 15 Minutes. The Above Process Is Continued Until The Solute Is Fully Saturated. After The Saturation, The Solution Was Taken In A 100 Ml Beaker Which Was Tightly Closed With The Filter Paper, So That The Rate Of Evaporation Could Be Minimized. Good Transparent Mixed Crystals Are Obtained After 4 Weeks. The Grown Crystals Are Shown In Figure.1. Then, The Synthesized Crystals Are Characterized Using Xrd, Sem, Ft-Ir, Uv-Visible Studies.

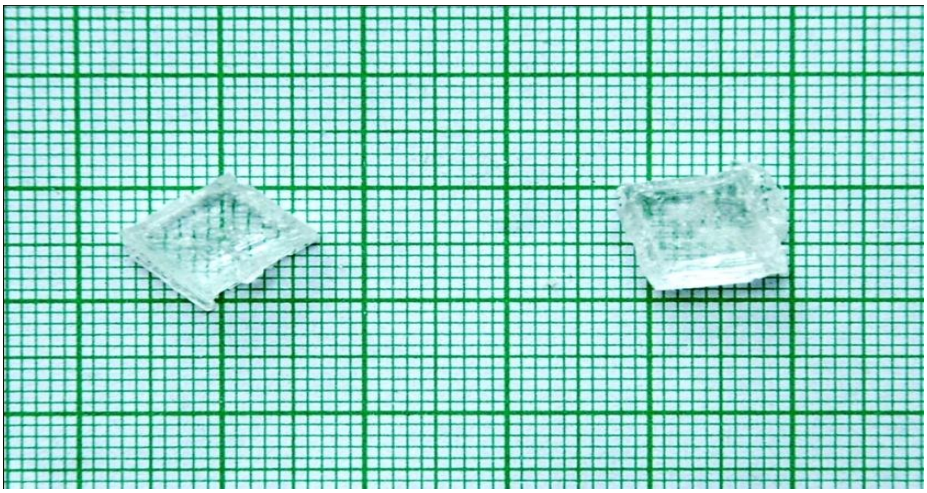

Figure.1. Butylamine, Ascarpic Acid And Leucine (Bal) Mixed Crystals

III. Characterization Methods

3.1. Xrd Analysis

X-Ray Powder Diffractograms Were Recorded For Butylamine ,Ascarpic Acid And Leucine Mixed Crystals At Room Temperature. The Sample For The Powder X-Ray Diffraction Was Prepared By Crushing A Small Piece Of A Crystal Into Fine Powder. The Fine Calcined Particles Were Characterized For Crystal Phase Identification By Powder X-Ray Diffraction (Xrd) Using A Xrd-6000 (Shimadzu, Japan). The Powder Was Then Spread Over A Glass Plate With Uniform Thickness. Methanol Was Used As A Binder To Spread The Powder On The Glass Plate With Uniform Thickness. The Radiation Used Was Ni Filtered Cu Kα (Λ=1.5418å). The Scan Speed Was 10°/Minute And The Scan Range Was 4.0000°-90.0000°.

3.2. Ft-Ir Spectral Analysis

Fourier Transmission Infrared (Ft-Ir) Spectra Of The Butylamine, Ascarpic Acid And Leucine Mixed Crystals (As Pellets In K_{br}) Were Recorded Using Ir Affinity-1 Fourier Transmission Infrared Spectrometer (Shimadzu, Japan) In The Range Of 4000-400 Cm^{-1} With A Resolution Of $1cm^{-1}$.

3.3. Sem Analysis

The Particle Size And External Morphology Of The Fine Calcined Powders Were Characterized By Scanning Electron Microscopy Jsm-6390 (Shimadzu, Japan).

3.4. Uv-Visible Spectrum

The Optical Absorption Spectra Was Studied Using Uv–1700 Pharma Spectrophotometer (Shimadzu, Japan) In The Range Of 200–800 Nm.

IV. Results And Discussion

4.1. Xrd Spectrum

Single Crystal X-Ray Diffraction Analysis Was Carried Out To Determine The Lattice Constants.The Powder Xrd Patterns Of Butylamine (**$C_4h_{11}n$**) , Ascarpic Acid(**$C_6h_8o_6$**) And Leucine(**$C_6h_{13}no_2$**) Mixed Crystals Are Shown In Figures **A=B=5.070, C=16.82 ; A=14.234 B=11.052, C=5.860 ; A=4.01 , B =2.68 , C = 1.24** And The Data's Are The Lattice Parameters And The Crystal System Are Presented.The Data Indicates That Bal Crystal Crystallizes In Orthorhombic Structure [6]. To Confirm The Xrd Data, Powder Xrd Studies Were Also Carried Out For The Sample. The Grown Crystals Of Bal Were Crushed Into Fine Powder And Powder X-Ray Diffraction Analysis Has Been Carried Out Using A Powder X-Ray Diffractometer. The Recorded Pattern Is Shown In Figure.2. The Sharp Peaks Of Xrd Pattern Indicate High Degree Of Crystalline Structure Of Grown Crystal. The Observed Diffraction Pattern Has Been Indexed And Miller Indices Were Estimated By Indexing Software Package.

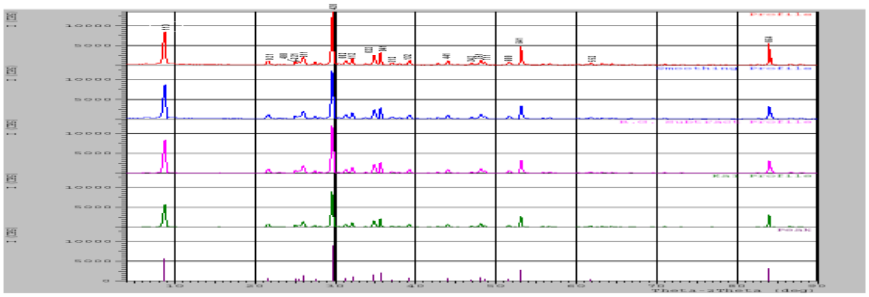

Figure.2. Xrd Diffraction Of Butylamine, Ascarpic Acid And Leucine (Bal) Mixed Crystals

4.2. Ft-Ir Spectral Analysis

The Presence Of N-O Stretching Asymmetric Is Indicated By A Sharp Absorption Peak At 1649.14cm^{-1}. The Sharp Absorption Peak's Observed At 1161.15cm^{-1} Respond To Co_2 Symmetric Stretching. The Sharp Absorption Peak Observed At 1788.01cm^{-1}. The Absorption Peak Observed At 1379.10cm^{-1} Respond To C=O Asymmetric Stretching Of Acid Group. Stretching Of N-O Linkage Produces Very Strong & Broad Peak At 837.11cm^{-1} Is Attributed To Nitrate Group. The Sharp Absorption Observed At Longer Wavelength Near 725.23 Cm^{-1} Likely Result From No_2 Bending Vibration.

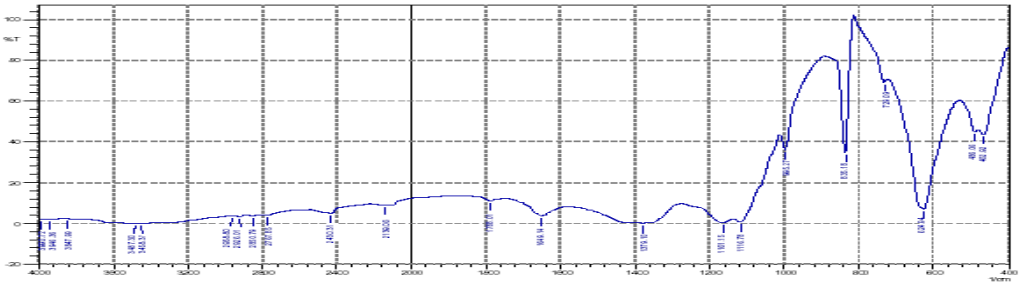

Figure.3. Ft-Ir Spectrum Of Butylamine, Ascarpic Acid And Leucine (Bal) Mixed Crystals

Table.1. Vibrational Assignments For Bal Crystal

Frequency Cm^{-1}	**Assignment**
3400	Oh - Stretching
1649.14	N-O Stretching Asymmetric
1161.15	Co_2 Symmetric Stretching
1788.01	Sharp Absorption
1379.10	C=O Asymmetric Stretching Of Sulphate Group
837.11	N-O Linkage Of Nitrate Group
729.09	No_2 Bending Vibration

4.3. Sem Analysis

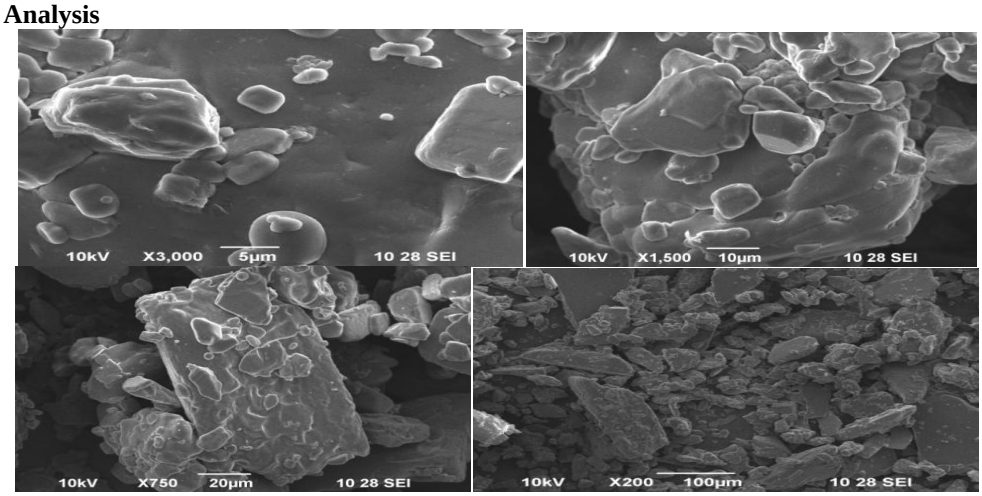

Figure.4. Scanning Electron Micro Graph Of Butylamine , Ascarpic Acid And Leucine (Bal) Mixed Crystals

Typical Sem Micrographs For Powder, Synthesized Powders At Different Magnifications Are Shown In Figure.4 It Is Evident From The Photograph That The Background Of The Surface Was Crystalline And Morphology Crystalline Butylamine, Ascarpic Acid And Leucine Mixed Crystals The Size Of The Particle Determined From The Sem Images Are 5-100 µm.

4.4. Uv-Visible Absorbance

The Lower Cut-Off The Weak Concentration Of Butylamine, Ascarpic Acid And Leucine Mixed Crystals Is Around Strong Absorbance 1.253 In The Mid Range Uv-302 Nm And High Concentration Of Butylamine, Ascarpic Acid And Leucine Mixed Crystals Is Around Strong Absorbance 3.010 In The Mid Range Uv-301 Nm. The Mixed Crystals Of Butylamine , Ascarpic Acid And Leucine Mixed Crystals Are Used In The Field Of Medical Phototherapy And Dermatological, Solar Experimentation.

A) Weak Concentration

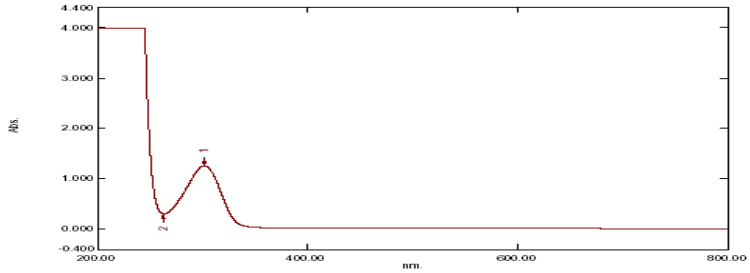

Figure.5a. Uv-Visible Absorption Spectrum Of Butylamine, Ascarpic Acid And Leucine (Bal) Mixed Crystals

No.	P/V	Wavelength	Abs.
1		302.00	1.253
2		263.00	0.297

B) High Concentration

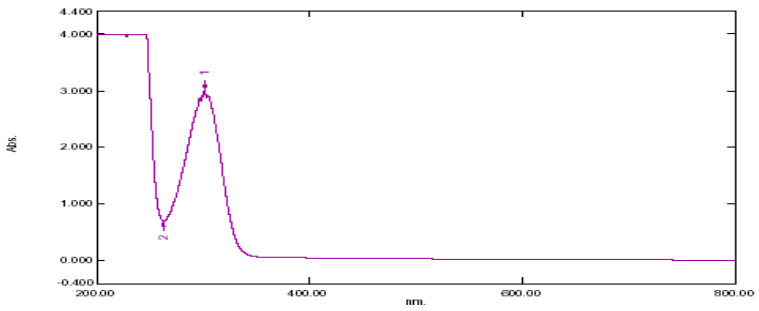

Figure.5b. Uv-Visible Absorption Spectrum Of Butylamine, Ascarpic Acid And Leucine (Bal) Mixed Crystals

No.	P/V	Wavelength	Abs.
1		301.00	3.010
2		262.50	0.696

V. Conclusion

Butylamine, Ascarpic Acid And Leucine (Bal) Mixed Crystals Have Non Centro Symmetry Nature Crystals Were Successfully Grown At Low Temperature By Slow Evaporation Method. Butylamine, Ascarpic Acid And Leucine (Bal) Mixed Crystals Have Positive Temperature Co-Efficient To Solubility, Xrd Studies

Reveal That Butylamine, Ascarpic Acid And Leucine (Bal) Mixed Crystal Crystallizes In Orthorhombic Structure. Ft-Ir Analysis Confirmed The Presence Of Various Functional Groups In The Grown Butylamine, Ascarpic Acid And Leucine (Bal) Mixed Crystal. The Background Of Scanning Electron Microscope (Sem) Shows The Formation Of Microparticles. The Uv-Vis-Spectrum Indicates The Weak Concentration Of Butylamine, Ascarpic Acid And Leucine (Bal) Mixed Crystals Is Around Strong Absorbance 1.253 In The Mid Range Uv-302 Nm And High Concentration Of Butylamine, Ascarpic Acid And Leucine (Bal) Mixed Crystal Is Around Strong Absorbance 3.010 In The Mid Range Uv-301 Nm.

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