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Growth and Characterization of Potassium Aluminium Sulphate Dodecahydrate Single Crystals

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ABSTRACT:

Single crystals of Potassium aluminium sulphate dodecahydrate with very high transparency were grown form aqueous solution by slow evaporation technique. Single crystals X-ray diffraction analysis reveals that the crystal belongs to cubic system with the space group Pa3. The dielectric studies were carried at room temperature. The spectra analysis and conductivity studies were carried out for single crystals.

Keywords: Solution growth; Single crystals XRD; FTIR; Conductive; Dielectric studies.

1. INTRODUCTION:

In recent years, the growth of the single crystals has assumed enormous importance for both academic and applied research and communication [1]. Single crystals are required mainly for the construction of solid state devices. Number of devices has been identified effectively for optical and electronic phenomena. These include frequency controlled oscillators by quartz crystal, polarisers by calcite crystals, transducers by ammonium dihydrogen phosphate crystals and radiation detectors by alkali halides crystals etc. In most of these applications, the major role is played by bulk single crystals. In order to fabricate devices which high efficiencies we need to have a thorough understanding of the basic properties of crystals and understand how exactly the materials can be grown.

The history of the crystal growth and classification of crystal growth methods have been reviewed. Various methods of crystal growth, especially the fundamental and experimental aspect of low temperature solution growth technique have been explained in detail. The importance of nucleation kinetics to optimize the growth solution for growing optical quality bulk crystals is presented.

Presently, an attempt has been made to grow single crystals of Potassium Aluminium Sulphate Dodecahydrate (PASD) [KAl(SO4)2.12H2O]. The melting point of Potassium aluminium sulphate dodecahydrate crystal is 297□ C. The PH value of PASD is 2. FT-IR, Single XRD, Powdered XRD, Dielectric studies and Complex Impedance have been carried out.

ISSN No. 2395-0900
International Journal of
Engineering Studies and Technical Approach
IJESTA

2. EXPERIMENTAL METHODS

Preparation of Sample:

The Silicon samples are synthesized commercially outside. Potassium aluminium sulphate dodecahydrate [KAl(SO4)2.12H2O] single crystals are grown from aqueous solution by slow evaporation technique using water as solvent. The starting materials are synthesized by dissolving high purity Potassium aluminium sulphate dodecahydrate in double distilled water. The solution can be continuously stirred a period of eight hours. Constant stirrering of the solution in either direction was employed to homogenize the saturation solution in the beaker. The saturation solution was purified using 1 Watt mann filter paper. The saturated solution can be allowed to evaporate excess amount of water at room temperature 303K to obtain seed crystals due to spontaneous nucleation within ten days.

The single crystals of Potassium aluminium sulphate dodecahydrate with dimensions of 1.5×1.4×0.6 cm3 are successfully grown from aqueous solution by slow evaporation technique in a period of two weeks. The photographs of the grown single crystals PASD is shown in the Fig.1. The grown crystal can be studied by various characterization of method.

The grown crystals of Potassium aluminium sulphate dodecahydrate are subjected to different characterization studies like Single and powdered XRD, FTIR, Dielectric Studies and Impedance measurements with LCR meters.

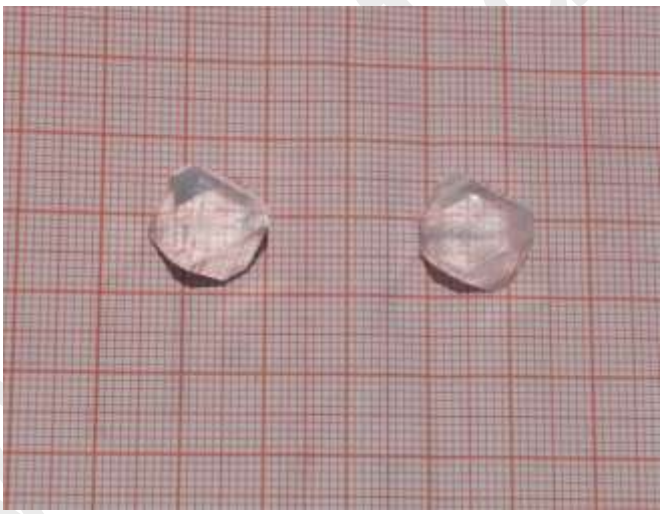

Fig.1 Grown single crystals of PASD

3. RESULTS AND DISSCUSSION

3.1 Single Crystal XRD

Single crystal X-ray diffraction analysis of PASD are carried out using Enraf Nonius CAD 4 single X-ray diffractometer with MoKα radiation. The XRD study reveals that the crystal PASD belongs to cubic system with lattice parameters. The grown alum crystals are found to be Centro symmetric in nature. The lattice parameters values are recorded from single crystal X-ray diffraction analysis are given in the Table 1.

ISSN No. 2395-0900
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Table 1: The Single Crystal X-Ray Diffraction data for PASD

Parameters	PASD single crystal $KAl(SO_4)_2.12H_2O$
System	Cubic
Space group	$P_a3\,(\mathrm{Th}^6)$
Lattice parameters	a=12.24 Å b=12.24Å c= 12.24Å α=β=γ= 90°
Volume	998 $(Å)^3$
Z	4

3.2 Powder XRD Data:

The X- ray diffraction patterns are shown in the Fig.2 X- ray diffraction measurements are made for Potassium Aluminium Sulphate Dodecahydrate [KAl(SO4)2.12H2O] using STOE Powder diffractometer.

The Powder X- ray diffraction is used to determine the particle size and structural parameters of the material. It is observed from the XRD pattern shown in the Fig.2. The PASD single crystals have prominent intensity peaks at 21°, 22 °and 28°. However the entire pattern shows sharp and intense peaks ensuring the crystalline nature of PASD single crystals. Fig.2. Powder X-ray spectrum of PASD Single Crystal

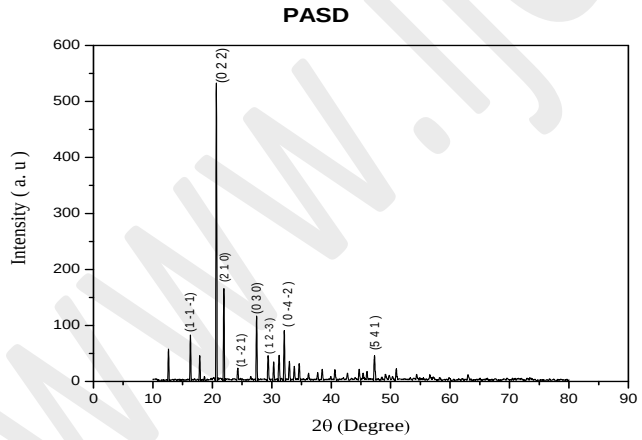

Fig.2. Powder X-ray spectrum of PASD Single Crystal

3.3 FT-IR Analysis of Potassium Aluminiumsulphate Dodecahydrate [KAl(SO4)2.12H2O] (PASD)

FTIR Spectra of Potassium Aluminium Sulphate dodecahydrate [KAl(SO4)2.12H2O] (PASD) recorded are shown in Fig.3. The IR Spectra as studied, may be observed in the

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International Journal of
Engineering Studies and Technical Approach
IJESTA

Table 2 and it can be subdivided into four spectral regions: (a) 3700 to 2500 cm-1 region where OH fundamental vibrations are observed. (b) 1800 to 1400 cm-1 range where the water bending modes are observed. (c) 1200 to 800 cm-1 region shows bands due S-O stretching vibrations and (d) the low wave number region (700-645 cm-1) displays a single mode for(SO4)2-

The OH bending of water molecules were observed in the band of 1647cm-1. A sharp band in the spectral wavelengths of 1528.06cm-1 and 1356.22 cm-1 is due to asymmetric bending modes of NH4+ ions. Sharp bands in the spectral wavelength of 700 cm-1 and 611 cm-1 were due to wagging vibrations of water molecules co-ordinated to the metal cation. From the FTIR analysis of PASD alum it is evident, in practically all α alums, the hydrogen bonds in which the water molecules co-ordinated to the trivalent metal cations act as proton donors. A medium absorption band in the wavelength 504 cm-1is unambiguously due to the bending mode of Al(H2O)63+ ion [14].

Table 2: The FTIR Band assignments for PASD

Wave number (cm^{-1})	**Assignment**
504	Bending Mode of $Al(H_2O)_6^{3-}$ ions
700	Wagging liberation of water molecules coordinated the metal cations
1356	Asymmetric Bending Mode of NH_4^+ ions
1528	Asymmetric Bending Mode of NH_4^+ ions

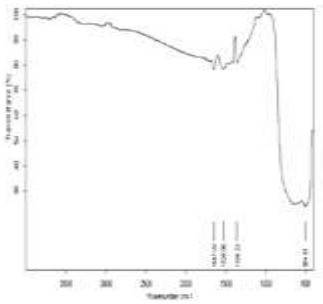

Fig.3. FTIR spectrum of PASD Single Crystals

3.3 Dielectric Studies

Single crystals of Potassium aluminium sulphate dodecahydrate of thickness 1 mm were subjected to dielectric studies at different temperatures 35, 55, 75˚C for various frequency ranging from 50 Hz to 5 MHz using LCR HITESTER. The dielectric constant is evaluated is using the relation

ISSN No. 2395-0900
International Journal of
Engineering Studies and Technical Approach
IJESTA

$$\varepsilon r = Ct/\varepsilon 0\ A$$

where d is the thickness and A is the area of the cross section of the grown crystal. The variations of dielectric constant as a function of frequency at different temperatures are shown in Fig.4. From the graph, the dielectric constant is seen to decrease with increase in frequency. The very low value of dielectric constant at higher frequencies is important for the fabrication of materials towards ferroelectric, photonic and electro – optic devices. The dielectric loss is also studied as a function of frequency at different temperatures and is shown in Fig.5. These curves suggest that the dielectric loss is strongly dependent on the frequency of the applied field, similar to that of dielectric constant.

3.3.1 Dielectric Constant and Dielectric loss

The dielectric constant of a material is composed of four contributions: electronic, ionic, orientational and space charge polarizations. All these may be active at low frequencies. Fig.4 shows the variation of dielectric constant Potassium Aluminium Sulphate Dodecahydrate at different temperatures varying from 308 to 348K. The dielectric constant rises a value 0.5 at 0.1Hz. It, however decreases as the frequency increases to 1Hz, after which it shows a little dependence on frequency. At 328K, ε' is raised to 0.9 at 1Hz which starts falling as the frequency is increased to 7 Hz after which it tries to attain saturation. The same is true for 348K raised to 1.4 at 5 Hz. The dielectric constant of PASD acquires space charge polarisation and orientational polarisation. This shows that dielectric constant is strongly depend on temperature [7, 8].

Fig.5 shows the dependence of the dielectric loss on log frequency in the range 0.5 kHz-5MHz. The data are provided directly by the instrument and represented in the Fig.5 by solid lines. The dielectric loss decreases with increase in log frequency at all the temperatures ranging from 308 to 348 K. The maximum value of dielectric ranging from 6 to 10 shifts towards the higher frequency side as the temperature increases from 308 to 348K. In the higher frequency range, the dielectric loss appears to achieve saturation at all the temperatures [7,8].

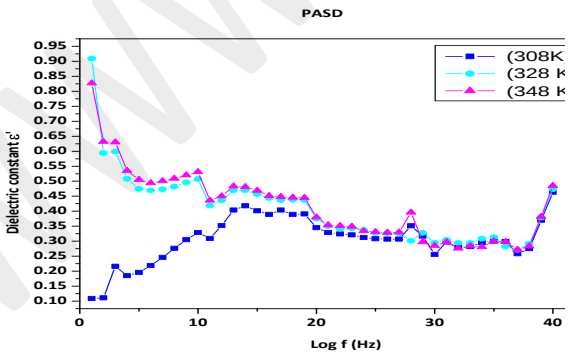

Fig.4. Plot of dielectric constant vs frequency of the applied field.

International Journal of Engineering Studies and Technical Approach
IJESTA
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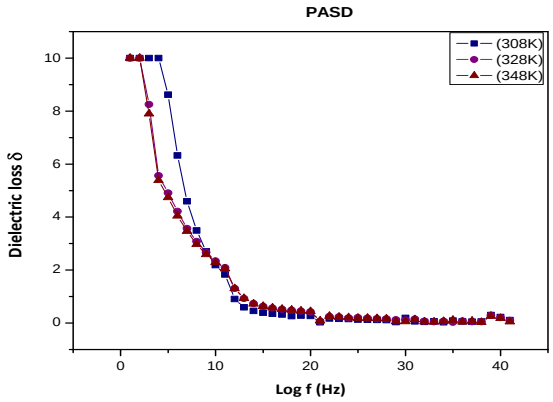

Fig.5. Plot of dielectric loss vs frequency of the applied field

3.4. CONDUCTIVITY STUDIES

3.4.1 Electrical Conductivity Results

This section deals with the results of the complex impedance analysis carried out on Potassium Aluminium Sulphate Dodecahydrate [KAl(SO4)2.12H2O] single crystals.

3.4.2 Complex impedance plots

The typical complex impedance plots obtained for the Potassium Aluminium Sulphate Dodecahydrate [KAl(SO4)2.12H2O] crystal at temperatures 308,328,348, K shown in Table 3. The point of intersection of the impedance plots on the real axis indicates the bulk resistance (Rb) of the crystal.

Table 3. Conductivity values obtained for crystal PASD

Temperature (K)	Bulk resistance R_b (ohms)	Conductivity $\sigma = \frac{t}{A.R_b}$ Scm^{-1}
308	22500000	3.154×10^{-10}
328	13000000	5.4593×10^{-10}
348	11500000	6.1713×10^{-10}

It is observed in Fig.6 that as the temperature increases, the point of intersection is shifted towards the origin. Hence, it is quite evident that the bulk resistance of the samples decreases with increase in temperature, resulting in an enhancement of electrical conductivity at higher temperatures. Activation energy obtained from the graph is 0.1851eV for PASD.

Fig. 7 shows the variation of log σT with temperature corresponding to the frequency of appliedfield. Fig.6. Complex impedance plot obtained for the crystal (PASD) at 308 K

International Journal of Engineering Studies and Technical Approach
IJESTA
ISSN No. 2395-0900

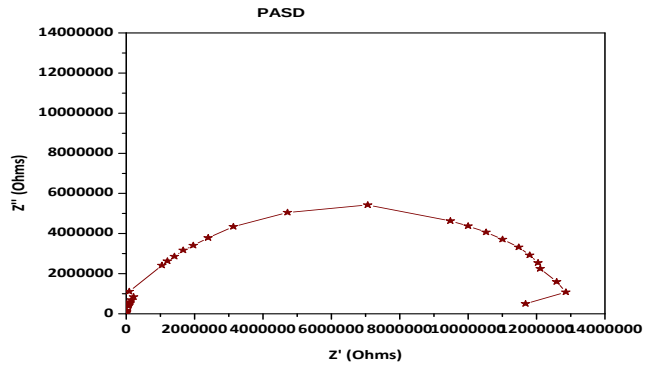

Fig.6. Complex impedance plot obtained for the crystal (PASD) at 308 K

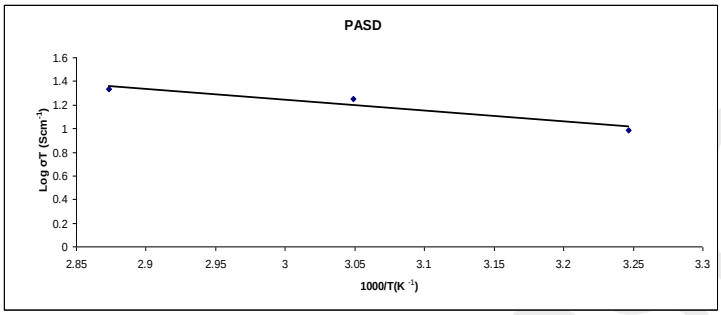

Fig.7. Variation of Log **σT vs** $\frac{1000}{T}$ **for PASD**

5. SUMMARY

The present work is aimed to investigate the grown single crystals of Potassium Aluminium Sulphate Dodecahydrate (PASD).Grown crystals were subjected to various characterizations which are essential for an assessment of the crystals suitability for device fabrications. Single crystal X-ray diffraction analysis enumerates the lattice unit parameters and the structure. FTIR studies were carried out to identify the functional groups present in the materials. The variation of the dielectric constant (ε'), the dielectric loss (tanδ) and the conductivities (σ) at different temperatures (308-348 K) and frequencies (0.5 kHz-5 MHz) of the applied alternating current field is analyzed.

CONCLUSION

The grown crystal are characterised by various tools like XRD, FTIR, Dielectric studies, analysis of impedance with LCR meter measurements.

- Single crystal XRD confirms that the material crystallizes in the cubic system with a space group Pa(Th).
- Powdered XRD reveals the crystalline nature of the material.
- FTIR study confirms the various functional groups present in the crystal and vibrational structure of the compound has also been elucidated.

- Dielectric constant and dielectric loss of the single crystal are strongly dependent on the frequency of the applied field.
- The ionic conductivity is present in the Potassium aluminium sulphate dodecahydrate single crystals.

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