Research Paper

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Structural, Optical and Dielectric Studies of Compound Crystal of Zinc Sulphate Hepta Hydrate Doped with Nickel Sulphate Hepta Hydrate.

Engineering

KEYWORDS : Crystal growth: Compound crystals of zinc sulphate hepta hydrate doped with nickel sulphate hepta hydrate; Structural, optical and dielectric studies.

S.Indira	Research scholar, Karpagam university, Coimbatore.
K. Ramesh	Vidyaa vikas college of engineering and technology, Tiruchengode.
G. Viruthagiri	Department of physics, Annamalai University, Annamalainagar.

ABSTRACT

Zinc sulphate hepta hydrate and nickel sulphate hepta hydrate are the inorganic compounds doped together to form a compound crystal. In this study, a compound crystal of size 6mmx5mmx4mm were grown in 20days by slow evaporation method at room temperature. The grown crystals were characterized by various studies such as powder crystal XRD which show the orthorhombic nature of the crystal, the optical quality and band gap for the grown crystal is studied by UV-VIS spectroscopy, the vibrational analysis of the crystal is done with FTIR spectroscopy and the major functional groups and their assigned vibrations are discussed, the temperature dependence of Dielectric constant and dielectricloss and AC conductivity have been studied and their results are discussed.

Introduction:

The crystallization of non linear optical materials is becoming more significant for finding the more efficient and effective non linear materials. These materials are of greater importance as these important inorganic substances have wide applications in the field of agriculture, medicine,optical communication,optical memory storage and luminescence studies *(Jagadeesh M.R et al.,2014)*. On this material Mahadevan and coworkers have reported on forming mixed crystals with zinc sulphate and nickel sulphate and with some other studies *(C. Mahadevan,1998)*. The grown compound crystals are hydrogen bonded crystals belonging to the orthorhombic crystal system with tetra molecular unit cell. In this article, the grown crystals are subjected to Structural, optical and dielectric studies. The results are reported for the effect of dielectric constant, dielectric loss and AC conductivity of the grown crystal with temperature.

Material and method:

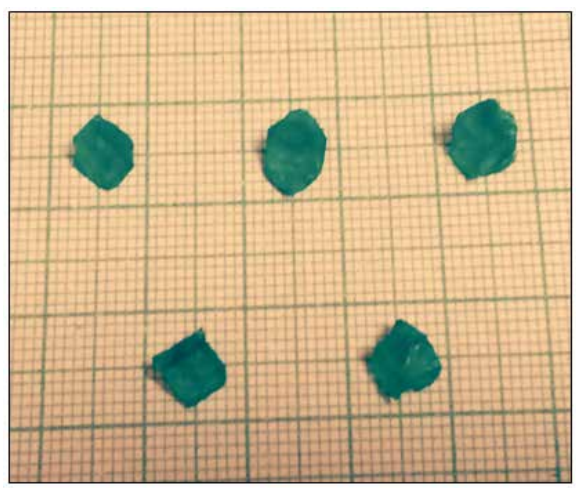
At least 90% of all the crystals are produced by low-temperature solution growth method are soluble in water. Crystals will grow from solutions if the solution is supersaturated i.e. it contains more solute than it can hold. Saturated solutions are those mixtures, which can retain their equilibrium indefinitely, in contact with solid phase, with respect to which they are saturated. The solubility of most substances increases with temperature i.e., positive temperature co-efficient. Crystals can only grown from supersaturated solutions, which contains an excess of the solute, above the equilibrium value.

To grow crystals, Analytical reagent grade (AR) chemicals were used. The crystals were grown by solution method with slow evaporation technique. The solvent used to grow crystals was deionised water. To grow mixed crystals of zinc sulphate hepta hydrate and nickel sulphate hepta hydrate, the salt were taken in the molar ratio of 1:1 and the super saturated solution was prepared. The solution was filtered and kept in a separate beaker covered with perforated paper for slow evaporation and allowed to evaporate at room temperature. It took 20 days to obtain the compound crystals.

$ZnSO_4.7H_2O+NiSO_4.7H_2O$ [$ZnSO_4.7H_2O.NiSO_4.7H_2O$]

The obtain crystals is of the size 6mm x 5mm x 4mm

Figure. 1.1: Photograph of compound crystals of [$ZnSO_4.7H_2O.NiSO_4.7H_2O$]



Results and discussions:

i) Single crystal X-ray diffraction:

X-ray diffraction is an efficient analytical technique used to identify and characterize unknown crystalline materials. Powder X-ray diffraction provides less information than single crystal diffraction; however it is much simple and faster. The single crystallinity of the grown crystals were checked by XRD studies. The sample is purely mixed crystals of zinc sulphate and nickel sulphate and were characterized by single crystal XRD studies. A single crystal diffractometer (Bruker – Nonius MACH3 / CAD4) was used to obtain the cell parameters of the samples. The obtained cell parameter values for all the samples are given in the table 1.1.

It is noticed from the results that the grown [$ZnSO_4.7H_2O.NiSO_4.7H_2O$] crystals are isomorphous and they belong to orthorhombic structure. The obtained results are in good agreement with data reported in the literature *(H.H. Willard et al., 1986)*. It is useful for confirming the identity of a solid material and determining crystallinity and phase purity *(S. Radika et al., 2012)*.

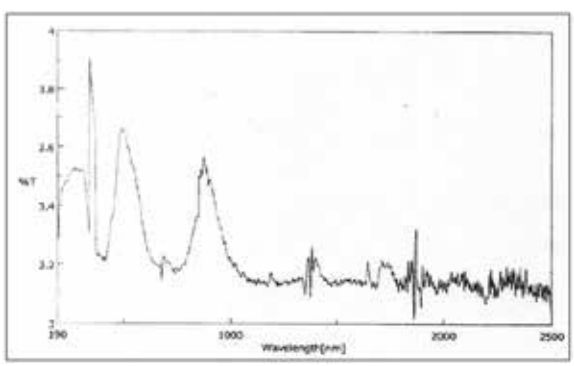
Table.1.1:The structural data for [$ZnSO_4.7H_2O.NiSO_4.7H_2O$] crystals

Lattice parameters	[$ZnSO_4.7H_2O.NiSO_4.7H_2O$] crystals
a	12.021 Å
b	11.884 Å
c	6.955 Å
α	90°
β	90°
γ	90°
structure	orthorhombic

ii) UV-VISIBLE Spectroscopic studies:

UV-VISIBLE spectra of the grown crystals were recorded using a UV-VIS spectrometer in the wavelength range 190-2500nm. The UV-VIS spectra of the grown crystals are shown in the figure.1.2.

Figure.1.2:UV-VISIBLE spectrum of [$ZnSO_4.7H_2O.NiSO_4.7H_2O$] crystals

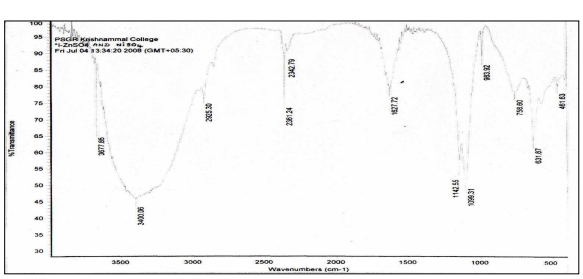


It is observed from the above figures that the samples show absorption at some wavelengths. This may be due to colour or presence of impurity in the crystals. For the grown crystals, the fundamental absorption band occurs below 200nm. This shows that band gap is wide and this proves that the grown crystals are insulating materials. UV transmittance window is quite small and the absence of absorption in the visible region is an advantage as it is the essential condition for materials having non linear properties *(B. Babu et al., 2014).*

iii) FTIR Studies:

The FTIR absorption spectrum in the infrared region 4000-400cm^{-1} *(D. Prabha and S. Palaniswamy., 2010)* was recorded on a spectrophotometer (shimadzu make) using the KBr pellet technique. The FTIR spectrum of the sample is presented in figure.1.3.

Figure.1.3: FTIR spectrum of [$ZnSO_4.7H_2O.NiSO_4.7H_2O$] crystals



The absorption band or peak of the FTIR spectra of sample has been given by the assignments using the data given in standard books on spectroscopy. The assignment for the absorption bands are given in the table 1.2.

Table.1.2: FTIR band and their assignments of [$ZnSO_4.7H_2O.NiSO_4.7H_2O$] crystals

Band or peak(cm^{-1})	Assignments
3400.06	OH stretching of water molecules
2361.24	Overtones of bands
1627.72	OH bending
1142.55	SO^{2-} stretching
1099.31	SO^{2-} stretching
758.6	Free H_2O vibration
631.87	Free H_2O vibration

The vibrational analysis of the crystal is done and the major functional groups and their assigned vibrations are tabulated. The various functional groups of the grown crystals were identified and the changes in the bands of the spectrum of the compound crystal are noticed.The absorption band or peak of the FTIR spectra of sample has been given by the assignments using the data given in standard books on spectroscopy.The assignments for the bands are given according the literature reported *(C.K. Mahadevan ., 2008).* The assignment for the absorption bands are given in the table 1.2.

vi) Dielectric constant and dielectric loss:

A good quality crystal was selected from the grown crystals and its faces were polished. Graphite paint was coated on the faces of the crystal to obtain a good ohmic contact. The capacitance with crystal and dielectric loss were measured using an LCR meter shown in figure 1.4 and a two-probe arrangement with fixed frequency 1kHz at temperature ranging from room temperature (30°C) to 70°C. Temperature of the crystal was measured using a sensitive digital thermometer. By varying the temperature, the capacitance for the respective temperature is noted. The dielectric constant (ε_r) was determined with the formula $\varepsilon_{r=}$

Where, c is the capacitance of condenser with crystal,

c_o is the capacitance of condenser without crystal.

Figure.1.4: Digital LCR- meter



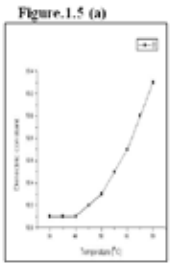
At the same time the equivalent series resistance is measured with the same LCR meter. The dielectric loss is determined by the formula $\tan\delta = (ESR/X_C)$

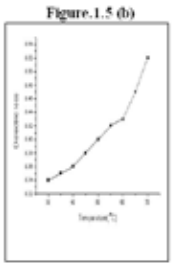
Where ESR is the equivalent series resistance, X_C is the capacitive reactance ($1/\omega_c$).

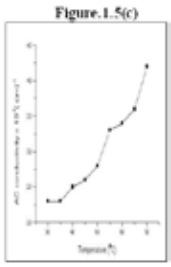
For a particular temperature the dielectric constant and dielectric loss were noted and a graph is plotted between dielectric constant, dielectric loss with temperature. Graph is shown by

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figure 1.5(a) and 1.5(b)


Figure.1.5 (a)


Figure.1.5 (b)


Figure.1.5(c)

V) AC conductivity:

For the calculated values of dielectric constant and dielectric loss, the AC conductivity is calculated for different temperature(30°C to 70°C).The AC conductivity is calculated by using the formula $\sigma_{ac} = 2\pi\upsilon\ \varepsilon_0\ \varepsilon_r \tan\delta$. A graph is plotted between temperature and AC conductivity shown by figure.1.5(c).

It is observed from the studies that AC conductivity of the grown crystals increases with temperature and it shows that the grown crystals are insulating in nature *(S. Boomadevi et al., 2004).* This increase in AC conductivity is due to transition of charge carriers from valence band to conduction band when temperature of crystals increases *(P. Selvarajan et al., 1994)*

Conclusion:

A good quality of [ZnSO4.7H2O.NiSO4.7H2O] crystals was grown by slow evaporation method. The grown compound crystals are observed to be transparent and the colour of the crystal is light green. Single crystal X-ray diffraction analysis confirms the crystalline nature of the grown crystal. The lattice parameter values show that the grown compound crystal belongs to orthorhombic crystal system. UV-VIS spectra of the grown crystals were recorded using a UV-VIS spectrometer in the wavelength range 190-2500nm. The fundamental absorption bands occur below 200nm. This shows that band gap for grown crystal is wide and this proves that the grown crystals are insulating materials. The FTIR spectrum confirms the presence of functional groups in the grown crystals. Using the values of dielectric parameters, it is noticed that AC conductivity of the grown crystals increases with temperature due to transition of charge carries from valence band to conduction band and the material is non conducting in nature.

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