Research Paper

Elemental, FTIR and Optical analysis of compound crystal of nickel sulphate heptahydrate and potassium dihydrogen citrate

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Abstract— The current paper discusses a study which is related to the growth and several characterizations of pure compound crystals of nickel sulphate heptahydrate (NSH) and potassium dihydrogen citrate (KDC) is reported. The crystals under study were grown at room temperature by the technique of slow solvent evaporation. After the duration of 6 weeks, good quality, square and rod-shaped crystals were found to have grown. Characterization of crystals was carried out by doing elemental, FTIR and optical analysis. The changes in the characteristics of pure NSH crystal and pure KDC crystal were compared with the compound crystal and discussed accordingly. The elemental analysis confirmed the presence of respective atoms of pure NSH and pure KDC in the grown product crystal. The FTIR spectroscopic analysis indicated the presence of functional groups of sulphate of NSH and characteristic vibrations of dihydrogen citrate in the compound crystal. The SHG analysis showed that pure as well as compound crystals of NSH and KDC did not possess the NLO property. The energy band gaps of pure and compound crystals of NSH and KDC were evaluated by using the KM function. The result showed an elevation in the value of the energy band gap in the value of pure KDC as it formed the compound with NSH. All the results are discussed.

Keywords— NSH, KDC, FTIR, elemental analysis, FTIR, SHG, energy band gap

1. Introduction

Nickel sulphate heptahydrate (NSH) belongs to a set of heptahydrate sulphate, the general formula of which is $RSO_4 \cdot 7H_2O$, where R = Ni, Mg, Zn etc [1]. Potassium dihydrogen citrate (KDC) is a salt of citric acid having formula ($C_6H_7KO_7$). It shows some important usefulness in the field of medical and pharmaceutical sciences, as discussed in our previous publication [2]. The study of the compound of KDC with NSH has technological importance because of the enhanced band gap energy of pure KDC. Generally, the formation of a compound crystal of organic substance with metal sulphate heptahydrate is known as a semi-organic compound crystal. In the literature, some reports are available on the growth and various characterizations of semi-organic compound crystals [3-5]. In most of the literature, neither the comparison of several properties of semi-organic compound crystal with the pure metal sulphate crystal and pure organic crystal is reported nor is the positive and/or negative effect of the formation of compound crystal on pure metal sulphate crystal and pure organic crystal is discussed except our report [6]. In this paper, we have discussed the effects of the compound crystal of NSH and KDC with the comparison of its properties with a pure crystal of NSH and KDC.

2. Experimental Technique

The crystals of pure as well as compound crystals of NSH and KDC are grown at room temperature. For that, a technique of slow solvent evaporation is employed. For the growth of pure crystals, the saturated solution of NSH and KDC is prepared by dissolving the required proportion of chemicals in double-distilled water. The chemicals were extra pure grade, purchased from Sigma Aldrich. The solutions are stirred well to get a homogeneous solution and then covered by filter paper having some pin holes for slow and controlled evaporation. For the growth of the compound crystal, both the saturated solutions are mixed at definite proportions, again stirred well to get the homogeneous solution and then covered with filter paper having some pin holes for the slow and controlled process of evaporation. After nearly 6 weeks, the greenish and rectangular crystal of pure NSH, colourless and transparent crystal of pure KDC and greenish and squared crystal of their compound are harvested. Figure 1 shows the photograph of the grown crystals.
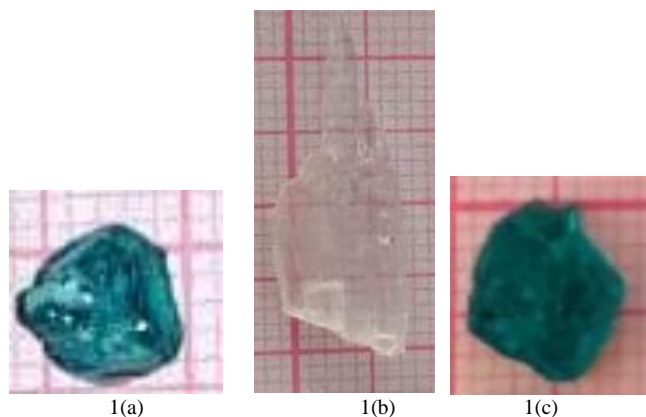
Figure 1: (a) Pure NSH crystal (b) Pure KDC crystal and (c) compound crystal of NSH and KDC

3. Result and Discussion

In order to find out the elemental composition of the grown compound crystal of NSH and KDC, the EDAX analysis is carried out. The elemental analysis is carried out by using Philips XI-30. The chemical formula of nickel sulphate heptahydrate (NSH) and potassium dihydrogen citrate (KDC) is $NiSO_4 \cdot 7H2O$ and $C_6H_7NKO_7$, respectively. Hence, in the EDAX spectrum of compound crystals of NSH and KDC, the presence of nickel (Ni) along with carbon (C) and oxygen (O) confirms that the formed crystal is a compound crystal of nickel sulphate heptahydrate (NSH) and potassium dihydrogen citrate (KDC). The analysis showed the presence of nickel (Ni) in the wt% of 18.74%, along with the presence of carbon (C) and oxygen (O) with their weight% of 0.76% and 45.71%, respectively. In the formation of a compound crystal, there is a strong possibility of ionic bonding between COO– ion and Ni^{2+} ions and as a byproduct, there may be either $KHSO_4$ or K_2SO_4 in the solution.

4. FTIR Spectroscopy Study

FTIR spectra of pure NSH crystal (sample – 1), pure KDC crystal (sample – 2) and compound crystal of NSH and KDC (sample – 3) are generated at room temperature, within the range of frequencies 400 to 4000 cm^{-1} and are presented in the following diagram 2.
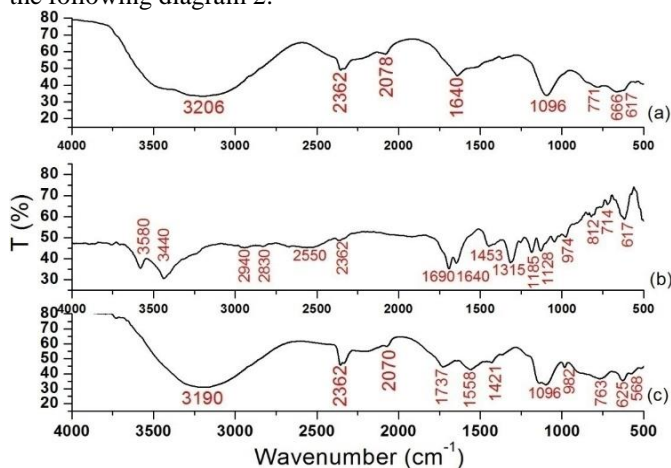

Figure 2: A spectra of FTIR of crystals of (a) sample – 1 (b) sample – 2 and (c) sample – 3

The discussion of each assignment of the bands shown in the figure for all three crystals is as under.

The crystal of pure NSH (sample – 1) shows a broad O-H stretching band in the range of 3000 to 3700 cm^{-1}, which can be assigned to the symmetric stretching mode of water molecules. The band observed at 1640 cm^{-1} can be attributed to HOH bending vibration [7], i.e., the bending modes of different water molecules of crystallization [8,9], indicating the hydrated nature of the NSH crystal. The absorptions within 2365 to 2080 cm^{-1} are attributed to O-H stretching vibrations of the cluster of water molecules of crystallization [8]. The characteristic absorptions of sulphate ion are observed within wavenumber 1130-1080 cm^{-1}, 680-610 cm^{-1} [10], 971-993 cm^{-1} and 445-490 cm^{-1} [11], which are known as triply degenerate asymmetric stretching vibration mode of sulphate, triply degenerate asymmetric bending vibration mode of sulphate, non-degenerate symmetric stretching vibration mode of sulphate and double degenerate vibration mode of sulphate, respectively [11]. In the present case, the broad and strong band observed at 1096 cm^{-1} and at 666 and 617 cm^{-1} could be assigned to triply degenerate asymmetric stretching vibration of sulphate and triply degenerate asymmetric bending vibration of sulphate, while the band that is observed at 771 cm^{-1} could be assigned to liberation mode of water molecules [12].

In the FTIR spectrum of pure KDC (sample 2), two absorption bands observed at wavenumbers 3580 and 3440 cm^{-1} correspond to the OH stretching vibrations of the hydroxy group, while the bending vibrations of this group manifest themselves as a single bend at wavenumber 1315 cm^{-1} [10]. The absorption bands observed at wavenumbers 2940 and 2830 cm^{-1} can be attributed to the asymmetrical and symmetrical stretching vibrations of the CH group, while the asymmetrical bending vibration of this group manifests itself as a single bend at wavenumber 1453 cm^{-1} [10]. The absorptions within 2365 to 2080 cm^{-1} correspond to O-H stretching vibrations of the cluster of water molecules of crystallization [8]. The absorption bands observed at wavenumbers 1690 and 1640 cm^{-1} can be ascribed to the stretching vibrations of the C = O functional group of COOH [13]. The absorptions observed at wavenumbers 1185, 1128, 974 and 812 cm^{-1} can broadly be ascribed to C – C and C – O stretching vibrations [14]. The band observed at wavenumber 812 cm^{-1} can be associated with out-of-plane CH bending vibration [14]. The bands observed at wavenumbers 714 and 617 cm^{-1} can be assigned to the out-of-plane bending vibrations of the OH group [10]. The vibrations at wavenumbers 812, 714 and 617 cm^{-1} can also be associated with CH rocking vibrations [14].

In the FTIR spectrum of compound crystal (sample 3), the band observed at 3190 cm^{-1} can be assigned to the OH stretching vibration [10]. In the compound crystal, O-H stretching vibrations of a cluster of water molecules of crystallization are observed at the same position as they are observed in pure NSH crystal. The characteristic vibration of the C = O group of COOH is shifted towards the higher wavenumber in the compound crystal and observed at

wavenumber 1737 cm[-1], while the vibration observed at wavenumber 1558 cm[-1] can be ascribed to the carboxylate [10]. The asymmetrical bending vibration of CH is observed to shift towards the lower wavenumber and appeared at 1421 cm[-1]. The C – C and C – O vibrations are shifted slightly towards the higher wavenumber and appeared at 982 cm[-1]. The absorption band observed below 600 cm[-1], i.e., at 568 cm[-1] can be ascribed to the metal-oxygen vibration Ni – O. The presence of these vibrations in the compound crystal confirms the association of KDC with NSH. In the compound crystal, the triply degenerate asymmetric stretching vibration mode of sulphate is observed at the same place, i.e., at 1096 cm[-1], as it is observed in the pure NSH crystal, while triply degenerate asymmetric bending vibration mode of sulphate is shifted towards lower wavenumber and observed at wavenumber 625 cm[-1]. The liberation mode of water molecules is shifted slightly towards the lower wavenumber and appeared at 763 cm[-1]. This also indicates the association of NSH with KDC.

5. Optical Analysis

The optical properties of pure and compound crystals of NSH and KDC have been estimated from the measurement of transmittance spectra, which are recorded as a function of wavelength ranging from the ultraviolet region (200 to 400 nm) to the visible region (400 to 750 nm) of the electromagnetic spectrum. From the study of the optical transmittance window of the grown crystals, the possible optical applications of the crystals can be suggested.

The optical transmission spectra of pure NSH crystal, pure KDC crystal and their compound crystal are shown in Figure 3.
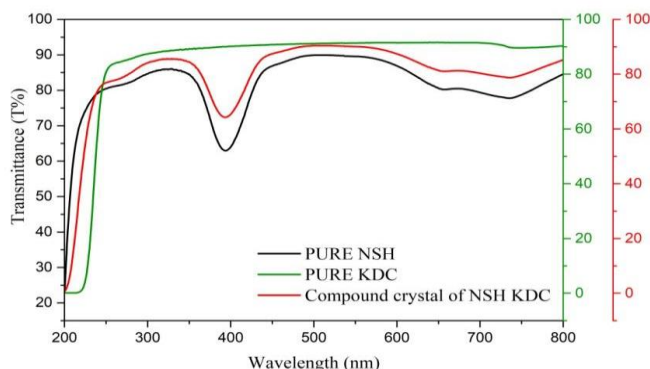
Figure 3: Transmission spectra

The separate analysis of pure NSH crystal shows a cut-off wavelength of 200 nm, which is expected for $\pi \rightarrow \pi*$ transition. Then transmittance increases and attains a value of 81.89% at wavelength 308 nm. It remains constant up to 340 nm wavelength and then starts to reduce and attains a minimum value of 63.26% at wavelength 395 nm. Then it starts to increase and attains 90% at wavelength 487 nm. It retains the same value up to 573 nm, then decreases up to 80% at wavelength 650 nm and again retains the same value up to wavelength 685 nm. It reduces, again, up to 77.6% at wavelength 735 nm and starts to raise up to 84.6% at the upper limit of wavelength, i.e., 800 nm. From the above

analysis, it can be said that the pure NSH crystal shows a reduction in the transmittance % at different regions of wavelength. The discontinuity observed in the transmittance spectrum shows its application as a band-pass filter. It can be observed from the figure that NSH possesses a bandwidth in the range of 220 nm to 395 nm, the flat portion of which is centred at 308 to 340 nm, thereby making it a good UV filter. As discussed above, an intermediate decrease in transmittance is observed, which indicates intrinsic loss mechanism is taking place by the interaction of electromagnetic radiation of UV and visible region. Within the visible region, pure NSH crystal shows a maximum value of transmittance of 90% within the wavelength range 487 to 573 nm, 80% within the wavelength range 650 to 685 nm and a minimum of 77.6% at wavelength 735 nm.

The separate analysis of pure KDC crystal shows a cut-off wavelength of 227 nm and then an increase in transmittance from 82.39 to 91.52% within the wavelength range 250 to 715 nm. Within the visible range of 400 to 750 nm, the pure KDC crystal shows a % transmittance of 91.52%. The separate analysis of the compound crystal shows a cut-off wavelength of 200 nm. The compound crystal shows similar transmittance behaviour as that of pure NSH crystal. Within the wavelength range of 308 to 340 nm, the maximum value of transmittance of pure NSH crystal increases from 81.89% to 85.60%. It shows the effect of KDC on the transmittance property of pure NSH. The minimum value of transmittance is observed at the same wavelength, i.e., at 395 nm in the case of both pure NSH and compound crystal.

The compound crystal shows a shifting of the cut-off wavelength of pure KDC from 227 to 222 nm, i.e., towards the lower wavelength side, which can be attributed to hypsochromic shift. It means, when the compound crystal is formed, the energy required for the electronic transition increases compared to pure KDC crystal.

The absorption coefficient is calculated by using formula

$$\alpha = \frac{2.303 \, A}{d} \qquad\qquad -- (1)$$

where, A and d indicates absorbance and thickness of the sample. The absorption coefficient as a function wavelength for pure and compound crystal of NSH and KDC is presented in the figure 4.
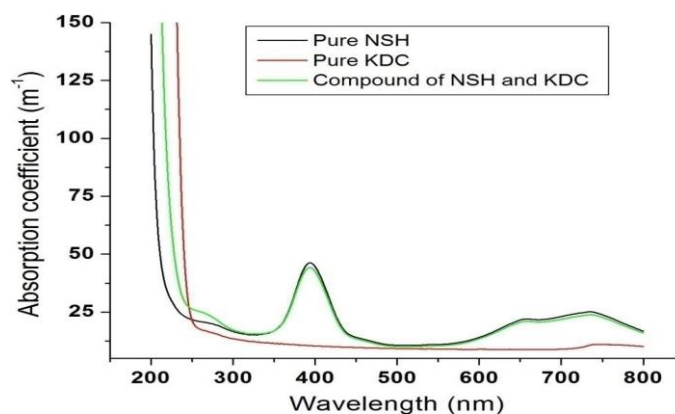
Figure 4: Absorption coefficient as a function of wavelength

From the figure, it is evident that absorption coefficient shows reverse nature of optical transmittance. Further, the shifting of the absorption edge is observed from the figure. Pure NSH shows absorption edge at 216 nm, pure KDC shows the same at 244 nm, while compound crystal shows an absorption edge at 235 nm. It means compound crystal increases the absorption edge of pure NSH from 216 nm to 235 nm, while decreases the same for pure KDC from 244 nm to 235 nm. The shifting in the absorption edge results in the alternation in the band gap.

The optical band gap is measured for all three crystals by using the well-recognized Kubelka-Munk (KM) theory. The KM function is given by [15]:

$$F(R) = \frac{(1-R)^2}{2R} \qquad\qquad -- (2)$$

Where, R is the value of reflectance, which is used in KM theory to estimate band gap value. A relation between KM function F(R) and energy band gap (E_g) is given by the equation [16]

$$F(R) = \frac{(h\nu - E_g)^n}{h\nu} \qquad\qquad -- (3)$$

As the samples possess direct optical transition, to estimate the band gap, the variation of $[F(R) h\nu]^2$ with respect to the photon energy ($h\nu$) is drawn, as shown in figure 5.
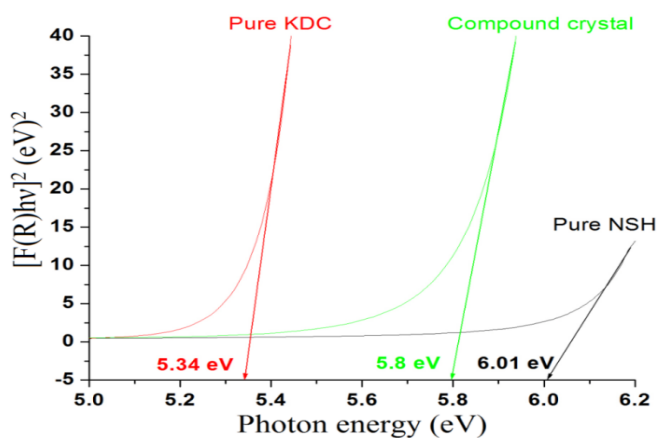

Figure 5: hv dependence of $[F(R)h\nu]^2$

The linear segment of the $[F(R)h\nu]^2$ versus $h\nu$ is extrapolated. It is taken to determine the value of E_g, which is shown in the figure for the pure crystal of NSH, KDC and their compound crystal. The energy band gap values obtained from both methods show good similarity. Pure NSH crystal possesses the value of a band gap that is the highest of all, while pure KDC crystal possesses the value of a band gap that is the lowest and compound crystal possesses the intermediate value of a band gap. So, compound crystal raises the band gap value of pure KDC from 5.3 to 5.8 eV. The increased value of the band gap of pure KDC in the compound crystal may be attributed to the stability and improvement of the KDC phase in the compound crystal. The decrease in the band gap value of pure NSH from 6 eV to 5.8 eV may be the

result of the charge compensation effect as nickel possesses +2 valency while potassium possesses +1 valency.

Figure 6 depicts the variation of refractive index (n) with the changes in the values of energy of the incident photons for all the crystals.
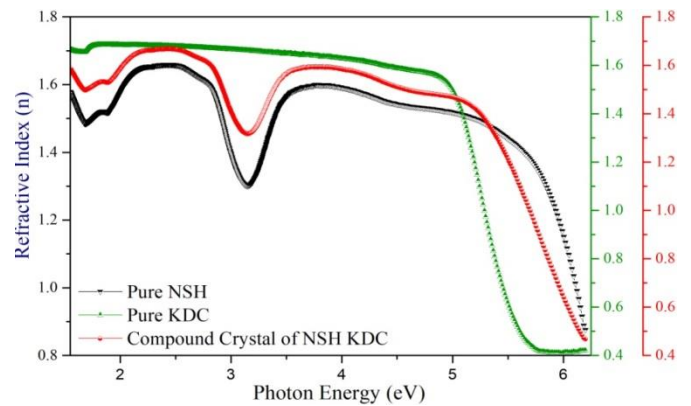

Figure 6: hv dependence of the refractive index

The refractive index is calculated by using the standard formula given in the literature [17] by incorporating the reflectance (R) data in the calculations. The refractive index decreases with the increase of photon energy for all the crystals, which reveals the regular dispersion behavior. Pure KDC crystal shows a variation in refractive index in the range of 1.556 to 1.686 corresponding to wavelength range 250 to 700 nm. Pure NSH crystal exhibits the refractive index value 1.521 to 1.651 within wavelength region 398.5 to 557 nm with the lowest value of 1.295 at 394.5 nm. Compound crystal shows an almost similar trend of variation in refractive index as that of pure NSH, except the refractive index value of 1.473 at the same wavelength, i.e., at 394.5 nm wavelength. From the above discussion, it can be said that when KDC forms a compound with NSH, it shows the behavior as that of pure NSH and if the compound of NSH with KDC is considered, then pure NSH retains its original behavior but the overall refractive index value increases. Figure 7 shows the variation of extinction coefficient (K) with respect to photon energy for all the crystals. K is evaluated by using the standard mathematical equation given in the literature [17].
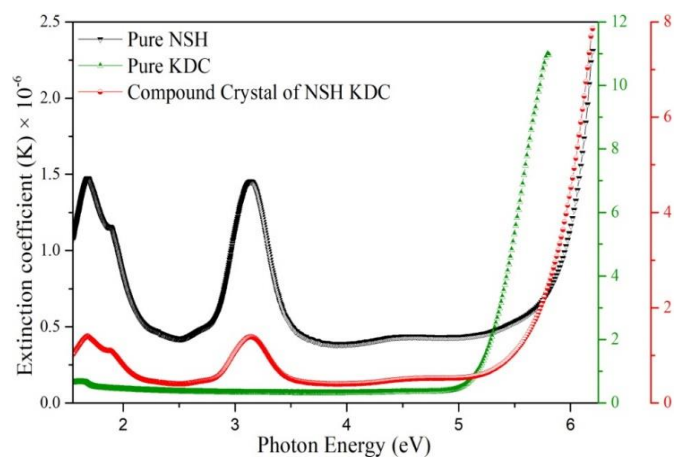

Figure 7: hv dependence of extinction coefficient

From the plot of figure 7, it is detected that pure NSH crystal retains a constant K value within the wavelength range 237 to 330 nm. Then it increases and shows a peak at wavelength 394 nm within wavelength range 330 to 440 nm. This indicates the absorption of light within this range of wavelength. For pure KDC crystal, it remains constant within the wavelength range 251 to 624.5 nm, while, compound crystal shows almost similar trend of variation as that of pure NSH crystal. The behaviour of K shown in the plot of figure 7 can be associated with the absorption of light, which is clear from figure 8, which is a plot of absorbance versus wavelength of incident photons.
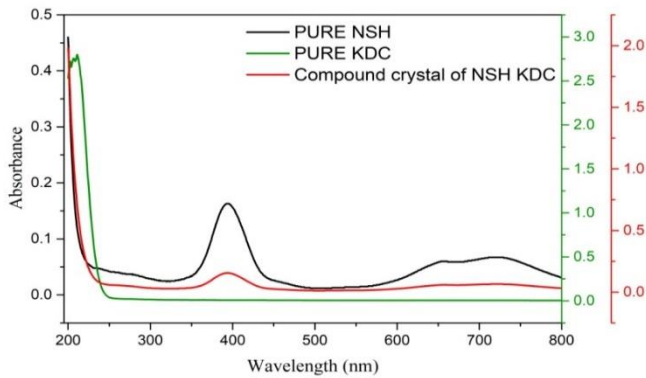

Figure 8: wavelength dependence of absorbance

Figure 8 represents the variation of optical conductivity (σ_{opt}) with respect to photon energy for all the crystals.
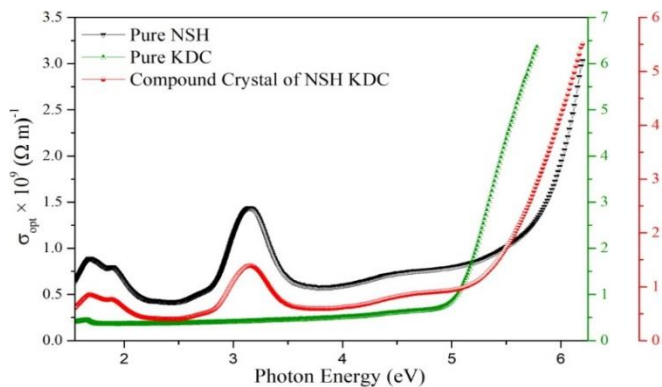

Figure 9: hν dependence of Optical conductivity

The optical conductivity is calculated by using the standard formula given in the literature [17]. It is observed from the figure that the optical conductivity value increases with an increase in photon energy for all the crystals and possesses higher order of magnitude [10^9 $(\Omega m)^{-1}$], thereby confirmative of the high photo-tuneable behaviour of all three materials [18]. It is observed from the figure that to obtain the same value of optical conductivity, for example, 1.88×10^9 $(\Omega m)^{-1}$, the highest value of photon energy is required for pure NSH crystal (6.18 eV), while the lowest value of photon energy is required for pure KDC crystal (5.39 eV). When the compound is formed, to obtain the same value of optical conductivity, the photon energy reduces compared to pure NSH crystal (from 6.18 eV to 5.83 eV) and slightly increases compared to pure KDC crystal (from 5.39 eV to 5.83 eV).

Though all three crystals possess a high magnitude of optical conductivity, the compound crystal raises the optical conductivity of pure NSH. These results confirm the presence of a high photo response nature of the crystals [18,19].

Absorption as a function of energy can be expressed by the mathematical relation, known as Urbach relation

$$\alpha(h\nu) = \alpha_0 \exp\left(\frac{h\nu}{E_u}\right) \qquad\qquad -- (4)$$

Where α_0 is constant, h is Planck constant, ν is the frequency of radiation and E_u is Urbach energy or Urbach edge. Its value evaluates the energetic disorder in the band edges of the material and sharpness of the onset of absorption near the band edge, i.e., the broadness of the density of states. Its lower value represents a sharper or steep onset of absorption. Figure 10 shows the plots drawn between ln(α) and photon energy for all three samples.
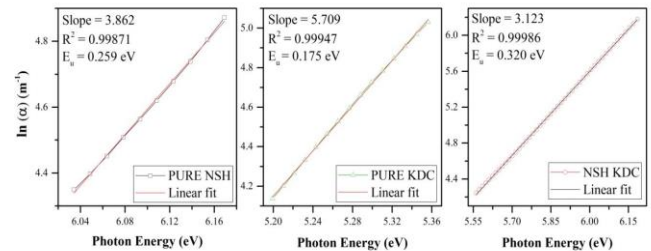

Figure 10: Plot of ln(α) versus photon energy

Urbach energy is evaluated from the reciprocal of the slope of linear portion, as shown in the figure for all the samples. Overall, all the three crystals possess low value of Urbach energy, which indicates minimum structural defect in the grown crystals. As the compound crystal is formed, increase in Urbach energy compared to pure NSH and pure KDC crystal is observed, which indicates that the sharpness of onset of absorption decreases and broadness of density of states increases, which results into increase in structural defect in the lattice of the compound crystal.

6. SHG Analysis

For the confirmation of NLO property, pure NSH crystal, pure KDC crystal and its compound crystal are subjected to Kurtz powder test using Q switched High Energy Nd: YAG Laser with modulated radiation of wavelength 1064 nm, having repetition rate 10 Hz and pulse width 6 ns on the powder of crystals through a visible blocking filter. The input laser beam is directed at the crystal powder to get maximum powder SHG. The NLO testing reports are shown in table 1.

Table 1: SHG efficiency

Sample Specifications	Output Energy (mJ)	Input Energy (mJ)	SHG Efficiency
Pure KDP	7.5	0.70	1
NSH Crystal	NIL	0.70	NIL
KDC Crystal	NIL	0.70	NIL
Compound Crystal	NIL	0.70	NIL

From the table, it is observed that the pure crystals of NSH and KDC do not possess NLO properties and hence, their compound crystal also do not show NLO properties. The presence of a strong electron donor or acceptor group can enhance the NLO property of the precursor organic compound. In the present case, the compound crystal of NSH and KDC does not show NLO properties. It means, in the compound crystal, the sulphate group of NSH (which is a strong electron acceptor group) might not be associated with the KDC and hence do not show NLO property.

7. Conclusion

The technique of slow solvent evaporation was employed to grow the crystals of pure NSH, pure KDC and its compound. The elemental analysis showed the presence of Ni along with C and O in the compound crystal. FTIR analysis showed the presence of characteristic functional groups sulphate of NSH and characteristic vibrations of dihydrogen citrate in the compound crystal. The SHG analysis showed the absence of NLO property in pure as well as in the compound crystal of NSH and KDC. The optical analysis showed an increase in the band gap value of pure KDC after the formation of its compound with NSH. Moreover, refractive index, extinction coefficient, absorbance and optical conductivity were calculated to examine the optical properties of pure NSH, pure KDC and their compound crystal.

Data Availability

The raw data is required for the ongoing study; hence it will not be possible to share.

Conflict of Interest

The authors declare that there are no competing interests.

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Authors' Contributions

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