ISSN PRINT 2319 1775 Online 2320 7876

Research paper UGC CARE Listed (Group -I) Journal Volume 11,S Iss 3, Dec 2022

Habit Modification Of Thiourea In The Presence Of Sodium Chloride.

Dr.A.Prabumarachen

Assistant professor,Department of Physics, Vivekananda college, Agasteeswaram-629701.Affiliated to Manonmaniam Sundaranar University,Tirunelveli,abishekapatti, Tamilnadu, India.

Dr.T.S.Jayanthi

Associate professor,Department of Physics, Vivekananda college, Agasteeswaram-629701.Affiliated to Manonmaniam Sundaranar University,Tirunelveli,Abishekapatti, Tamilnadu, India.

ABSTRACT

The pure and sodium chloride doped thiourea crystals were grown at low temperature by slow evaporation technique. The effect of the dopand concentration on the morphology and hence the structure was studied. The in corporation of sodium chloride in the mother solution was found to change the growth habits. The habit of the crystals changed from hexagonal to rhombohedral.

Keywords : sodium, crystals, electronic, solution, industry, demand

Introduction :

In many industrial crystallization the impurity plays an vital role on the crystallization of single crystals from aquas solutions [1-3] . During crystallization, the dimension (size) and shape of the crystals play an important factor, since the. undesirable habits such as needle like or plate like cause the problems of separating, washing and drying [4] . Single crystals of Thiourea are being extensively used and have demand in the electronic industry as polarisation filter, electro-optic and electron-acoustic devices. Thiourea crystals also exhibit pyro electric effect which is utilized in infrared, Scanning electron microscopy, detection and infrared imaging [5]

Thiourea belongs to orthorhomhic crystal system with lattice primitive space group pnma. The unit cell parameters are a = 7. 657A^0, b = 8.588 A^0 and c= 5.485 A^0 [6]

Thiourea is soluble water and its solubility at 13^{0}C is 9.8 Parts by weight of water [7]

Its molecular weight and density are 76.12 and 1.405 gm/CC respectively.

Sodium Chloride is an inorganic material.

In the present investigation we report the effect of sodium Chloride on the crystallization of thiourea.

ISSN PRINT 2319 1775 Online 2320 7876

Research paper UGC CARE Listed (Group -I) Journal Volume 11,S Iss 3, Dec 2022

1. Experimental Procedure.

Crystal growth.

Analar grade thiourea, sodium chloride and deionsed water was used in the present crystallization process. The solubility of the thiourea in water was determined at temperatures varying from 30^0C to 50^0C in steps of 5^0C.The solution was prepared using recrystallised salts of thiourea and stirred well with a maqnetic stirrer to attain saturation. The solution was filtered and distributed equally (100ml) in five petridishes. Sodium chloride of .25, .5, .75, 1 and Mol % were added in four crystallizers and the remaining one Petri dish containing the mother solution was used as standard. The solution was stirred well till the complete dissolution of sodium chloride. The P^H of the solution was maintained at a constant value. The crystallizers were closed with perforated polythene papers and kept at a constant temperature 30^0C. The crystallization was initiated by slow
evaporation technique.

Pure and sodium Chloride doped thiourea crystals were harvested after a period of three weeks.

2.2 Characterization:

Powder X-ray diffraction (XRD) has been recorded using a Rich seifert diffractometer with CuKα radiation (λ= 1.5418A^0). The crystals were characterized by Fourier Transform infrared spectroscopy (FTIR) using KBr pellet technique. The estimation of sodium chloride and other elements present was estimated using ICP analysis. The surface morphology was recorded.

2. Result and discussion.

3.1 The solubility of thiourea at different temperature shows that the solubility in creased almost linearly with the increase of temperature having a positive coefficient of solubility (Fig. 1)

ISSN PRINT 2319 1775 Online 2320 7876

Research paper UGC CARE Listed (Group -I) Journal Volume 11,S Iss 3, Dec 2022

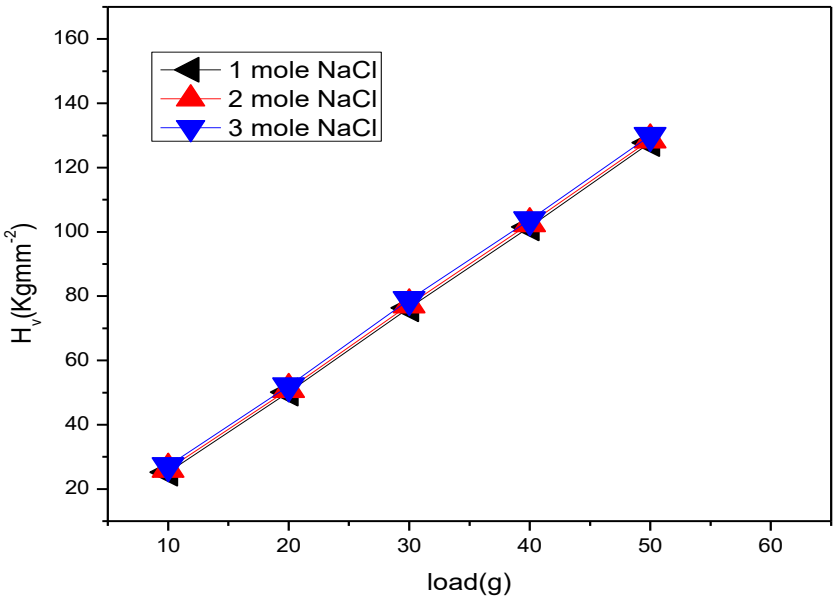


Figure 1: Variation of Vicker's hardness number with load for NaCl doped thiourea crystals

3.2 The effect of dopants on the crystal Habit.

It was observed that the growth rate and hence the Habit of the crystal mainly depend on the P^H and the dopnt concentration present in the mother solution. In the present investigation the P^H of the mother solution was found to be constant[5,6] .It was observed that increase in sodium chloride concentration changes the crystal Habit as shown in figure

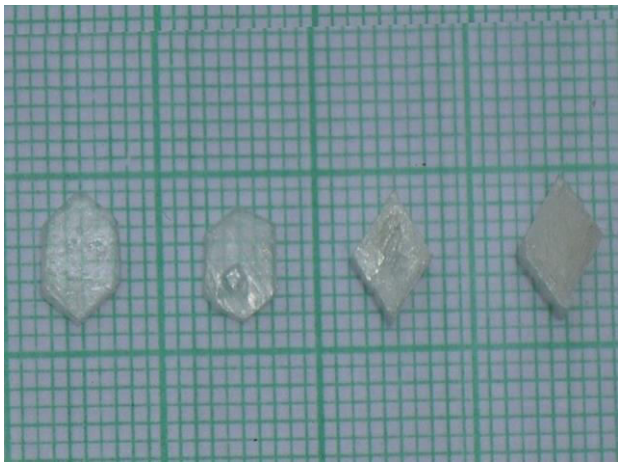


Fig : a. Pure thiourea single crystal and b NaCl doped thiourea single crystals.

The morphology of the Crystal changes from the morphology of the growing crystal is determined by the growth rates in different crystal graphic directions If the growth rate is inform, The distance from the centre of the crystal to the different.

ISSN PRINT 2319 1775 Online 2320 7876

Research paper UGC CARE Listed (Group -I) Journal Volume 11,S Iss 3, Dec 2022

Xray diffraction data for the Pure & Nacl doped thiourea single crystals.

	a A^0	b A^0	c A^0	Cell Volume A^0
JCPDS Data	7.657	8588	5.485	360.68
Pure Thiourea	7.665	8.588	5.498	362.02
NaCl Doped	7.658	8.600	5.515	363.27

Crystal faces are proportional the relative growth rate R of the Crystal faces [8] .

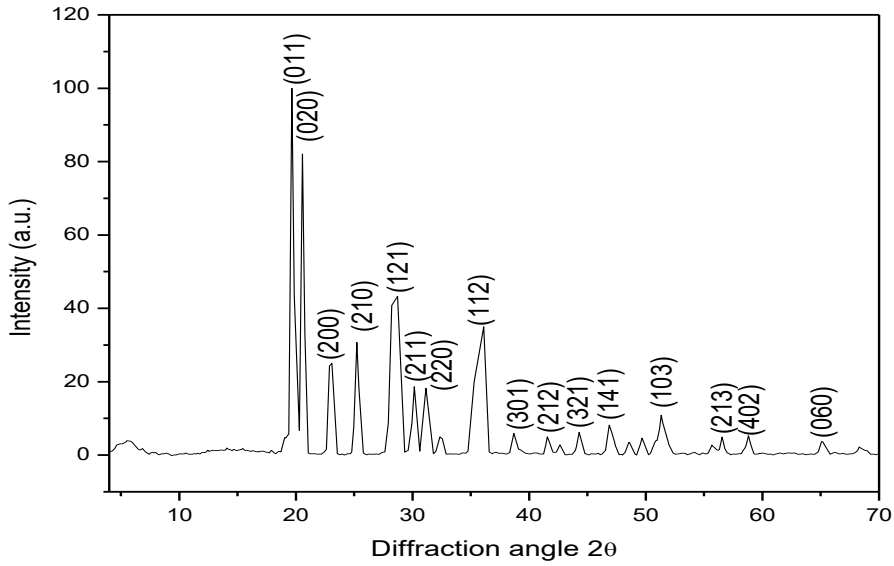
The relative growth rate of the face in a crystal Rα 1/d_{hkl}, Where d_{hkl} is the inter – planar distance. Under certain suitable conditions fast growing as well as slow growing faces disappear. If the growth rate Uniform in a dinective Perpendicular to a certain face, then that face grows larger in size [9, 10] .

In the pure thiourea single crystal the growth rate R along [0l0] and [110] were equal as indicated by the vectors Fig. 2a. The Presence of NaCl dopant in the mother solution induced varying growth rates in different faces as shown in Fig.2C. Sodium Chloride gets adsorbed in the crystal lattice, leading to the variation in the growth rates. The growth rates along [110] remaining constant where as an increase in growth rate along [010] direction was observed.

Hence the fast growing plane [010] disappears leading to tetragonal morphology of the sodium chloride doped thiourea single crystals. Habit modification was observed all the dopant concentration tried in these sets of experiments.

3.3. X-ray powder diffraction analysis

The X- ray power diffraction patterns for the pure thiourea and sodium chloride doped thiourea single crystals differed in their relative intensity (Fig 3). The lattice parameters were calculated by fitting the XRD data with “Least square method” using “CELN” program the calculated lattice parameters for the pure and doped thiourea crystals were presented in table 2. The Lattice parameters of the pure thiourea crystals well agreed with the JCPDS data. The calculated lattice parameters for the pure and doped thiourea crystals were presented in Table : 2 of thiourea.



ISSN PRINT 2319 1775 Online 2320 7876

Research paper UGC CARE Listed (Group -I) Journal Volume 11,S Iss 3, Dec 2022

Fig.4.5 X- ray powder diffraction patterns for the sodium chloride doped thiourea

Table 4.8 : X-ray diffraction data for NaCl doped thiourea

	a Å	b Å	c Å	Cell Volume Å
JCPDS Data	7.657	8.588	5.485	360.68
NaCl Doped thiourea	7.658	8.600	5.515	363.27

THE ICP-AES ANALYSIS FOR SODIUM CHOLORIDE DOPED THIOUREA SINGLE CRYSTALS

The sodium choloride doped thiourea given two samples were dissolved in HNO_3 and make up into 100ml using HPLC grade water and analyzed.The elements measured (ppm level)were presented in table 4.2.

Table -4.2 : Elements measured (ppm level) in NaCl doped thiourea

Dopant(NaCl) concentration	**Cu3275**	**Fe2599**	**k-7664**	**Mg2852**	**Na5889**
1Mol.%,	BDL	12O.22	99.86	18.88	7886.0
2Mol.%	BDL	122.05	102.18	20.31	7922.4
3Mol.%	BDL	125.15	103.39	21.41	7965.4

In the sodium choloride doped thiourea, the incorporation of sodium was detected from this analysis.It was also observed that the sodium element present increased with increase in the dopant concentration .Copper element was found to be below the detectable limit.

FTIR Studies for sodium chloride doped thiourea single crystals

The Fourier Transform infrared (FT-IR) spectra of the grown c The sodium chloride doped thiourea single crystals rystals were recorded using JASCO FTIR 460 spectrometer by KBr pellet technique in the range 450-2500 $cm^{-1}$and shown in the figure 4.2.

ISSN PRINT 2319 1775 Online 2320 7876

Research paper UGC CARE Listed (Group -I) Journal Volume 11,S Iss 3, Dec 2022

The observed bands were assigned and presented in Table4. 5. The change in vibrational frequencies for the NaCl doped thiourea may be due to the presence of the dopant.

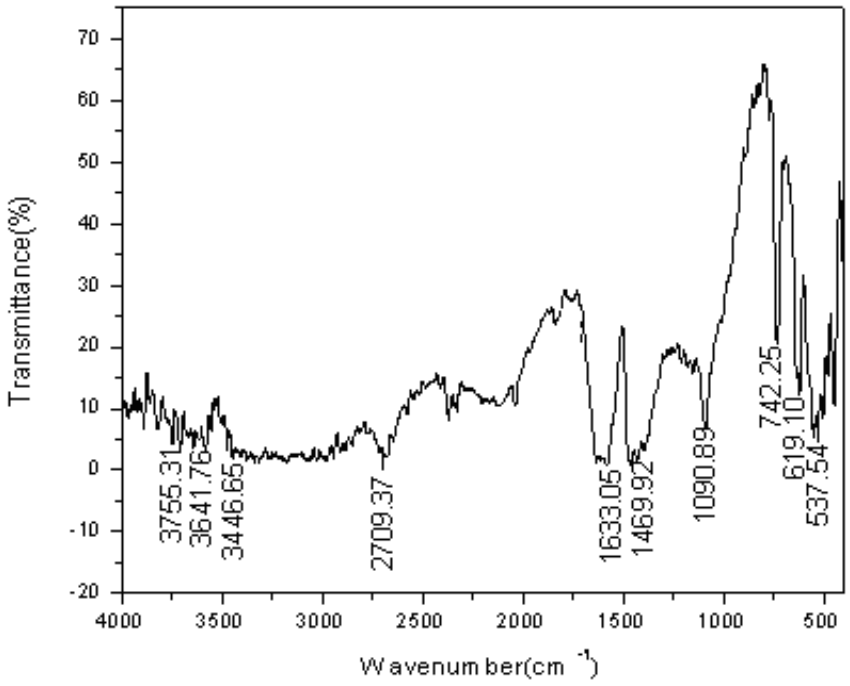


Figure 4.2 : FTIR spectra for NaCl doped thiourea

Table 4.5. Vibrational assigns for NaCl doped thiourea

Wave number (cm^{-1}) Thiourea	**Wave number (cm^{-1}) NaCl doped Thiourea**	**Assignments**
494	537.54	(N-C-N) Asymmetric stretch
740	619.10	(C=S) stretching
1089	742.25	(C-N) stretching
1417	1090.89	(C=S) Asymmetric stretching
1470	1469.92	(N-C-N) Symmetric stretching
1627	1633.05	(NH_2) scissors

ISSN PRINT 2319 1775 Online 2320 7876

Research paper UGC CARE Listed (Group -I) Journal Volume 11,S Iss 3, Dec 2022

CONCLUSION

Sodium chloride doped with (1mole %,2mole %, 3 mol%) thiourea single crystals could be grown by slow evaporation technique at average room temperature. The crystals are found to be highly transparent and well faceted. The lattice parameters were calculated and found to be orthorhombic. The various functional groups were assigned. The morphology of the sodium chloride doped thiourea crystals changed from hexagonal to tetragonal. The influence of foreign molecules present in growth media in changing the growth habit of crystals has been recognised. The presence of NaCl dopant in the mother solution induced varying growth rates in different faces. Sodium Chloride gets adsorbed in the crystal lattice, leading to the variation in the growth rates. Hence the hexagonal morphology disappears. The habit of the pure thiourea single crystal changes from hexagonal to tetragonal when doped with sodium choloride. The SEM Micro photograph shows that the surface is smooth and plane reveals the perfection of the crystal.

[1].J.W. Mullin, Crystallization, Butterworths, London,1972.
[2].H.Fischer, Elektrolytische Abscheidung and Electrokristallisation on Metallen, Springer, Berlin,1954.
[3].H. Fleisch, J. Crystal Growth 53 (1981) 120.
[4]. H.Cano, N. Gabas, J.P.Canselier, J.Crystal Growth 224(2001) 335.
[5].K. Sangwal, "Micro Morphology as grown surfaces of crystals"Proc. Crystal Growth 19,189,1989.
[6].Jcpds Data PPF number 832252 Ret mullen, D, Heiger G, Trentmann W.Z Krisallogr, Kristallgeom Kristallphy, Kristallchom. 148, 95(1979)
[7].John AD, "Langes' Handbook of chemistry" 12th Edn, McGraw Hill, Newyork 1979.
[8].B. Bennema, J.P. Van der Eerden, in: I. Sunagawa (Ed).Morphlolgy of crystals, Part A, Terra Science, Tokyo, 1987, P.1
[9].P. Hartman, In. I Sunagawa (Ed.) Morphology of crystals, Terra, Tokyo, 1987, P. 269.
[10]. X. Y. Liv, P. Bennema, Phys. Rev. B53 (1996) 2314

ISSN PRINT 2319 1775 Online 2320 7876

Research paper UGC CARE Listed (Group -I) Journal Volume 11,S Iss 3, Dec 2022

ISSN PRINT 2319 1775 Online 2320 7876

Research paper UGC CARE Listed (Group -I) Journal Volume 11,S Iss 3, Dec 2022