



IJREB

ISSN 2321-743X

International Journal of Research in
Engineering and Bioscience

Volume 7 Issue 1 (38-45)

Journal home page: www.ijreb.org

**A STUDY ON THE PREPARATION, PROPERTIES AND APPLICATIONS OF
PURE AND DOPED NINHYDRIN CRYSTALS BY SLOW EVAPORATION
TECHNIQUE**

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ABSTRACT

Research papers related to the preparation, properties and applications of pure and doped Ninhydrin crystals by slow evaporation technique were reviewed and the findings were as follows. Pure and doped Ninhydrin crystals belongs to monoclinic system. Pure and doped Ninhydrin crystals belongs to soft material category, except copper oxide doped Ninhydrin crystal. Characterization techniques like x-ray diffraction, FTIR, UV Spectroscopic analysis, Thermal (TGA/DTA) analysis, microhardness studies, dielectric studies and SHG efficiency were carried out for the grown crystals by the authors.

Keywords: Ninhydrin, slow evaporation technique, microhardness studies, FTIR

INTRODUCTION

Ninhydrin is an organic material, which can be dissolved in many solvents like water and ethanol (A.Ponchithra and K. Balasubramanian, 2020). This material has high melting point. Uma Devi et.al, (2008) discovered that this compound has aromatic ring with higher optical coefficient and large band gap. Ninhydrin has broad application in forensic science, food industry, agriculture and soil biology. Ninhydrin plays a vital role in synthesizing organic heterocyclic compounds. The compound Ninhydrin has been used in fabrication devices and photovoltaic devices.

Ninhydrin is a white solid and has various properties and it has been used in various fields. The IUPAC name of Ninhydrin is 2, 2-Dihydroxyindane-1, 3-dione. Its molar mass is about 178.14g/mole and has a density of 0.862g/cm³. Ninhydrin is widely used as a reagent for the detection of latent fingerprints. It is used to titrate arginine residues of proteins.

MATERIALS AND METHOD

T.Uma Devi et.al (2008) followed solvent evaporation method to grow single crystals of Ninhydrin. They added required amount of Ninhydrin in 100ml of distilled water and the solution was stirred continuously. The prepared solution was kept undisturbed for solvent evaporation. After a growth period of ten days, well developed single crystal with dimension 11mm × 10mm × 7mm was

harvested.

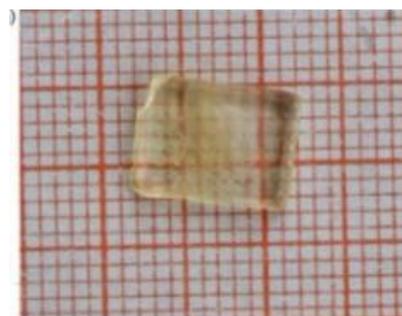


Fig 1. photography of pure Ninhydrin crystal grown by T.Uma Devi et.al (2008)

Sirine Hawech et.al (2022) followed solvent evaporation technique to grow potassium nitrate Ninhydrin crystal. They mixed Ninhydrin and potassium nitrate in the ratio of 2:1 and the solution was stirred well using magnetic stirrer for 30 mins. The solution was kept undisturbed for solvent evaporation. Good quality transparent potassium nitrate Ninhydrin crystals were grown within 30 days.

A.Ponchithra and K.Balasubramanian (2020) used solvent evaporation method to grow Magnesium doped Ninhydrin single crystals. In 250- 300ml double distilled water they dissolved 5 gram of Ninhydrin. After forming a clear solution 0.4 mole percentage of Magnesium nitrate was added and stirred well using magnetic stirrer for about 4-5 hours. The solution was kept undisturbed for solvent evaporation. Good quality single crystals were grown within 36 to 60 days.



Fig 2. photography of magnesium doped Ninhydrin crystal grown by A.Ponchithra and K.Balasubramanian

Anand Ponchithra et.al, (2020) used solvent evaporation method to grow Nickel doped Ninhydrin single crystals. They dissolved 5 gram of Ninhydrin in double distilled water. After forming a clear solution 1 mole percentage of Nickel nitrate was added and stirred well using magnetic stirrer for about 4 hours. The solution was kept undisturbed for solvent evaporation. Good quality nickel doped Ninhydrin single crystals were grown within 36 to 60 days.



Figure 3. Photography of nickel doped Ninhydrin single crystals grown by Anand Ponchithra et.al, (2020)

K.Lilly Mary Eucharista et.al, (2018) followed solvent evaporation method to grow DL Malic acid Ninhydrin single crystals. They dissolved 5 gram of Ninhydrin in 100 ml of deionized water. After forming a clear solution 1:1 ratio of DL

Malic acid was added and stirred well using a magnetic stirrer for about two hours. The solution was kept undisturbed for solvent evaporation. Good quality Malic acid doped Ninhydrin crystals were collected after a period of 30 days.

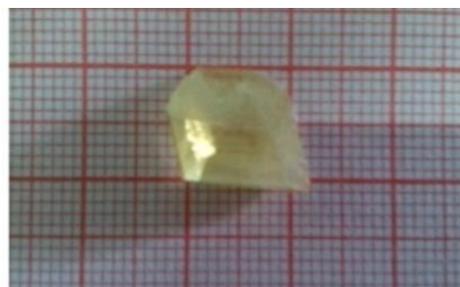


Figure 4. photography of Malic acid doped Ninhydrin crystal grown by K.Lilly Mary Eucharista et.al, (2018)

A.Ponchithra and K.Balasubramanian, (2017) followed solvent evaporation method to grow Zinc nitrate doped Ninhydrin single crystals. They dissolved Ninhydrin in 100 ml of distilled water. After forming a clear solution one mole percentage of Zinc nitrate was added and stirred well using magnetic stirrer. The prepared solution was kept undisturbed for solvent evaporation. Good quality Zinc nitrate doped Ninhydrin crystals were collected after a period of 38 days.



Figure 5. Photography of Zinc nitrate doped Ninhydrin crystals grown by A.Ponchithra and Balasubramanian, (2017)

R.S.Sreenivasan et.al, (2012) used solvent evaporation method to grow metal doped Ninhydrin single crystals. In 100ml saturated solution of Ninhydrin they added 0.2 mole percentage of copper chloride and stirred well using magnetic stirrer for about 2 hours. The solution was kept undisturbed for solvent evaporation. After a growth period of ten days well developed Copper doped Ninhydrin single crystals with dimension 5.07mm×3.41mm×2.69mm was collected.

T.Prasanyaa et.al, (2012) followed solvent evaporation method to grow copper nitrate doped Ninhydrin single crystals. They dissolved Ninhydrin in 100 ml of distilled water. After forming a clear solution one mole percentage of Copper oxide was added and stirred well using magnetic stirrer. The solution was kept undisturbed for solvent evaporation. Good quality single crystals were developed after a period of 15 days.



Fig 6. Photography of Copper nitrate doped Ninhydrin single crystals grown by T.Prasanyaa et.al, (2012)

RESULT AND DISCUSSION

Single Ninhydrin Crystal

The x-ray diffraction study was carried out by T.Uma Devi et.al (2008) for pure Ninhydrin

crystal. In this study they confirmed that Ninhydrin crystal belongs to monoclinic system.

Using KBr pellet technique the fourier transform infrared spectrum was recorded in the range of 400-4000 cm^{-1} . In this study the aromatic C-H stretching is observed at 3089 cm^{-1} and the aromatic C-H bond is observed at 740 cm^{-1} .

The optical transmittance spectrum is very essential for any NLO material because if the material has wide transparency window it possess many practical applications. To measure the optical transmittance spectrum of Ninhydrin they used Shimadzu spectrophotometer model 1601. The spectrum was recorded in the range of 300-1000nm. In this study the Ninhydrin shows less absorption around the value of 532nm.

The strength and plastic nature of a material is one of the mechanical properties which is determined using microhardness test. From this it was found that Ninhydrin comes under soft material category.

Potassium Nitrate Doped Ninhydrin Crystal

The X-ray diffraction study of Potassium nitrate Ninhydrin crystals was carried out by Sirine Hawech (2022). In this study they confirmed that potassium nitrate Ninhydrin crystal belongs to monoclinic system.

Thermal Analysis technique was carried out by Sirine Hawech (2022) to determine how a sample's mass is varied. The crystal begins to

lose weight at a temperature range of about 180°C-238°C which is achieved due to the release of carbondioxide.

Second harmonic generation study was carried out by Sirine Hawech et.al (2022).The emission of green colour light confirms the second harmonic efficiency of the grown crystal. The second harmonic efficiency of potassium nitrate Ninhydrin crystal is 0.20 times greater than potassium dihydrogen phosphate.

Hyperpolarisability calculation provides information regarding the nonlinear optical response of the grown crystal. It is also utilized to determine the nonlinear properties and links between the crystals.

Magnesium Doped Ninhydrin Crystal

The x-ray diffraction study was carried out by A.Ponchithra and Balasubramanian (2020). In this study they confirmed that Ninhydrin crystal belongs to monoclinic system.

A.Ponchithra and K.Balasubramanian (2020) analysed the mechanical property of Magnesium doped Ninhydrin crystals using micro hardness tester. It was found that the Magnesium doped Ninhydrin crystals come under soft material category.

Using Kurtz and Perry powder technique the second harmonic generation efficiency was calculated by A.Ponchithra and K.Balasubramanian (2020).Magnesium doped

Ninhydrin crystals showed little changes in second harmonic generation efficiency.

Nickel Doped Ninhydrin Crystals

The x-ray diffraction study was carried out by Anand Ponchithra et.al (2020). In this study they confirmed that the Nickel doped Ninhydrin crystal belongs to monoclinic system.

FTIR study is used to determine the molecular structure of crystals. In O-H vibration the peak is acquired at 3238cm^{-1} and C-H vibration is acquired at 3086cm^{-1} .

Anand Ponchithra et.al (2020) analysed the mechanical property of Nickel doped Ninhydrin crystal using micro hardness tester. It was found that the Nickel doped Ninhydrin crystals come under soft material category.

Anand Ponchithra et.al (2020) used dielectric analysis method to determine the dielectric constant, dielectric loss and conductivity at different temperature. For Nickel doped Ninhydrin crystal the dielectric constant value is 1096.

The second harmonic generation efficiency of Nickel doped Ninhydrin crystal was analysed using Kurtz and Perry powder technique by A.Ponchithra and K.Balasubramanian (2020). The second harmonic efficiency of Nickel doped Ninhydrin crystal is 1.47 which is comparable to Potassium dihydrogen phosphate.

Malic Acid Doped Ninhydrin Crystal

The x-ray diffraction study was carried out by K.Lilly Mary Eucharista et.al (2018). In this study they confirmed that the Malic acid doped Ninhydrin crystal belongs to monoclinic system.

K.Lilly Mary Eucharista et.al (2018) used UV-Visible spectral analysis method to determine the optical transmittance property of the crystal which give important information about the material. The transmittance of Malic acid doped Ninhydrin crystal is approximately calculated as 75%. The material is used in optical window application because its transmission extents from 270-1100nm. Malic acid doped Ninhydrin crystal have wide optical band which is suitable for optoelectronic applications.

K.Lilly Mary Eucharista et.al (2018) used this Vickers microhardness test to determine the mechanical character of Malic acid doped Ninhydrin crystal. The material carry information about yield strength, molecular binding and elastic stiffness constant. K.Lilly Mary Eucharista et.al (2018). Different magnitude has been applied during micro hardness test. It was found that Malic acid doped Ninhydrin crystal comes under soft material category.

K.Lilly Mary Eucharista et.al (2018) used fracture mechanics to find out the toughness of the grown material. Fracture toughness determines how much fracture stress is applied under uniform loading and it is an important

parameter for the selection of material for device application.

The second harmonic generation efficiency of Nickel doped Ninhydrin crystal was analysed using Kurtz and Perry powder technique by A.Ponchithra and K.Balasubramanian (2020). The second harmonic efficiency of Malic acid doped Ninhydrin crystal is 2.4 times greater than Potassium dihydrogen phosphate. This crystal is suitable for nonlinear devices.

Zinc Nitrate Doped Ninhydrin Crystal

The X-ray diffraction study was carried out by A.ponchithraet.al (2017). In this study they confirmed that the Zinc nitrate doped Ninhydrin crystal belongs to monoclinic system.

A.Ponchithra et.al (2017) used EDAX analysis to confirm the presence of different elements in the crystal sample. From this they concluded that 0.07% of Zinc ion is present in the interstitial sites of the Ninhydrin crystal.

A.Ponchithra et.al (2017) analysed the mechanical property of Zinc nitrate doped Ninhydrin crystal using micro hardness tester. It was found that the Zinc doped Ninhydrin crystals come under soft material category.

Metal Doped Ninhydrin Crystal

Powder X-ray diffraction analysis study was carried out by R.S.Sreenivasan et.al (2012). In this study they confirmed that the metal doped Ninhydrin crystal belongs to monoclinic system. The grown crystals have good crystallinity due

to strong peaks. Due to the addition of copper there is a small change in the intensity peak position.

The optical transmission spectral analysis spectrum is very important because the material possess many practical applications. The wavelength for upper cut off is 441nm. There is no absorption between the range 450 and 1200nm. This shows that the grown crystal is used as the window for material in optical application.

FTIR analysis plays a vital role in investigating the molecular structure of the crystal. The carbonyl peak is found at the range of 1747cm^{-1} and 1717cm^{-1} . The medium intense peak is observed at 1592cm^{-1} .

R.S.Sreenivasan et.al (2012) used dielectric analysis method to determine dielectric constant and dielectric loss at two different temperature i.e., 353K and 373K. It was found that dielectric constant decreases with increase in frequency and attains a constant value in the higher frequency range.

Second Harmonic generation test has been conducted to determine non-linear optical properties of the grown crystal. The pulse width is measured at 8ns. The emission of green colour confirms the presence of second harmonic efficiency of the grown crystal.

Copper Oxide Doped Ninhydrin Crystal

The x-ray diffraction study was carried out by T.Prasanyaa et.al (2012). In this study they confirmed that Ninhydrin crystal belongs to monoclinic system.

T.Prasanyaa et.al (2012) used UV-Vis method to determine the absorption peak in the given sample. The crystals are transparent in the entire visible region, the absorbing properties of doped ones are less than the pure Ninhydrin crystal.

T.Prasanyaa et.al (2012) analysed the mechanical property of the crystal using Leitz Weitzler micro-hardness tester. It was found that the Copper oxide doped Ninhydrin crystal comes under hard materials category.

The second harmonic generation efficiency of Copper doped Ninhydrin crystal was analysed using Kurtz and Perry powder technique by T.Prasanyaa et.al (2012).

CONCLUSION

From all the reviewed research papers it can be concluded that pure and doped Ninhydrin crystals can be grown by slow evaporation method. From single xrd study it was found that both pure and doped Ninhydrin crystals belongs to monoclinic system, second harmonic generation study revealed that both pure and doped Ninhydrin crystals can be used in industrial communication, optical electronic device fabrication and non-linear device fabrication.

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