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POWDER X-RAY PATTERN ANALYSIS OF 4-AMINOPYRIDINE BASED COPPER (II) NITRATE TRI HYDRATE CRYSTALS

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Abstract

The copper (II) nitrate tri hydrate reacts with 4-aminopyridine and crystalized by slow evaporation method in hexagonal crystal system with lattice parameter a = 9.2329 Å, b = 9.2329 Å, c = 15.3081 Å. Using Full width half maximum plot, we could confirm that the Breadth decreases with increase in particle size (t) with mosaic defect.

Keywords

4-aminopyridine, copper (II) nitrate tri hydrate, powder XRD, particle size, hexagonal.

1. Introduction

4-aminopyridine is a potassium channel-blocking agent, used as a bird repellent, and also it increases calcium influx at presynaptic terminals thereby enabling improvement of neuromuscular transmission in myelinated neurons [1-7]. Copper II nitrate has a number of uses, such as in ceramics, in dyeing as a mordant, in fireworks and in photography. Nitrate materials are also used as oxidizing agents. When nitrate materials mixed with hydrocarbons, they can form a flammable mixture. Nitrates are excellent precursors for production of ultra-high purity compounds and certain catalyst and nano-scale materials [8].

2 Materials and Methods

2.1 Preparation

Solutions of 4-aminopyridine and copper (II) nitrate tri hydrate in water (20 ml) each are mixed in molar ratio of one is to one. Blue colour crystals were obtained by slow evaporation after a period of two weeks.

2.2 Powder X-ray diffraction

Powder x-ray diffraction pattern were collected from diffracto-meter XPERT-PRO, with starting position $2\theta = 10.0231^{\circ}$, end position $2\theta = 80.9231^{\circ}$, step size $2\theta = 0.0500^{\circ}$, specimen length = 10.00 mm, measurement temperature = 25°C, Cu as anode material and K_a = 1.54060 Å.

3. Result and Discussion

X-ray diffraction is a tool for the investigation of the fine structure of matter. The copper (II) nitrate tri hydrate reacts with a 4-aminopyridine and results in stable blue colour crystal in hexagonal crystal system with lattice parameter a = 9.2329 Å, b = 9.2329 Å, c = 15.3081 Å. $\alpha = \beta = 90^{\circ}$ and $\gamma = 120^{\circ}$. The powder x-ray diffraction pattern for the as grown crystals is shown in figure 1 and they were indexed and tabulated in Table 1.

Good crystal growth were identified in the direction of planes (0 1 0) and (0 2 0) for the crystal which was grown in water environment. Figure 2 shows the fill width half maximum value for the one peak. The Scherrer formula is used to estimate the particle size of very small crystals.

The calculated breadth B of 2θ ° due to small crystal effect alone of powder pattern line of particle is shown in table 2. From this table we could confirm that the Breadth decreases with increase in particle size t with mosaic defect.



Figure 1 Powder x-ray diffraction pattern of complex

Table 1 Indexing of powder x-ray diffraction pattern

Pos. [°2Th.]	Height [cts]	FWHM left [°2Th.]	d-spacing [Å]	Rel.Int. [%]	h k l
10.9293 11.5424	6511.25 2379.08	0.1476 0.1476	8.09539 7.66675	68.99 25.21	0 1 0 0 0 2
19.5309	1690.43	0.1476	4.54522	17.91	110
22.1735	9438.60	0.0984	4.00913	100.00	020
29.8004	110.88	0.1476	2.99816	1.17	220
33.6443	4896.15	0.1476	2.66390	51.87	030
40.9247	795.04	0.1476	2.20525	8.42	222
45.4691	2034.24	0.1476	1.99486	21.55	040
57.7756	648.08	0.1968	1.59583	6.87	050

Crystal data

Cell parameter a = 9.2329 Å,

b = 9.2329 Å, c = 15.3081 Å, $\alpha = \beta = 90^{\circ}$, $\gamma = 120^{\circ}$.

Crystal system: Hexagonal



Sample: Copper Nitrate





S.No	Calculated t Å	$B^{\circ} = \theta 1 - \theta 2$	Θ _B °
1	190.85	0.74	10.983
2	205.10	0.69	11.580
3	249.54	0.6	22.181
4	288.56	0.51	19.597
5	308.56	0.64	45.436
6	427.02	0.39	33.643

Conclusion

The 4-aminopyridine interact with copper II nitrate tri hydrate in water, and blue coloured crystals were harvested using slow evaporation method and the crystal system and the particle size of the as grown crystals were calculated using the powder X-ray diffraction pattern.

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