

Reduced morphological change in copper II Chloride in caprylic acid solution

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ABSTRACT

In this paper, slow evaporation method was used for the synthesis of Copper II chloride in orthorhombic crystal system and it was characterized by the powder X-ray diffraction pattern. The grown crystals have reduced cell parameters compared to the earlier work. The variation found for 'a' is 1.3841, 'b' is 2.6193 and 'c' is 0.0078. This is because of the crystal growth in caprylic acid environment. Using the X-ray broadening, the crystallite sizes and lattice strain on the peak broadening were studied by Williamson-Hall plot. The result of mean particle size showed that the particle size increases with the decrease in breadth.

Key words: Cupric chloride dihydrate, caprylic acid, Powder X-ray diffraction pattern, Williamson-Hall plot

1. INTRODUCTION

Cupric chloride dehydrate is used as catalyst for organic and inorganic reactions; in petroleum industry as deodorizing, desulphurising and purifying agent. As mordant for dyeing and printing textiles; as oxidizing agent for aniline dyestuffs; Also used in indelible, invisible and laundry marking inks. Manufacture of fast black - melanin. In wet process metallurgy recovering mercury from ores, in refining copper, silver, gold. In tinning baths for iron. Electroplating copper on aluminium. In photography as a fixer, desensitizer. Colour in

pyrotechnic compositions. Pigments for glass, ceramics. Component of wood preservative, disinfectant. Component of animal feed supplement.

The crystal structure of cupric chloride dihydrate and the positions of the copper, chlorine and oxygen atoms were first obtained by X-ray diffraction by Sydney Brownstein co-workers [2]. CuCl_2 reacts with acid to give complex ions in green or yellow solid.

Caprylic acid is used as disinfectant in health care facilities, schools/colleges, animal care/veterinary facilities, industrial facilities, office buildings, recreational facilities, retail and wholesale establishments, livestock premises, restaurants, and hotels/motels. In addition, caprylic acid is used as an algicide, bactericide, and fungicide in nurseries, greenhouses, garden centers, and interiorscapes on ornamentals. Products containing caprylic acid are formulated as soluble concentrate/liquids and ready-to-use liquids[3]

2 Materials and Methods

2.1 Preparation

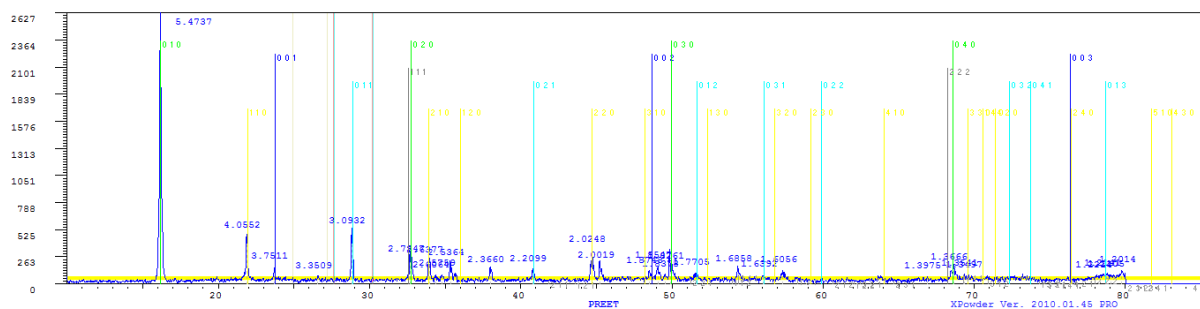
All the reagents used for the preparation of sample are analytical grade and the solutions are prepared using pure deionized water. Solutions of cupric chloride dihydrate and caprylic acid in water (10 ml) each are mixed in molar ratio of one is to two. The solution was uniformly stirred for 30 min and heated at 303 K for 2 h. The resulting solution was allowed to cool slowly to room temperature. Green crystals were obtained by slow evaporation after a period of two weeks.

2.2 Powder X-ray diffraction

Using the Rigaku Ultima III *XRD diffractometer with Graphite monochromator* the powder X-ray diffraction pattern were collected. The generator power settings were at 40 kV and 40 mA. Data were collected between a 2θ of $10-80^\circ$ with a step size of 0.02° at a scanning speed of 2.0 deg/min.

3. Result and discussion:

The X-ray crystallographic pattern of the sample in orthorhombic crystals system is shown in the figure 1. The values of $\sin^2 \theta$ for each peak have been calculated with help of cell parameters and the corresponding h, k, l in all cases are in good agreement with observed values as in tables 1. The lattice constants a, b, c for each unit cell have been found out and are tabulated.

Figure 1 X-ray diffraction pattern**Table 1 Indexing of the sample**

N 2-theta d-spacing Counts InT/100

1	16.18	5.4737	2627	100.0
2	21.90	4.0552	456	17.3
3	23.70	3.7511	159	6.0
4	26.58	3.3509	66	2.5
5	28.84	3.0932	501	19.1
6	32.72	2.7347	231	8.8
7	33.96	2.6377	224	8.5
8	34.38	2.6064	74	2.8
9	34.80	2.5759	90	3.4
10	35.36	2.5364	197	7.5
11	38.00	2.3660	129	4.9
12	40.80	2.2099	131	5.0
13	44.72	2.0248	325	12.4
14	45.26	2.0019	169	6.4
15	48.52	1.8748	119	4.5
16	49.08	1.8547	167	6.4
17	49.56	1.8378	87	3.3
18	49.90	1.8261	157	6.0
19	51.58	1.7705	103	3.9

20	54.38	1.6858	131	5.0
21	56.06	1.6392	83	3.2
22	57.34	1.6056	127	4.8
23	66.90	1.3975	70	2.7
24	68.62	1.3666	154	5.9
25	69.34	1.3541	96	3.6
26	69.60	1.3497	77	2.9
27	78.20	1.2214	75	2.8
28	78.76	1.2141	95	3.6
29	79.04	1.2105	84	3.2
30	79.76	1.2014	130	4.9

Table 2 Cell parameters

$$a = 6.0300 \text{ \AA}$$

$$b = 5.4693 \text{ \AA}$$

$$c = 3.7380 \text{ \AA}$$

$$\alpha = 90^\circ$$

$$\beta = 90^\circ$$

$$\gamma = 90^\circ$$

$$v = 123.2788 \text{ \AA}^3$$

Figure 2 shows the full width half maximum value for the peak having hkl (010). The Scherrer formula $t = 0.9 \lambda / B \cos \theta_B$ is used to estimate the particle size of very small crystals from the measured width of their diffraction curves. The calculated breadth B of $2\theta^\circ$ due to small crystal effect alone of powder pattern line of particle is shown in table 3. From this table we could confirm that the Breadth decreases with increase in particle size t.

Figure 2 Full width half maximum curve for hkl (010)

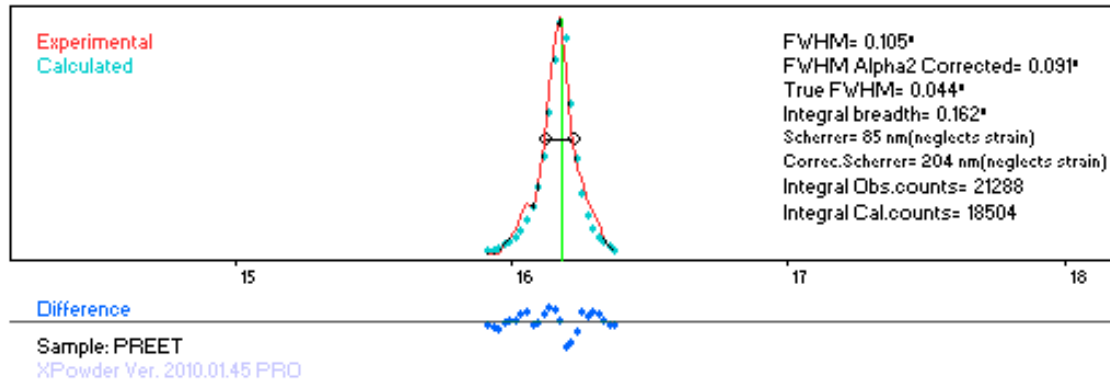
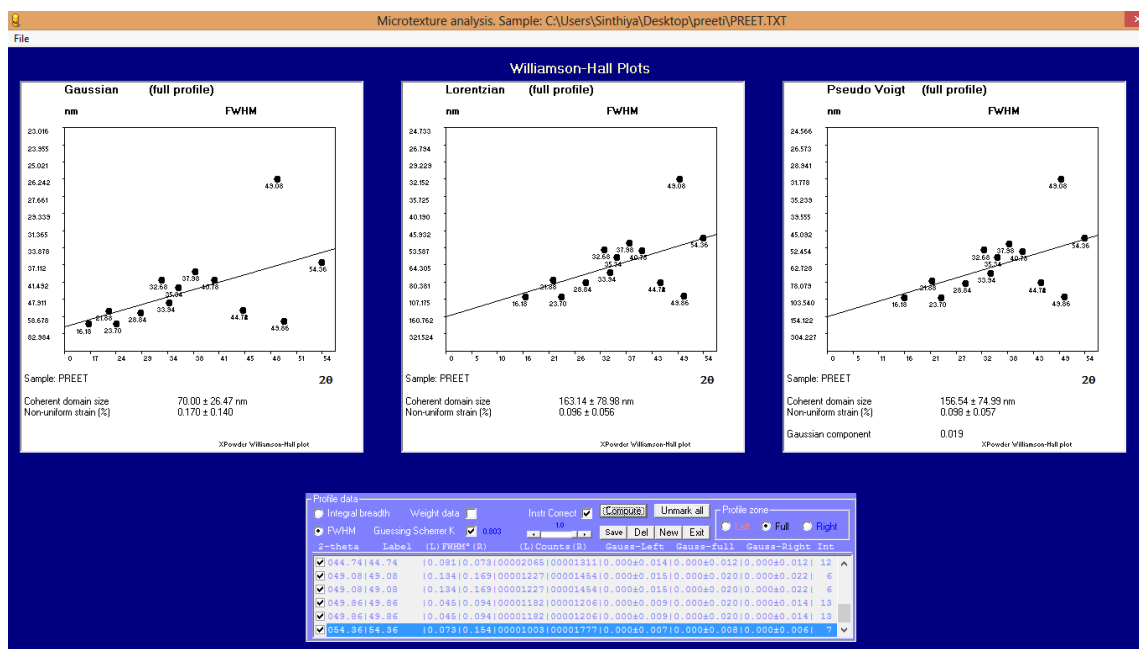


Table 3 Calculated t, B

S.No	Calculated t Å	$B^{\circ} = \theta_1 - \theta_2$	θ_B°
1	285.36	0.3	10.94
2	348.81	0.26	14.42
3	356.63	0.225	8.1

Williamson-Hall plot has illustrated that line broadening was basically isotropic. Due to microstrain contribution the diffracting domains were isotropic. Size-strain parameters can be obtained from the “size-strain plot” (SSP). This has a benefit that less importance is given to data from reflections at high angles. In this estimation, it is assumed that profile is illustrated by “strain profile” by a Gaussian function and the “crystallite size” by Lorentzian function.

Figure 4 Williamson-Hall plot



4 CONCLUSION

In summary, we have successfully synthesised the green color copper II chloride with orthorhombic crystal system by slow evaporation method. And we could grow the crystals with reduced cell parameters that is the variation found for 'a' is 1.3841, 'b' is 2.6193 and 'c' is 0.0078 compared to the earlier work. The particle size and broadening were analysed by the scherrer formula, modified form W-H analysis and the size strain plot method. As a result we could conform the uniformity of atoms present in the prepared crystal.

REFERENCE

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