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Powder X-ray diffraction pattern analysis of 4-aminopyridinium adipate

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ABSTRACT

In this paper, slow evaporation method was used for the synthesis of 4-aminopyridinium adipate in triclinic crystal system and it was characterized by the powder X-ray diffraction pattern. 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 (in degrees) with mosaic defect.

1.Introduction

Fampyridine, a new sustained release oral tablet of 4-AP is currently under phase III clinic trial for its therapeutic efficacy in patients with multiple sclerosis (MS) and chronic spinal cord injury. Low concentration of 4-AP are considered to block transient, voltage activated, outward K+ currents [1]. Potassium ions (K+) and sodium ions (Na+) flow between the neurons and the extracellular fluid to set up the state of electrical charge potential when the neuron is at rest (the resting potential) and to release that potential when the neuron is sending a nerve impulse (the action potential). Sodium ions flow through other special gates called “sodium channels”. . Voltage – gated channels open or close in response to the surrounding electro – potential while chemically gated ones open and close in response to chemical stimuli. Dendrites, the branched filaments that receive nerve transmissions from other neurons, tend to have more chemically – gated potassium channels and fewer voltage – gated potassium channels by comparison with axons, the long extensions that send these transmissions onto other neurons. Clinically for Lambert – Eaton myasthenic syndrome 4aminopyridine is used because it is blocking potassium channels. It prolongs action potentials thereby increasing transmitter release at the neuromuscular junctions [2,3,4,5]. The structure of 4- aminopyridine C5H6N2 has been redetermined at 150 K [6].

Adipic acid (hexanedioic acid) is a high purity, white, crystalline powder which is primarily used by INVISTA in its nylon polymer production. Exhibiting typical carboxylic acid chemistry, its excellent uniformity and consistent end-use performance make it a valuable intermediate in a wide range of applications [1]. As a monomer in nylon, paper additives, copolyamides, terpolymers, and unsaturated polyester resins (UPRs), [2]. As polyester polyols for polyurethanes, [3]. In polymer additives for epoxy curing agents and plasticizers, [4]. As a chemical intermediate in synthesis of polyesters/diesters, polyester polyols, cyclopentanone, 1,6-hexanediol and dimethyl adipate. [5]. In end-uses such as solvents, lubricants, electronics, soil conditioners, glass protection agents, briquetting agents, leather tanning agents, flue gas desulfurization scrubbers and cleaning aids [7].

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 4-aminopyridine and adipic acid in water (20 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. Colourless crystals were obtained by slow evaporation after a period of two weeks.

2.2 Powder X-ray diffraction

Using the Rigaku Ultima III XRD difractometer 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◦ 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 4-aminopyridinium adipate is shown in the figure 1. The complexes, belongs to the triclinic crystal system. The values of Sin 2θ 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 in table 2.

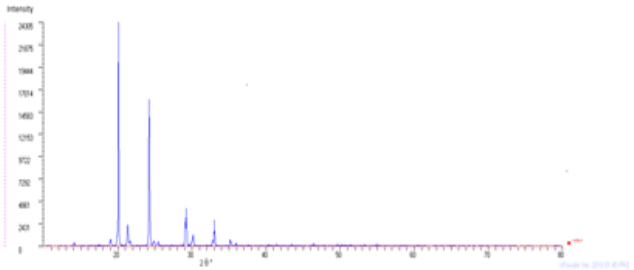

Figure 1. X-ray diffraction pattern for 4-aminopyridinium adipate.

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Table 1. Indexing of 4-aminopyridinium adipate.

S.No	2 θ °	d-spacing Å	hkl	Intensity count
1	14.10	6.2761	001	320
2	19.00	4.6671	010	650
3	20.08	4.4185	100	20845
4	21.32	4.1642	-101	2375
5	24.22	3.6787	011	14670
6	24.26	3.5787	-111	525
7	29.18	3.0580	1-11	405
8	30.14	2.9627	-102	2705
9	33.02	2.7106	-112	2700
10	35.20	2.5475	012	500

Table 2 Cell parameters

a = 4.7917 Å
b = 4.9116 Å
c = 6.5863 Å
α = 88.6647°
β = 104.7288 °
γ = 107.5901°
v = 142.6636 $Å^3$

Figure 2 shows the fill width half maximum value for the peak having hkl (100). The Scherrer formula t = 0.9 λ/ B cos θ_B is used to estimate the particle size of very small crystals from the measured width of their diffraction curves.

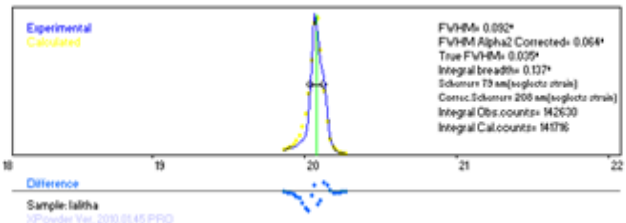

Figure 2. Full width half maximum curve for hkl (100).

The calculated breadth B of 2θ ° 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 with mosaic defect.

Table 3. Calculated t, B for 4-aminopyridinium adipate

S.No	Calculated t Å	B ° = θ_1- θ_2	θ_B°
1	154.90	0.52	9.52
2	186.99	0.44	15.08
3	200.13	0.4	7.08
4	202.09	0.4	10.66
5	216.05	0.38	14.62
6	270.82	0.3	12.1
7	295.90	0.28	16.5
8	403.39	0.2	10.04

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.

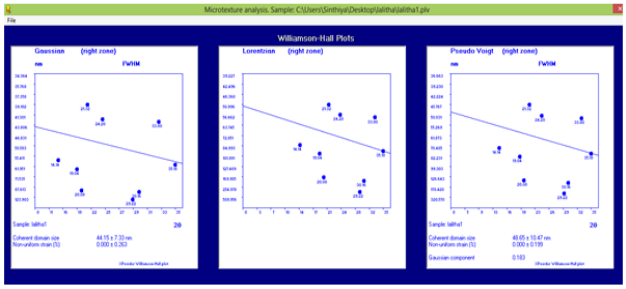

Figure 4. Willamson-Hall plot of 4-ainopyridinium adipate.

Conclusion

In summary, we have successfully synthesised the 4-aminopyridinium adipate with triclinic crystal system by slow evaporation method. 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.

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