International Journal of Physics and Applications.
ISSN 0974-3103 Volume 5, Number 1 (2013), pp. 1-5

http://www.irphouse.com

A Comparative Report on Micro Structural Analysis

A. Sinthiya and S. Amsareka

Department of Physics, St. Joseph's College (Autonomous), Tiruchirappalli – 620 002, INDIA
Email : r.brightson2010@gmail.com

Abstract

Blue colored crystals were harvested by slow evaporation method under two conditions. That is one sample is prepared by ordinary method and the other by the ultrasonic bath. The I_{max}=36 for the sample 2 which was prepared using ultrasonic bath and the I_{max}=54 for the sample 1 which was prepared by ordinary condition. Therefore by using ultrasonic bath we could get the crystals with reduced size than the normal and ordinary conditions. From the cell parameters we could compare the growth of the crystal that is the crystals atomic distance along the x-axis is reduced for the crystal which was prepared by ultrasonic bath. HRSEM results shows that, the presence of impurity is completely blocked by using the ultrasonic bath.

Key words: Ultrasonic bath, cell parameters, HRSEM

Introduction

L-proline (C5H9NO2) is a non-essential amino acid which means the body can synthesize sufficient quantities to support its needs. L-proline is an essential component of Collagen and is important for proper functioning of joints and tendons. It is also used for the synthesis of various pharmaceuticals, such as hypertensive agents. L-proline is employed for parenteral and enteral nutrition. L-proline is manufactured by fermentation from carbohydrate sources. Ajinomoto uses vegetables as the raw materials. L-proline is 100% free of materials of animal origin. Ajinomoto produces L-proline under cGMP conditions in its US facility. L-proline meets USP and EP standards and a Drug Master File is on record at the FDA.[1] .

Sodium chloride is used in veterinary medicine as emesis causing agent. It is given as warm saturated solution. Emesis can also be caused by pharyngeal placement of small amount of plain salt or salt crystals. In humans, a high-salt intake has long been suspected to generally raise blood pressure. More recently, it was demonstrated to attenuate nitric oxide production. Nitric oxide contributes to vessel homeostasis by inhibiting vascular smooth muscle contraction and growth, platelet aggregation, and leukocyte adhesion to the endothelium [2].

Experimental procedure

L-proline was dissolved in water. Saturated solution was prepared. Water containing sodium chloride was added to this saturated solution. Blue colored mixture (sample 1) was harvester after 20 days.

Same solution was prepared under the same condition and it was kept at ultrasonic bath for 5 minutes to make miscible liquid from immiscible liquid. Blue colored mixture (sample 2) was harvested after 2weeks.

Results and discussion

X-ray data collection

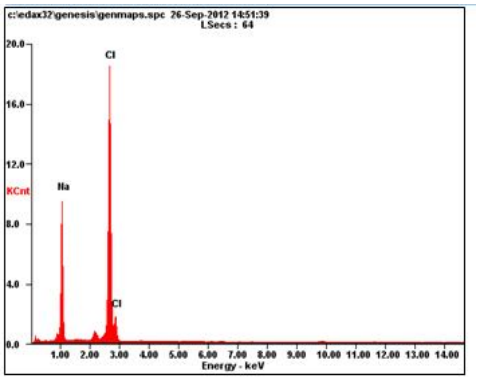
The X-ray data was collected using Bruker SMART APEXII CCD diffractometer (Mo Kα, λ = 1.54178Å). Indexing was performed using difference vector method. For the sample 1 the cell parameters are a=b=c=5.57Å, α=β=γ =90°, Volume = 173Å^3. The peak intensity data were collected for the reflection in the range of 54 at 2θ=14.81°. The measured miller indices is [-1.24,-0.05,-1.48]. For the sample 2 the cell parameters are a=5.80Å,b=c=5.77Å, α=β=γ =90°, Volume = 193Å^3. The peak intensity data were collected for the reflection in the range of 36 at 2θ=45.06°. The measured miller index is [-8.46,6.45,-1.76].

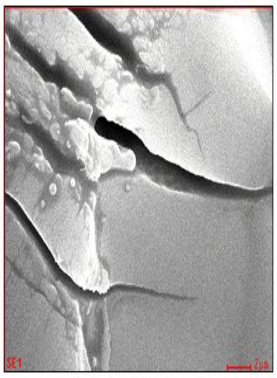
A mosaicity is defined as the rocking angle , in degrees, in both the vertical and the horizontal directions, which would generate all the spots seen on a still diffraction photograph. It includes contributions due to X-ray band width and beam crossfire. Mosaicity is refineable when integrating our frames if the three dimension window is set to be at least twice as large as the refined mosaicity [3]. For the sample 1 the data was analyzed with the mosaicity of about 0.80°. and for the sample 2 it is about 0.74° .

Microanalysis report

The data was collected using the HIGH RESOLUTION SCANNING ELECTRON MICROSCOPE, an advanced microanalysis instrument. While the regularity and order of crystals have been stressed thus far, it is important to note that this order can be distributed. Generally the cause is the inclusion of an impurity. Sometimes this is the result of a crystal forming around a

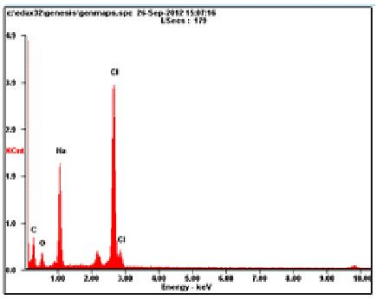
Foreign particle. This can usually be detected by microscopic examination. But other times it is actually an invasion by an atom with approximately the same size and shape as the host crystal, and the pattern is not disrupted. For sample 1 the presence of particles is shown in figure 1,2,3 and for sample 2 the presence of particle is shown in figure 4,5 .





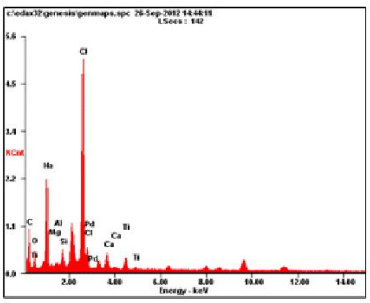
Element	Wt%	At%
NaK	43.12	53.89
ClK	56.88	46.11
Matrix	Correction	ZAF

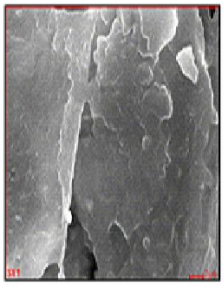
Figure 1: Presence of particles for 30.00kV at 2 micrometer (t_1=64sec)



Element	Wt%	At%
CK	55.83	71.18
OK	10.64	10.19
NaK	17.73	11.81
ClK	15.80	06.82
Matrix	Correction	ZAF

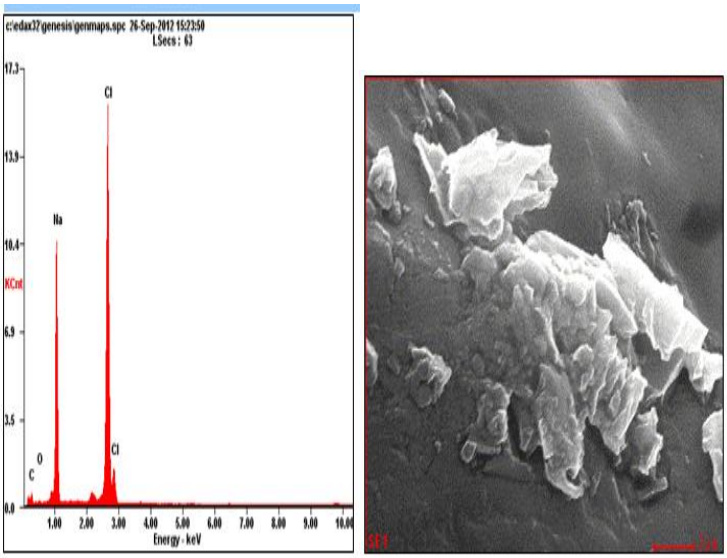
Figure 2: Presence of particles for 30.00kV at 2 micrometer (t_2=142sec)





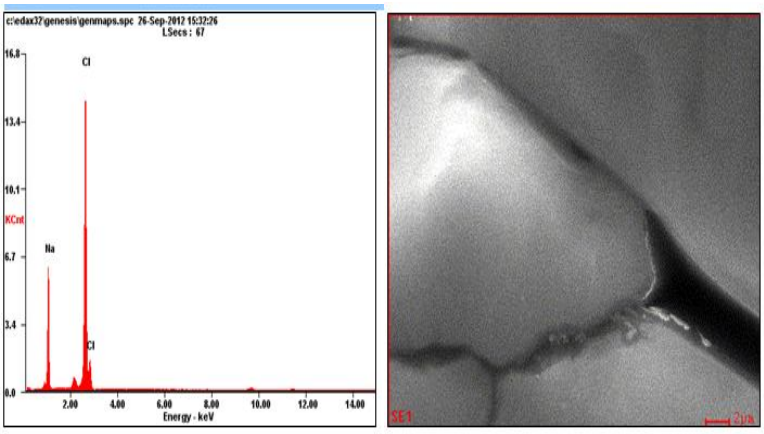
Element	Wt%	At%
CK	55.45	71.48
OK	11.34	10.97
NaK	13.19	08.88
MgK	00.81	00.52
AlK	00.82	00.47
SiK	01.40	00.77
ClK	13.36	05.83
PdL	01.11	00.16
CaK	01.38	00.53
TiK	01.16	00.38
Matrix	Correction	ZAF

Figure 3: Presence of particles for 30.00kV at 2 micrometer (t_3=179sec)



Element	Wt%	At%
CK	34.20	54.21
OK	01.47	01.75
NaK	32.61	27.01
ClK	31.72	17.03
Matrix	Correction	ZAF

Figure 4: Presence of particles at 30.00kV for 2 micrometer (t_1=63 sec)



Element	Wt%	At%
NaK	38.10	48.70
ClK	61.90	51.30
Matrix	Correction	ZAF

Figure 5: Presence of particles for 30.00kV at 2 micrometer (t_2=67sec)

From figure 1,2 and 3 for the sample 1, we could identify the impurities at micrometer level when we increase the exposure of electron beam to the sample 1. At t=142sec the average impurities atoms percentage is 0.404 % and the average impurities weight percentage is 0.95 %. But at t=179 sec, we could identify the coordination of L-proline and NaCl with the atom percentage of 71.18%,O=10.19%, Na=11.81% and Cl=6.82%. For t=64 sec the electron beam still separates the particles and shows the presence of NaCl with the atom percentage of Na=53.89% and Cl=46.11%.

From figure 4 and 5 for the sample 2, we could identify the coordination of Nacl and L-proline at t=63sec with the atoms percentage of C=54.21%,O=1.75%,Na=27.01% and Cl=17.03%. After four seconds we could get the presence of NaCl with the atom percentage of Na=48.70% and Cl=51.30%.

Conclusion

From the maximum intensities of X-ray diffraction we could compare the size of the samples. Therefore by using ultrasonic bath we could get the crystals with reduced size than the normal and ordinary conditions. The maximum intensity for the sample 2 which was prepared using ultrasonic bath is 36 and the maximum intensity for the sample 1 which was prepared by ordinary condition is 54. From the cell parameters we could compare the growth of the crystal that is the crystals atomic distance along the x-axis is reduced for the crystal which was prepared by ultrasonic bath. HRSEM results shows that, the presence of impurity is completely blocked by using the ultrasonic bath.

Reference

[1] *www.ajiaminoscience.com › Products >*
[2] http://www.saltinstitute.org/about-salt/chemical-properties
[3] www.hkl-Xray.com/sites/default/files/hkl2000manual/chapter2/step12-1.htm