Scientific Journal Impact Factor (SJIF) : 1.711
International Journal of Modern Trends in Engineering and Research
ISSN (ONLINE) : 2349 - 9745
ISSN (PRINT) : 2393 - 8161

XRD- analysis of alginate- Impregnated Hydroxyapatite Nano-Composites

Deepika Garg[1], Dr. Manoj Gupta[2], Dr. Ashsish Chouksey[3], Dr Hemlata Bundela[4]
[1]*Junior research fellow (MPCST&TIECT)*
[2]*Professor & Co-coordinator Deptt Humanity & Science*
[3]*Professor& Chief Co-coordinator , Electrical and Electronics*
[4]*Professor& Co-coordinator ,Deptt of Humanity & Science*
[2, 3, 4]*(Takshshila Institute of Engineering and Science, Jabalpur)*

Abstract—Different procedures for analysis of particle sizes by the X-ray diffraction method are compared by the example of nanoparticles of alginate-inpregnated-hydroxyapatite . A modified Warren-Averbach method is proposed for the analysis of the X-ray diffraction line profile based on the approximation by the Voigt function, which yields stable solutions, and the efficiency of the method is shown. The analysis within the frame-work of the Warren-Averbach method makes it possible to restore the distribution function of nanoparticles (crystallites) over true diameters, which satisfactorily correlates with electron microscopy data. The applicability of the Warren-Averbach method to the estimation of crystallite sizes by the analysis of a single diffraction line is substantiated. The range of the applicability of the Scherrer, Williamson-Hall, Warren-Averbach, and modified Warren-Averbach methods to the substructure analysis by the X-ray diffraction is determined as depending on the method of nanostructure formation.
Key words—Nano particles, alginate-inpregnated hydroxyapatite. X- ray diffraction.

I. INTRODUCTION

Powder X-ray diffraction study Powder X-ray diffraction has routinely been used as a nondestructive fingerprinting technique in laboratory and industry for several decades. Every solid crystalline compound gives its own unique X-ray diffraction pattern consisting of a set of Bragg peaks. The diffraction pattern for a compound can be considered analogous to a fingerprint, or barcode, with the peak positions determined by the unit cell symmetry and lattice parameters. When we collect XRD data, we can use these fingerprints to identify not only what phases are present in our sample but also index the pattern to obtain information about the unit cell size and shape. Powder diffraction patterns of the title compound has been collected with a SIEMENS D 5000 diffractometer using CuKα1 radiation (λ = 1.5406 Å). X-ray diffraction (XRD) is an effective method for identifying the phases present in unknown polycrystalline powders. The analysis is performed by comparing the diffraction pattern collected from an unknown sample with the diffraction patterns of known compounds. The automated process is called Search/Match (S/M) analysis. XRD is an important technique in the manufacture of ceramic materials. It provides phase analysis of materials throughout the manufacturing process,. Standard θ/2θ data were collected using a Rigaku MiniFlex benchtop diffractometer. The experimental patterns were compared with the diffraction scans of pure compounds maintained in the ICDD Powder Diffraction File (PDF). All scans were smoothed, theta corrected, and the background was removed.

II. MATERIALS USED

alginate-hadroxyapatite nano composites were used for XRD-analysis.

III. RESULTS AND DISCUSSION

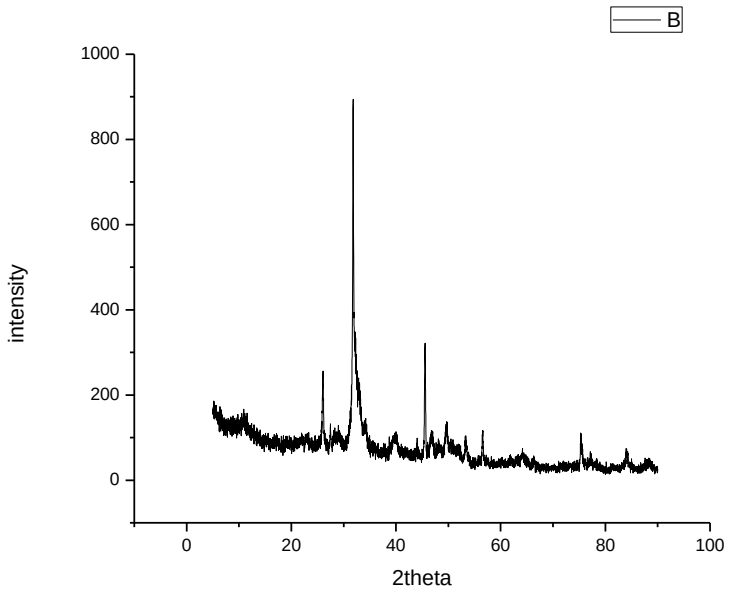


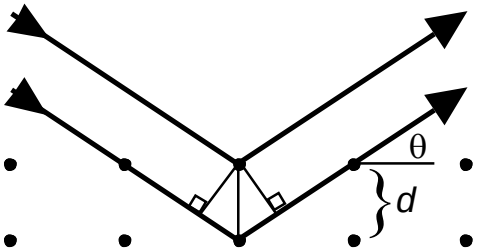
Fig1- XRD- graph of alginate-hydroxyapaptite-inpregnated hydroxyapaptite Nano composites

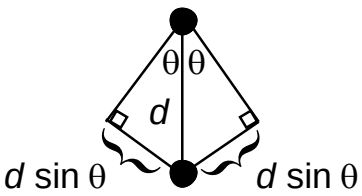
Scherrer Equation

It is assumed that if there are N different peaks of a specific nano crystal in the range of 0 - 180˚ (2θ) or 0 - 90˚ (θ), then all of these N peaks must present identical L values for the crystal size. But, during the extensive research of the first author of thins paper, on different nano ceramic crystals, which were synthesized or minerally achieved, it was surprisingly observed that each peak yields a different value and there is a systematic error on the results obtained from each peak. Further investigation approved the presence of a systematic error in Scherrer formula. In fact since , if L is going to be a fixed value forθ.cosβ λ =L K different peaks of a substance, considering that K and λ and therefore Kλ are fixed values, then it is essential that β.cosθ be a fixed multiple during 0 < 2θ < 180˚ or 0 < θ < 90˚. Suppose that for a crystallite size of 5nm, obtained at a peak of say 2θ = 10˚ (θ = 5˚) by using K = 0.89 and λCukα1 = 0.15405 nm. must be,°then β10 0.89 0.15405) (10) (0.0275 1.576 (8)⌋ ⌊ == = ° rad β5cos5 Now, suppose that the N th peak of this nano crystal occurs at 2θ = 170˚ (or θ = 85˚), then; 0.89 0.15405) (170) (0.3146 18.03 (9)⌋ ⌊ == = ° rad β5cos85 11.44=This means that the ratio of 18.03 1.576 . In other words if the firstβ / ° ° βmust be applied 170 10 for of around 2 mm on the monitor of°peak has a β10 computer plot, or for example, a paper plot 21 cm width more°on A4 paper, then the last peak must have a β170 than 22.88 mm and a base of peak more than 45.76 mm (4.576 cm). This has never been observed and cannot be true. Modified Scherrer formula is based on the face that we must decrease the errors and obtain the average value of L though all the peaks (or any number of selected peaks) by using least squares method to mathematically decrease the source of errors. We can write the basic Scherrer formula as: 1 . .cos cos K K L L (10)= = θ θ β λ λ Now by making logarithm on both sides; 1 ln ln ln ln .cos cos K K L L θ θ β λ λ (11)+= =), then aθIf we plot the results of lnβ against ln(1 cos straight line with a slope of around one and an intercept about lnK/L must be obtained. Theoretically this 3. Experimental straight line must be with a slope of 45˚ since tg45˚ = 1 (Figure 1). But, since errors are associated with experimental data, the least squares method gives the best slope and most accurate lnK/L. After getting the intercept, then the exponential of the intercept is obtained: Bovine bones were boiled for 2 hr to remove flesh and fat. The bones were heated at 60˚C for 24 hr to remove moisture.

Crystal System	Lattice Parameter Restrictions
Cubic	$a = b = c$ $\alpha = \beta = \gamma = 90^\circ$
Tetragonal	$a = b \neq c$ $\alpha = \beta = \gamma = 90^\circ$
Orthorhombic	$a \neq b \neq c$ $\alpha = \beta = \gamma = 90^\circ$
Monoclinic	$a \neq b \neq c$ $\alpha = \gamma = 90^\circ; \beta \neq 90^\circ$
Triclinic	$a \neq b \neq c$ $\alpha \neq \beta \neq \gamma \neq 90^\circ$
Hexagonal	$a = b \neq c$ $\alpha = \beta = 90^\circ; \gamma = 120^\circ$
Trigonal*	$a = b \neq c$ $\alpha = \beta = 90^\circ; \gamma = 120^\circ$

Bragg diffraction





For constructive interference,
$2(d \sin\theta) = n\lambda$

To prevent blackening with soot during heating, the bones are cut into small pieces of about 10 mm thick and heated at 400˚C (bone ash) for 3 hr in air to allow for evaporation of organic substances. The resulting black bone ash was heated for 2 hr at 600˚C, 700˚C, 900˚C and 1100˚C [5]. ln K L K e L (12)= λ λ Having K = 0.9 and λ(such as λCukα1 = 0.15405 nm), a single value of L in nanometer can be calculated

conclusion- 1) Scherrer equation systematically show increased vaue of nano crystalline size as d values decrease and 2θ values increase, since β.cosθ cannot be maintained as constant. 2) If lnβ is plotted against ln(1/cosθ) and least squares method is employed, the intercept gives ln = Kλ/L, from w to give a more ac 00˚C, 700˚C, 90˚C and 1100˚C, the values of 22 for producing HA for biomateria REFERENCES [1] P. Scherrer, " e und der Inneren Struktur von Röntgenstrahlen, s in the Determination of hich a single value of L can be obtained. 3) Modified Scherrer equation can provide the advantage)βlnΔ±(∑of decreasing the errors or 2 Cry curate value of L from all or some of the different peaks. 4) In a study on natural hydroxyapatite of bovine bone, fired at 6 .8, 35.5, 37.3 and 38.1 nm were respectively obtained for nano crystallite size.

ACKNOWLEDGMENT

Grateful to MPCST providing financial aids as projects, and also acknowledge to UGC-DAE for support in analysis and collecting related data.

REFERENCES

[1] Bestimmung der Gröss Kolloidteilchen Mittels Nachrichten von der Gesellschaft der Wissenschaften, Göttingen," Mathematisch-Physikalische Klasse, Vol. 2, 1918, pp. 98-100.

[2] J. Langford and A. Wilson, "Scherrer after Sixty Years: A Survey and Some New Result stallite Size," Journal of Applied Crystallography, Vol. 11, 1978, pp. 102-103. doi:10.1107/S0021889878012844

[3] H. Klug and L. Alexander, "X-Ray for Polycrystallite and Amorphous Diffraction Procedure Materials," 2nd Ediation of the ly-3-Hytion, John Wiley and Sons, New York, 1974.

[4] S. Dinilchenko, O. Kukharenko, C. Moseke, I. Protsenko, L. Sukhodub and B. Sulkio-Cleff, "Determin Bone Mineral Crystallite Size and Lattice Strain from Diffraction Line Broadening," Research and Technology Crystal, Vol. 37, No. 11, 2002, pp. 1234-1240.

[5] M. R. Foroughi, S. Karbasi and R. Ebrahimi-Kahrizsangi, "Physical and Mechanical Properties of a Po droxylbutyrate-coated Nanocrystalline Hydroxyapatite Scaffold for Bone Tissue Engineering," Journal of Porous Materials, Vol. 19, No. 5, 2011, pp. 667-675. doi:10.1007/s10934–011–9518–1.

[6] P. Shipman, G. Foster and M. Schoeninger, "Bu and Teeth: An Experimental Study of rnt Bones Color, Morphology, Crystal Structure and Shrinkage," Journal of Archaeological Science, Vol. 11, No. 4, 1984, pp. 307-325. doi:10.1016/0305–440

