# X-ray diffraction studies of dry bones

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**Abstract** : The regularlity in the microstructure of embalmed dry human skull bone has been investigated by X-ray diffraction technique The bone specimens were cleaned and cut to suitable size The X-ray diffraction pattern was obtained by a diffractometer

The intensity *versus* $2\theta$ profile shows only one main peak at $2\theta = 32\ 52°$ with intensity 118 CPS and width of the line as 1 11° This indicates regularlity in microstructure in one direction only Computation yields the value of crystallinity as 0.45 and the crystallite size as 83 Å approximately

The utilisation of X-ray methods of analysis is continuing to expand at a rapid rate. A regular arrangement of the lattice units, even though very imperfect, produces diffraction maxima. Many plastics are partly crystalline; composed of very long molecules, generally in a state of great disarray but here and there organized into ordered regions called crystallities. These regions produce broad diffraction lines. By comparing the intensity and width of lines, degree of crystallinity and crystallite size can be estimated [1].

Bones also have a regular microstructure. It consists of collagen fibrils and bone salts in crystalline and amorphous phase. The present work reports X-ray diffraction studies of dry human bones and computes the value of crystallinity and crystallite size.

Embalmed dry human skull bones were chosen for the investigation. The specimens were collected from Government Medical College, Jabalpur. These were cleaned by boiling in NaOH and then polished by the buffing technique. Samples of about $1 \times 1$ cm$^2$ dimensions were obtained from the outer compact layer of flat portion of the specimens. X-ray diffraction studies were carried out on Rotating Crystal Ribacu Diffractometer at the Thaper Centre for Research and Development, Patiala. The intensity *versus* $2\theta$ profile was

obtained using automatic step-scanning and strip-chart recorder. The intensity and width at various peaks were determined by computerised programme.

Figure 1 shows the plot of the diffractometer recorder trace of intensity *versus* $2\theta$ profile for embalmed dry bone. It can be observed that there is only one main peak at $2\theta = 32.52°$ with maximum intensity, 118 CPS and the width of this line at half maximum intensity, corrected for instrumentation broadening, is $1.11°$. This gives inter-planar distance as 2.75 Å. The intensity at all other diffraction angles is less than 25 CPS and may be attributed to scattering from amorphous portion.
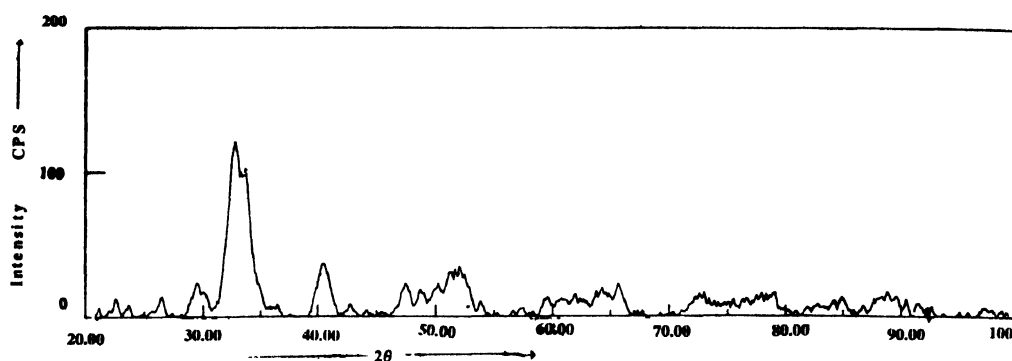


**Figure 1.** Intensity *versus* $2\theta$ profile for embalmed dry lone.

The peak at $2\theta = 32.52°$ shows certain regularlity in the microstructure of bones Corresponding to this peak, crystallinity and crystallite size have been calculated.

*Crystallinity :*

Crystallinity is a measure of regularlity in the arrangement of structural elements. The diffracted intensity scan can be separated into contributions from the sharp diffractions and diffused halo. A comparision of the relative areas under the resolved curves gives the percent crystallinity. The crystallinity of the sample is given by

$$CrI = \frac{\text{Area of crystalline fraction (under peaks)}}{\text{Area of crystalline + Area of amorphous fraction}}.$$

Various methods have been developed for measuring the areas under various fractions. Here, the crystallinity has been measured on the premise that increasing amorphousness tends to broaden the line width whereas increasing crystallinity increases the intensity. The height (CrH) of the main peak above its adjacent minimum represents the crystallinity of the sample and the width (AmW) of the peak at this adjacent minimum, is considered to represent the amorphousness of the sample [2]. The crystallinity CrI, is calculated from the relation.

$$CrI = 1 - t \times \frac{AmW}{CrH},$$

where $t$ is the scale factor relating the height of CrH to full scale (total blackness).

The height of the main peak at $2\theta = 32.52°$ is 10 mm above the adjacent minimum in the original tracing and width at this point is 0.0145 radian. Considering $t = 380$ mm for 1000 CPS, the crystallinity is obtained as

$$\text{CrI} = 1 - 380 \times \frac{0.0145}{10} = 0.449 \simeq 0.45.$$

*Crystallite size :*

The three dimensional crystal lattice diffracts X-rays in a manner analogous to the reflection of visible light from a ruled grating. The diffracted beams become diffused when crystal size is nearly the same as the wavelength of the incident beam. As crystals decrease in size, the diffracted beam becomes more diffused until it is lost in the general background. Thus, the divergence of X-ray beam is able to give the crystallite size. The relationship between crystalite size, CS and diffracted X-ray line broadening is given by Scherrer [3] as

$$\text{CS} = \frac{K\lambda}{\beta \cos \phi},$$

where
$\lambda$ = wave length of X-rays,

$\beta$ = pure diffraction broadening, in radian

$\phi$ = Bragg's angle, and

$K$ = a constant depending upon the crystalline shape, usually taken as unity.

In the present case, the pure width of the main peak is $1.11°$, the wavelength of X-ray used being 1.54 Å, the crystalite size is

$$\text{CS} = \frac{1.54}{\beta \cos \phi} = \frac{1.54 \times 360}{1.11 \times \cos 16.26 \times 2\pi} = 82.80 \text{ Å} \simeq 83 \text{ Å}.$$

Thus, the X-ray diffraction pattern of embalmed dry human skull bone shows that it has regularity in the arrangement of structural elements only in one direction. One can consider regular arrangement of lamellas which are 2.75 Å apart. The crystallite size of the order of 83 Å reveals that such regular arrangement of lamellas continues only upto 83 Å. This gives the degree of crystallinity as 0.45, in the particular direction.

**Acknowledgment**

**References**

[1]    B D Cullity *Elements of X-ray Diffraction* (California : Addision-Wesley) p 281 (1978)

[2]    W O Statton *Hand Book of X-ray Diffraction* ed. E F Kaelble (New York : McGraw Hill) Chap 21 p 1 (1967)

[3]    S F Bartram *Hand Book of X-ray Diffraction* ed. E F Kaelble (New York : McGraw Hill) Chap 17 p 1 (1967)