

6 ANALYTICAL METHODS

6.1 X-Ray Diffraction Spectroscopy

X-rays are defined as short-wavelength electromagnetic radiation produced by the deceleration of high-energy electrons or by electronic transitions involving electrons in the inner orbitals of atoms. The range of wavelengths is from 10^{-5} Å to about 100 Å. Conventional X-rays are confined to the region of approximately 0.1 Å to 25 Å [59]. The X-rays used in diffraction lie between 0.5 and 2.5 Å [59].

As with other types of electromagnetic radiation, the interaction of X-rays with particles in their pathway results in scattering. In a perfect crystal, scattering centres are located at fixed distances and spatially distributed in a regular way. Thus, at a certain angle of incident beam and a determined wavelength, the scattering will result in a constructive interference and maximum diffraction. The diffraction angles are intimately related to the interplanar distance of crystal sheets. This angle can be determined by *Bragg's Law* [59-61].

$$\sin \theta = \frac{n\lambda}{2d}$$

where θ is the angle of the incident beam on the crystal's surface; n is an integer; λ is the wavelength of the incoming X-ray radiation; and d is the interplanar distance of the crystal.

The interplanar distance of the crystal, d , in Bragg's equation is characteristic for each crystal structure. This is the basis of the X-ray powder diffraction method [59-61].

6.2 X-Ray Fluorescence Spectroscopy

A primary X-ray beam is focused on the sample in order to excite a secondary and characteristic X-ray beam. It is reasonable to think that the sample can be used as an X-ray source, thus producing the characteristic X-ray beam. This suggests the use of an electron beam as excitation source. The problem with such a procedure is that a conducting sample is needed and that most of the electron energy is converted into heat, causing problems of volatility [59-61].

The X-ray fluorescence method was used to quantify the different phases encountered during the different stages of the process. For quantitative purposes, it is sufficient to select one major line in the characteristic spectra of the element and relate that line to a concentration [59-61].

The X-ray spectrometer consists on an excitation source, a means of separating and isolating the characteristic lines, and a device for measuring characteristic line intensities [59-61].