MICROSCOPIC TECHNIQUES It is known that microscopic techniques such as scanning electron microscopy (SEM) and transmission electron microscopy (TEM) are widely employed to characterize the crystal structures, for example, morphologies as plates, rods, discs, cubes, ellipsoids, among others.In a usual transmission mode, the infrared beam passes through the sample, and the transmittance or absorbance of IR radiation as a function of wavenumber or wavelength is measured (Ryczkowski 2001).Image contrast can be achieved by elastic and inelastic scattering through interaction of the incident primary electron beam with the specimen and is divided into three classes: absorption contrast (sample image forming polycrystalline monophasic) (Mannheimer 2002); diffraction contrast (the scattered electrons can be arranged in the form of spots or rings, according to the Bragg's Law) (Mannheimer 2002; Canevarolo Jr 2004); phase contrast (obtaining of crystalline lattice images, range: 1 nm) (Canevarolo Jr 2004).From these crystallographic structures, the crystallographic descriptions were established, such as orthorhombic, hexagonal, cubic, octahedral, tetrahedral, and the atomic coordinates of polymorphous iron oxides (Cornell and Schwertmann 2000); (ii) energy dispersive X–ray (EDX) spectroscopy: the X–rays are emitted during the interaction with the electron beam, allowing the determination of chemical elements in the samples.The results are shown as spectra, which consists of a series of peaks representing the type and quantity of each chemical element of samples (Cornell and Schwertmann 2000; Dedavid et al. 2007); (iii) Fourier transform infrared spectroscopy (FT–IR) has been used as a technique for the investigation of the characteristic functional groups of iron oxides and characterization of precursors from different synthesis methods.As a result of the electron beam interaction with the sample surface, a series of radiation are emitted: (i) secondary electrons, which are emitted from the sample atoms with lowenergy (typically 50 eV or less); (ii) backscattered electrons (the reflection of images with topography contrast); (iii) X–ray detectors (used for chemical analysis) (Canevarolo Jr 2004).Spectroscopic techniques provide additional information to identify the chemical composition: (i) X–ray diffraction can be performed to obtain the crystalline structure of particles (angle position, width and intensity).