

XRD AND FESEM CHARACTERIZATION OF CRYSTALLITES AND PARTICLES OF FINE POWDERS FROM KAOLIN PROCESSING

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The commonly measured aspects of airborne particles are grouped (Klein, 1993) as: 1) Morphological; grain size, shape, habit (aspect ratio, fibrous nature) evaluated by high magnification optical microscopy, SEM, TEM. 2) Chemical composition; usually evaluated by EDS attached to TEM or SEM. 3) Structural information, produced from electron diffraction or structure images. These techniques among other including X-ray diffraction (XRD) are now used to analyze the finer particles in earth systems (Hochella et al, 2008). XRD quantitative analysis of silica crystalline phases in airborne particulate in industrial and urban environment was extensively discussed by Smidt (1992) and it is a standard technique (OSHA Method ID-142, 1996; NIOSH Method 7500, INSHT MTA/MA 056/A06) Silicates and phyllosilicates are frequent mineral components of natural and anthropogenic atmospheric dust, as result of the processes involved in the comminution size (e.g. weathering or other natural processes, and milling or other industrial processes) and show microstructural characteristics (crystallite size, shape and strain, size distribution, strain distribution, crystallite aggregation) that can be analysed by XRD microstructural methods These methods are successfully used for silicates and sheet silicates. Dimensions of particles of these materials often range in the nanometric scale, and thus accurate characterization is of great technological interest, having also implications considering human health. Some examples concerning XRD microstructural methods are referred to industrial minerals, focusing on the statistic value of these techniques, where results correspond to thousands of effective particles. New results are provided concerning kaolinite produced both by hydrothermal synthesis and high energy milling of reference kaolinites. Voigt function and Warren-Averbach methods are used, and their results are compared with results of analysis of images from TEM, FESEM and AFM. The found average crystallite sizes for hydrothermal kaolinites are in the range 18.4 - 29.4 nm. Electronic microscopy observations are in good agreement with XRD data, following the trends observed for average crystallite size and CSDs width when particle thickness (corresponding to the crystallographic direction studied by XRD) is measured with statistical significance.

Keywords: powder X-ray diffraction, dust, size