
Refinement of Goethite and Quartz from a Brazilian soil through the Rietveld Method

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The use of the Rietveld Method (RM) with X-ray diffraction data (XRD) in soils becomes difficult in relation to the best adjustment and higher reliability of the mineralogical quantification. One of these difficulties is related to the mineral phase starting crystalline structures, once they derive from natural samples. Therefore, it is necessary to seek starting structures of the mineral present in the soil closer to that found in Brazilian soils. This study analyzed samples coming from the hardsetting of a Yellow Latosol (Oxisol), Pará State. The procedure consisted of separating the mineral Goethite from the remaining minerals present in the clay fraction, through chemical treatment. The clay fraction was centrifuged and a very fine clay fraction was obtained from the supernatant which was possible to identify, through XRD, the minerals Goethite and Kaolinite and, through chemical treatment, Kaolinite was eliminated. The XRD enabled to identify the minerals Goethite, Analcime, Muscovite and Sodalite in the extraction product. In this chemically treated sample, the RM refinement was carried out and the Goethite structure was obtained. In the sand fraction sample, it was possible to identify, through XRD, the minerals Quartz and Goethite, and through RM the quartz structure presents in this fraction was refined by employing the previously refined Goethite structure. XRD analyses with CuK α radiation, in the step-by-step mode and extension and the RM refinements were carried out employing the GSAS. The refinement indices: Rwp=10.13%; S=1.80; R(F²)=7.58%, chemically treated fraction; Rwp=12.76%; S=3.96; R(F²)=9.84%, sand fraction.