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Structure solution from powder data using a combination of real-space method and Rietveld refinement

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Structure Determination from Powder Data (SDPD) is rapidly becoming a feasible alternative when suitable single crystals for structure determination cannot be grown, even for flexible organic molecules. We have been working on SDPD of flexible organic molecules from laboratory X-ray data using simulated annealing as implemented in DASH for several years now. Recently, while working on three molecular Ca^{2+} salts, we suspected, based on the unit-cell volumes, that the crystal structures were hydrates. Although far from unexpected for molecular Ca^{2+} salts, and although the presence of water molecules could readily be deduced, the exact number of water molecules (two or three) could not a priori be established due to the small size of a water molecule with respect to the organic fragments. Moreover, because the molecules were flexible and due to the additional degrees of freedom of the Ca^{2+} counter ion, structure solution attempts with the maximum number of waters (three) were hampered by the increase in the number of degrees of freedom due to the water molecules. Therefore, the crystal structures were solved in an iterative approach using a combination of simulated annealing in DASH[1] to determine the positions of the molecular ions followed by multiple partial Rietveld refinements in TOPAS[2] to locate the missing water molecules one by one; we note that this is common practice in structure determinations from single-crystal data. The partial Rietveld refinements progressed smoothly, yielding chemically sensible Ca^{2+} coordinations; the final Rietveld refinements showed excellent fits.

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Keywords: structure determination from powder diffraction, Rietveld refinement, hydrates

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Crystal structure refinement of waste magnesium by Rietveld analysis of X-ray diffraction data

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In the resent years, magnesium and magnesium-based materials have generated considerable interest for hydrogen storage medium because of high storage capacity. Magnesium in the form of hydride provides 7.6 wt % of hydrogen storage. The present study deals with the crystal structure refinement of waste-chip magnesium (WCM) by Rietveld analysis of X-ray powder diffraction (XRD) data. WCM, which is produced in the machining process of plastics made by molding, provided from gold manufacturing factory. Diffraction data were collected by using CuKα radiation (λ=0.15418 mm) at operating parameters of 40 mA and 45 kV (step size 0.05° and speed of 0.05°/s) in a range of diffraction angle from 10° to 90° using Philips Panalytical X'Pert-Pro diffractometer. Phase identification of sample was performed by International Centre for Diffraction Data (ICDD) data base and the XRD data indicated that the Magnesium (PDF: 01-089-5003) is the main phase presence with aluminum (PDF: 01-089-2769). Rietveld analysis was performed using the X'Pert High Score Plus analysis program based on Inorganic Crsytal Structure Database (ICSD). Rietveld analysis results was controlled by reliability factors; Rp=12.95%, Rexp=12.52%, Rwp=19.00%, GoF=2.3. Rietveld analysis of waste-chip magnesium as a possible hydrogen storage medium indicated that the Mg with P63/mmc hexagonal space group and the unite cell parameters are; a= 3.203642, b=3.203642, c=5.200827 is the main phase with a percentage 94.7 %. The other phase with a percentage 5.3 % is Al (Fm-3m space group and the cubic unite cell parameters a,b,c= 4.070235).

Keywords: waste magnesium, hydrogen storage, Rietveld analysis

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Structural phase transitions: Refining distortion-mode amplitudes instead of atomic coordinates

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For any crystal structure that can be viewed as a low-symmetry distortion of some higher-symmetry parent structure, one can represent the details of the distorted structure via distortion-mode amplitudes rather than the traditional list of atomic xyz coordinates. Because atomic coordinates and group-theoretically-derived distortion modes are related by a simple linear transformation, the number of structural degrees of freedom is the same with either basis. Many structural phase transitions, however, tend to activate a relatively-small number of specific distortion modes, while other modes have negligible amplitudes. In monoclinic room-temperature WO_3, for example, we find that only 5 of 24 available cubic-perovskite modes are substantially active. Thus the distortion-mode basis is far more convenient in terms of identifying and isolating the degrees of freedom that are important. By calculating distortion modes and communicating them to a Rietveld-analysis package, we will demonstrate that distortion-mode amplitudes can now be directly refined in place of atomic coordinates, even for phase transitions that break a great deal of symmetry. We will also show that simulated-annealing refinements of powder data, when performed on the distortion-mode basis, can be used to determine space-group symmetry.

Keywords: phase transitions, Rietveld refinement, distortion modes