Structure Determination and Rietveld Refinement Study of Coordination Polymers

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Structural evaluation of coordination polymers is extremely important if the synthesis and material properties of these species are to be understood. Ab initio powder X-ray diffraction structure solution has made a significant progress over recent years and has led to the evaluation of coordination polymer structures. Studies of the crystal structures of polymers are generally performed by X-ray diffraction (XRD) of powder samples containing randomly oriented crystals. The XRD patterns of semi-crystalline polymers present a few number of Bragg reflections and a large amount of diffuse scattering. The diffuse scattering originates from the amorphous contribution and the presence of structural disorder. The indexing of the diffraction pattern and the determination of the parameters of the unit cell of the semi-crystalline polymers are generally performed through trial and error methods. In crystals of polymeric materials, the macromolecules are longer than the unit cell parameters and each chain passes through many unit cells. Disorder may be present in the polymeric crystals, as some structural features maintain periodic positions, due to the presence of defects in the mode of packing. The degree of disorder in the packing or in the single macromolecules is sometimes so high that it is difficult to define this state as crystalline, even though we can still observe crystalline entities with a regular shape by microscopy. These crystalline forms that present large amounts of disorder with lack of periodicities in one or two dimensions are very common in solid semi-crystalline polymers. The presence of a high amount of disorder frequently prevents the identification of a unit cell and only average periodicities along some lattice directions may be predicted. Polymers are weak scatterers and frequently have only a few weak peaks. Spectral overlap is a major problem in these polymers. A lot of information about defects and disorder is hidden in the background of a powder pattern. When the number of observed independent reflection is not too low, the trial and error process may be replaced by the least square procedures treating the polymer chain as a rigid body. The advantage of using full-profile refinement method consists in the fact that starting from the structural models characterized by diffraction patterns in reasonable agreement with the experimental profile; it is possible to refine, on the same ground, structural and non-structural parameters. In the present work, we report the Rietveld Refinement study of Mn-adipoyl bis-isoniazid (Mn-ADBI) coordination polymer, through refinement of the powder XRD data, by taking into consideration an initial guess model. With the choice of a theoretical model of the structure through a database and subsequent entry of the theoretical data in the program followed the step of refining the experimental parameters. Structure factors calculated for trial structural models are compared with the observed values and satisfactory agreement was evaluated. One of the key reasons in obtaining the crystal structures is to draw relationships between structure and properties. This will provide insights that can be utilized to engineer materials for practical applications.

Figure 1. Structure of coordination polymers.

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