Powder and Single Crystal X-ray Diffraction Techniques in the Study of Packing Arrangements and Reactivity of Unsaturated Metal Carboxylates

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Powder and single crystal X-ray diffraction techniques continue to be the most valuable tools for the characterization of materials. One of our research interests includes the characterization of unsaturated carboxylic acids such as maleic, fumaric, methylmaleic, methylfumaric, itaconic, *cis*-4-cyclohexene-1,2-dicarboxylic, nicotinic, and isonicotinic acids and of their metal derivatives prepared in solution at room temperature, under hydrothermal conditions, and by mechanochemical routes. Metal-organic hybrid materials are particularly interesting due to their potential use as precursors to important metal oxides, as catalysts, and in gas storage, among other applications.

An unsaturated moiety may undergo solid state reactions such as degradation, isomerization, dimerization, and polymerization, induced by thermal and/or radiation sources that may lead to the formation of compounds not accessible by direct solution synthesis. Additionally, hydrothermal conditions may also induce rearrangements, hydrogen abstraction, loss of substituent, and loss of CO_2, among many other possibilities. Preliminary characterization of reagents, intermediates, and products is carried out by spectroscopic (FT-IR, NMR) and thermal methods of analysis (TGA-DTA, DSC), but the information obtained by X-ray diffraction techniques allows us to rationalize the outcome of the reactions in terms of coordination of the metal atoms, packing arrangements, hydrogen bonding patterns, etc.

In this presentation, the application of X-ray diffraction techniques to the study of [2+2] photopolymerization in the solid state, *cis-trans* isomerization, decompositions and rearrangements of the organic moiety under hydrothermal conditions will be discussed.

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