



Review

Structural transformations and solid-state reactivity involving nano lead(II) coordination polymers via thermal, mechanochemical and photochemical approaches



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ABSTRACT

Coordination polymers (CPs) may undergo structural transformations as a result of external stimuli (e.g., heat, light, or mechanochemical force). Structural transformations are accompanied by the movement of atoms or molecules and involve significant rearrangement of CP structures including rotation, bending, swinging, sliding, shrinking, or swelling. Lead(II) CPs display interesting structural features, varying properties and can be employed to investigate such transformations. In the present review, we focus on solid-state reactions of lead(II) CPs from a crystallographic point of view. Structural transformations of lead(II) CPs have been reported by our research group along with relevant examples from the literature. We also demonstrate that lead(II) CPs can create diverse structures with interesting features through such transformations. All the coordination networks have been determined by single-crystal X-ray diffraction before and after the structural transformations.

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Abbreviations: CPs, coordination polymers; SCSC, single-crystal-to-single-crystal; PXRD, powder X-ray diffraction; TG, thermogravimetry; DTA, differential thermal analysis; 3D, three-dimensional; C.T.C, crystal-to-crystal; NLO, nonlinear optics; CN, coordination number; CSD, cambridge structural database; 8-Quin, 8-hydroxyquinoline; 4-pyc, 4-pyridinecarboxylic acid; MPOAc⁻, mono phenyl acetate; Q-2-c⁻, quinoline-2-carboxylate; bpe, 1,2-di-(4-pyridyl)-ethylene; HTFA, trifluoroacetic acid; MOFs, metal-organic frameworks; 4-bpdh, 2,5-bis(4-pyridyl)-3,4-diaza-2,4-hexadiene; EDS, 1,2-ethanedithiolate; ⁻O₂C(CH₂)₂CO₂⁻, succinate; ⁻O₂C(CH₂)₃CO₂⁻, glutarate; LAG, liquid assisted grinding; Htrza, 1H-1,2,4-triazole-3-carboxylic acid; tpcb, tetrakis(4-pyridyl)cyclobutane; TFA, CF₃CO₂⁻; MOP, metal-organic polygons/polyhedral; ZIFs, zeolitic imidazolate frameworks; H₃hett, 5,5',10,10',15,15'-hexaethyltruxene-2,7,12 tricarboxylic acid; 9-HBN, 4-hydroxy benzotriazole.

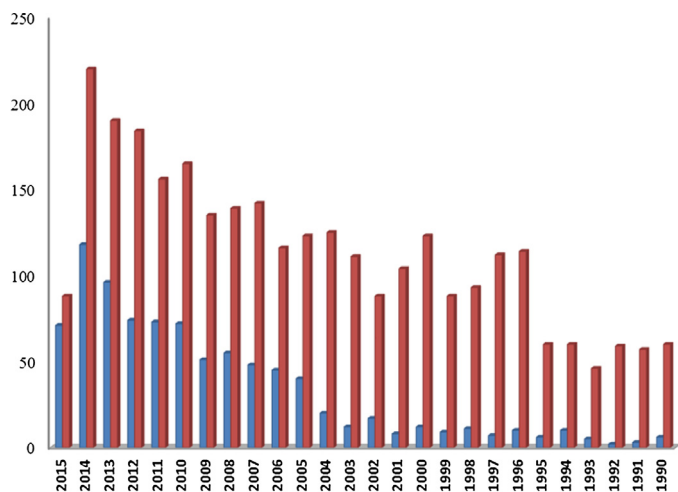
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1. Introduction

Recently, the modification in architecture of a compound via structural transformation has attracted considerable attention. Such methods have yielded new materials with special properties without the need for complex and long reactions [1,2]. Most of the structural transformations can be induced by applying external stimuli such as temperature [3–5], pressure [6–8] and radiation [9–11]. Moreover, these transformation can be achieved by soaking CPs in solvents [12–14] and treatment by gases [15,16]. The most essential point is that the crystalline state of the materials must be maintained during these transformations. As the crystalline



Scheme 1. The number of published articles containing the keywords “structural transformation” (red) and “SCSC and C.T.C.” (blue), survey by Scopus. (For interpretation of the references to color in this figure legend, the reader is referred to the web version of this article.)

order of a solid is maintained through a transformation, it is called single-crystal-to-single-crystal (SCSC) transformation. The principal method to clearly determine the structure of CPs is single-crystal X-ray diffraction [17]. These transformations are still challenging since crystals can hardly retain single crystallinity after a drastic solid-state rearrangement of the atoms. In some cases, even a minor structural adjustment would lead to the loss of overall single-crystallinity of the initial sample. In the absence of single crystals, other techniques such as powder X-ray diffraction (PXRD), thermogravimetry (TG) and differential thermal analysis (DTA) are also used, but their results are not conclusive without three-dimensional (3D) structural information. When 3D structural information is not available, recrystallization and solvent change can be employed to prepare an appropriate single crystal. In contrast to SCSC, if the compound loses its single-crystalline state, the transformation is called crystal-to-crystal transformations (C.T.C) (see Section 1.3)

Since CPs possess reactive functional groups and flexible frameworks, they have been thoroughly investigated in structural transformation studies [18,19]. They are employed in various applications such as ion exchange, catalysis, sensing, luminescence, nonlinear optics (NLO), magnetism, electrical conductivity, separation and storage [20–23]. Since structural transformations cause alterations in the CPs [24–28], only an external stimulus can produce a new CP with desirable properties. [29–31]. Structural transformation research has considerably advanced over the last decade. Scheme 1 shows the increase in the number of articles published in this area. Herein, factors that have a crucial role in these transformations are briefly discussed, and features of lead(II) as a sensible central metal for C.T.C transformation are described.

1.1. Special features of lead(II) CPs for structural transformation

Most of research on structural transformation has been focused on CPs containing transition metal ions [32–36] and lanthanide ions [37–40] while main group elements remained less investigated. Coordination environment around the central metal, in addition to the linkers, has a pivotal role in formation of a flexible structure. Our recent observations have indicated that lead(II) provides a flexible coordination environment owing to its $6s^2$ lone pair, variable coordination number (CN) (2–10) and large ionic radius, hence lead(II) is a suitable candidate for investigation of such transformations [41–45]. Shimoni-Livny evaluated the possible stereo-chemical

activity of the lone pair of lead(II) compounds based on a thorough review of crystal data available in the Cambridge Structural Database (CSD) [46]. They classified lead(II) coordination geometry as holodirected or hemidirected. The whole coordination sphere is evenly occupied by the bonds to ligands in the holodirected geometry. In contrast, the bonds are present only in a portion of coordination sphere leaving a gap in the distribution of bonds in the hemidirected [47,48]. The lead(II) CPs have photophysical properties such as luminescence, birefringence, photovoltaic conversion and fluorescence [49–51]. Lead(II) CPs rarely exhibit rigidity during structural transformation. To date, structural transformation of lead(II) CPs and their solid-state processes have been limitedly exploited in academic research, although they are suitable candidates for these reactions. For these reasons mentioned above, we focus on structural transformations of lead(II) CPs.

1.2. Role of solvent in the formation and structural transformation of CPs

Solvent has a strong influence on the behavior of crystalline systems and their structurally-related properties [52]. As a rule, solvent is an essential component for any reaction in solution. Intense interest in the dynamic role of solvents in chemical processes has also been evoked, in both theoretical and experimental aspects. In 1976, Bouren and Davey theoretically analyzed importance of the solvent in determining the mechanism of crystal growth and considered methods of quantifying the effects [53]. Also, Threlfall thermodynamically discussed the role of solvent in crystallization of polymorphs [54].

Macroscopic parameters of a solvent such as dielectric constant, polarity, viscosity and ionization potential etc., and microscopic ones such as van der Waals volume and electron density affect the coordination dynamics [55–57]. Most of the usual solvents are electron donors, thus they are highly influential in solution coordination processes [58,59]. Du and Li [60] described that coordination assemblies of specific reactants are influenced by the solvents used in the reactions. They discussed that a solvent can play its role as: (i) ligand, (ii) guest, (iii) both ligand and guest and (iv) structure-directing agent. As a ligand, solvents containing N or O donor atoms have different tendencies to coordinate to metal ions based on the hardness-softness of both solvent and metal [61,62]. As a guest molecule, several factors including steric effects [63,64], being aprotic or protic [65,66], hydrolysis [67] and synergy effects [68] are determinative. Different solvents with diverse coordination abilities and distinct steric effects may be included in the final structures in both ligand and guest roles when the assemblies take place in mixed solvent media [69]. In the fourth case, the solvent is absent in the final product, but indeed influences the crystal growth, crystalline morphology and lattice structure of the product (e.g., solvent-induced polymorphism) [70]. Most organic ligands show low solubility in water despite its unique features such as abundance, cheapness and harmlessness to environment etc., so organic solvents are usually used for synthesis of CPs [60].

Removal or exchange of solvents can cause structural transformations often manifested by various physical properties like color, magnetism, luminescence, chirality, porosity etc., (Scheme 2a) [71–80]. In many cases, removal of coordinated solvents from the CPs can occur in two ways: (I) both CN and coordination geometry change; (II) coordination geometry changes, but CN remains constant. These pathways are discussed in detail in this paper. This leads to a drastic movement of the molecular fragments resulting in structural transformation [81,82]. Occasionally, structural transformations occur during removal/addition of guest molecules. Guest molecules occupy void space in an open framework where they are involved in several types of non-covalent interactions such as H-bonding, π - π stacking etc. These guest molecules can

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