



Review

Applications of ultrasound to the synthesis of nanoscale metal–organic coordination polymers



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ARTICLE INFO

Article history:

Received 20 September 2014

Accepted 13 February 2015

Available online 23 February 2015

Keywords:

Ultrasound

Metal–organic frameworks

Coordination polymers

Supramolecule

Nanostructure

ABSTRACT

The purpose of this review is to provide a broad overview of sonochemical synthesis of crystalline metal–organic coordination polymers (MOCPs), i.e., the chemistry that takes place in the application of high-energy ultrasound to a reaction mixture of organic ligands and metal cations. Recent advances in nanostructured MOCPs have been led by the development of new synthetic methods that provide control over size, morphology, and nano/microstructure. The utilization of high intensity ultrasound offers a facile, environmentally friendly, and versatile synthetic tool for these nanostructured materials that are often unavailable by conventional methods. By focusing on recent advances, this article covers the sonochemical synthesis of nanostructured metal complexes, including mononuclear complexes, one-, two-, and three-dimensional coordination/supramolecular polymers (CPs), and metal–organic frameworks (MOFs).

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Abbreviations: 1,3,5-H₃BTC, 1,3,5-benzentricarboxylic acid; 1D, one-dimensional; 2D, two-dimensional; 3-bpbd, 1,4-bis(3-pyridyl)-2,3-diaza-1,3-butadiene; 3-bpbdh, 2,5-bis(3-pyridyl)-3,4-diaza-2,4-hexadiene; 3D, three-dimensional; 4-bpbd, 2,5-bis(4-pyridyl)-3,4-diaza-2,4-hexadiene; AOT, dioctyl sulphosuccinate sodium salt; ba, benzylacetylacetonate; bibp, 4,4'-bis(1-imidazolyl)biphenyl; bpydc, 2,2'-bipyridine-5,5'-dicarboxylate; CE, conventional electric; DEF, diethylformamide; DMF, N,N-dimethylformamide; DPOAc, diphenylacetate; Fum, fumarate; H₂BDC, 1,4-benzenedicarboxylic acid; HDPAcO, di-phenyl acetic acid; hfipbb, 4,4'-hexafluoroisopropylidenebis-benzoate; Htrza, 1H-1,2,4-triazole-3-carboxylic acid; ip, isophthalate; IRMOF, isoreticular metal–organic framework; MIL, Material of Institut Lavoisier; MOCP, metal–organic coordination polymer; MOF, metal–organic framework; MW, microwave; NCP, nanoscale coordination polymer; NMP, 1-methyl-2-pyrrolidone; PVP, polyvinylpyrrolidone; SEM, scanning electron microscopy; ST, solvothermal; TEA, triethylamine; TPOAc, triphenylacetate; tyr, tyrosine; UiO, University of Oslo; US, ultrasonic or ultrasound; ZIF, zeolitic imidazolate framework.

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1. Introduction

During the last two decades, synthesis and characterization of infinitely extended metal–ligand networks with metal centers and organic linkers, the so-called metal–organic coordination polymers (MOCPs), i.e., coordination polymers (CPs) and metal–organic frameworks (MOFs), has attracted immense attention [1–11]. This is the result of having the possibility to obtain a large variety of attractive structures that are of interest for applications in the fields related to porous solids, such as ion exchange, adsorption and storage of gases, purification and separation, drug delivery, sensors, catalysis, magnetism, non-linear optics, and adsorptive removal of hazardous materials [12–20]. High quality MOCPs crystals, such as those suitable for single-crystal X-ray diffraction measurements, can usually be obtained by a variety of approaches using wet solution chemistry and/or solvothermal methods [21]. In many cases, long reaction times are needed, usually several days for solvothermal, and several weeks for diffusion methods. Furthermore, the reaction of framework building blocks under solvothermal conditions has to be carried out at high temperatures (usually 373–523 K) and pressures (1–10 MPa). Also, because of the huge potential of MOCPs for industrial applications, it is important to develop novel and more efficient alternative synthetic techniques, that are techno-economically viable and able to be scaled up for the production of these compounds [22]. The newly developed approaches, such as mechanochemical [23–26], microwave (MW) [27–30] and electrochemical synthesis [31,32] provide a promising solution to this problem by dramatically shortening the synthesis times and simultaneously increasing the amount of product. Compared to traditional and even newly developed synthetic techniques, the sonochemical approach for the construction of MOCPs is more simple, convenient and controllable [33–39]. The use of ultrasound (US) for MOCPs synthesis is now positioned as one of the most powerful tools in CPs and MOFs preparation [40–48]. More recently, Jhung and Khan reviewed the synthesis of MOFs using MW and US irradiation [49]. They compared these two non-conventional methods with each other as well as with conventional electric (CE) heating in order to find which techniques were faster, and also get smaller nanoparticles. The excellence of US and MW over CE heating was found in terms of the degree of acceleration on the reaction rate, the selective synthesis of some MOF structures and the size reduction of MOF crystals [49–53].

In this review, we want to show how US-assisted synthetic methods could be successful in the preparation of hybrid metal–organic coordination materials at nano/micro-scales. In addition, we highlight how the reaction conditions, such as reaction time and temperature, power level of US irradiation, solvent content, concentration of reactants, pH and additive could be varied to get specific structures and morphologies. Table 1 summarizes some of the studies that undertaken for the sonochemical synthesis of some of MOCPs.

2. Sonochemistry

In comparison to the traditional energy sources (e.g., heat, light, and electric potential), US provides rather unusual reaction conditions (a short duration of extremely high temperatures and pressures in liquids) that cannot be realized by other methods [54]. The chemical effects of US do not come from a direct molecular level interaction with chemical species. Indeed, extraordinary conditions are not directly derived from US itself. Instead, sonochemistry mainly derives from acoustic cavitation [55]. Acoustic cavitation is the formation, expansion, and subsequent implosive collapse of bubbles in a liquid medium [56]. When liquids are irradiated with US, the alternating expansive and compressive acoustic waves create bubbles (i.e., cavities) and make the bubbles to oscillate.

The oscillating bubbles can accumulate ultrasonic energy while growing to a certain size (tens of μm). A bubble can overgrow and subsequently collapse within a very short lifetime (with a heating and cooling rate of $>10^{10} \text{ K s}^{-1}$) and can be considered as adiabatic. A large energy concentration is achieved during the collapse, resulting in a local temperature of $\sim 5000 \text{ K}$ and a pressure of $\sim 1000 \text{ bar}$. This energy is dispersed to the surroundings during the collapse, so that the gas temperature in the hot spot quickly returns to the ambient value [57]. Indeed, it is cavitation that is the key phenomenon responsible for sonochemical effects, by releasing enough kinetic energy to drive reactions to completion [58,59]. As a result, various chemical reactions, even some reactions that were previously difficult to realize by other traditional methods, can also proceed at room temperature using US irradiation [60–62]. The US method has been widely employed in several research areas including biological cell-disruption, medical imaging, thermoplastic welding, waste treatment and food processing [63–65]. Application of US to solid materials chemistry has been extensively explored by several research groups [66–68]. A large number of organic reactions and supramolecular polymer–metal complexes have been carried out under US irradiation in high yields within a short reaction time [69–85].

3. The primary efforts for the sonochemical synthesis of MOCPs

The first US synthesis of MOCPs was reported by Qiu et al. with a rapid preparation of a fluorescent microporous Zn carboxylates framework [86]. The nanocrystals of $\text{Zn}_3(\text{BTC})_2 \cdot 12\text{H}_2\text{O}$ were prepared from an aqueous solution of zinc acetate and 1,3,5-benzenetricarboxylic acid (H_3BTC) in a mixture of ethanol and water using an US bath at a frequency of 40 KHz (60 W). When compared to the CE synthesis (140 °C, 24 h), milder reaction conditions and faster reactions were reported (room temperature, 5–90 min). The size and morphology of the product varied with the reaction time. Shorter reaction times (5–10 min) led to spherical nanoparticles in the 50–100 nm range, while reaction times of 30–90 min resulted in needles with a diameter of up to 900 nm. Interestingly, no product was obtained by mixing zinc acetate with H_3BTC in the same reaction medium in the absence of US. Another research related to the US synthesis of MOCPs described the fabrication of nanosheets of a fluorescent MOF, $[\text{Zn}(\text{BDC})(\text{H}_2\text{O})]_n$ (BDC = 1,4-benzenedicarboxylate), at ambient temperature and atmospheric pressure using the same US bath [87]. Similar to the previous report, the size and morphology of the product were varied with reaction time. Reaction times of less than 20 min led to nanobelts of 150–300 nm width and a length of 2–5 μm and yield of 43.4%. Reactions for 20 min resulted in regularly quadrate nanosheet morphologies with sizes in the range of 500 nm–2 μm . Further, the increasing of reaction time led to an increase in dimensions as well as a yield of the nanosheets. However, when the reaction time was increased up to 90 min, nanosheets in a wide size range with irregular shapes were observed with the yield of 53.2%.

The kinetic study was reported for sonocrystallization of zeolitic imidazolate frameworks (ZIFs), ZIF-7, ZIF-8, ZIF-11, and ZIF-20 [88]. Coronas et al. revealed that pure crystals of ZIFs can be achieved by US with a power of 110 W and a frequency of 47 kHz in times shorter than with the CE heating (6–9 h vs. 24–72 h). In addition, the synthesis temperature was reduced in all three cases from those of the CE method of 140, 100, and 65 °C for ZIF-8, ZIF-11, and ZIF-20, respectively, to 45–60 °C in US technique. Moreover, small crystals with narrow particle size distribution were obtained by US, due to promotion of the nucleation process in the solution (see Fig. 1). Furthermore, crystallization curves were calculated from the XRD patterns, and the Gaultieri's model was applied to simulate the extent of crystallization as a function of time.

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