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Optimization of Cobalt Oxide Conversion to Co-Based Metal-Organic Frameworks

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Abstract : Gaining control over particle shape, size and crystallinity is an ongoing challenge for many materials. Especially metal-organic frameworks (MOFs) are recently widely studied. Besides their remarkable porosity and interesting topologies, morphology has proven to be a significant feature. It can affect the further material application. Thus seeking new approaches that enable MOF morphology modulation is important. MOFs are reticular structures, where building blocks are made up of organic linkers and metallic nodes. The most common strategy of ensuring metal source is using salts, which usually exhibit high solubility and hinder morphology control. However, there has been a growing interest in using metal oxides as structure-directing agents towards MOFs due to their very low solubility and shape preservation. Metal oxides can be treated as a metal reservoir during MOF synthesis. Up to now, reports in which receiving MOFs from metal oxides mostly present ZnO conversion to ZIF-8. However, there are other oxides, for instance, Co_3O_4, which often is overlooked due to their structural stability and insolubility in aqueous solutions. Cobalt-based materials are famed for catalytic activity. Therefore the development of their efficient synthesis is worth attention. In the presented work, an optimized Co_3O_4transition to Co-MOFviaa solvothermal approach was proposed. The starting point of the research was the synthesis of Co_3O_4 flower petals and needles under hydrothermal conditions using different cobalt salts (e.g., cobalt(II) chloride and cobalt(II) nitrate), in the presence of urea, and hexadecyltrimethylammonium bromide (CTAB) surfactant as a capping agent. After receiving cobalt hydroxide, the calcination process was performed at various temperatures (300–500 °C). Then cobalt oxides as a source of cobalt cations were subjected to reaction with trimesic acid in solvothermal environment and temperature of 120 °C leading to Co-MOF fabrication. The solution maintained in the system was a mixture of water, dimethylformamide, and ethanol, with the addition of strong acids (HF and HNO_3). To establish how solvents affect metal oxide conversion, several different solvent ratios were also applied. The materials received were characterized with analytical techniques, including X-ray powder diffraction, energy dispersive spectroscopy,low-temperature nitrogen adsorption/desorption, scanning, and transmission electron microscopy. It was confirmed that the synthetic routes have led to the formation of Co_3O_4 and Co-based MOF varied in shape and size of particles. The diffractograms showed receiving crystalline phase for Co_3O_4, and also for Co-MOF. The Co_3O_4 obtained from nitrates and with using low-temperature calcination resulted in smaller particles. The study indicated that cobalt oxide particles of different size influence the efficiency of conversion and morphology of Co-MOF. The highest conversion was achieved using metal oxides with small crystallites.

Keywords : Co-MOF, solvothermal synthesis, morphology control, core-shell

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