Fe_3O_4纳米材料的可控制备及其电化学性能研究

摘　要

材料、能源与信息，并称为21世纪经济社会的三大支柱产业。材料，特别是新兴的纳米材料与科技的高度发展密切相关，目前已广泛应用于石油、新能源、生物医疗、光学以及传感器等领域，纳米技术已俨然成为衡量国际科技水平的重要标准。纳米颗粒的尺寸、形貌以及结构是决定纳米材料在化学及物理方面特殊性能的重要因素。颗粒的几何结构决定材料性质的例子不胜枚举，所以对纳米材料不同结构的形成过程的研究以及对其进行有效调控是十分重要的。

本课题通过制备两种前驱体（MIL-53、MIL-88A），再通过不同条件下的煅烧（400℃氮气氛围、400℃氩气氛围、500℃氩气氛围）最终制备四氧化三铁的纳米材料。MIL-53的制备：称量0.540g $FeCl_3 \cdot 6H_2O$和0.332g对苯二甲酸（H_2BDC）。并分别用10mL DMF溶解这两种物质。通过使用磁力搅拌器混合两种溶液。将混合好的溶液放入超声波浴清洗器中处理10分钟。将溶液转移到反应釜中，放入已经加热到180℃的烘箱中，反应12小时。用DMF、乙醇各洗样两次，60℃真空干燥。MIL-88A的制备：称量64mg的九水合硝酸铁、32mg的反丁烯二酸，加8ml DMF。超声处理30分钟。油浴10分钟。用DMF、甲醇各洗样两次，60℃真空干燥。

通过XRD进行表征。采用三电极体系，将制备好的纳米材料活性物质与预处理后的泡沫镍进行压片制成工作电极，电解质采用2mol/L的氢氧化钾溶液，对电极采用铂电极，参比电极采用Hg/HgO电极。通过电化学工作站，测试制备的纳米材料的循环伏安曲线、恒流充放电曲线，确定电化学性能最优的制备条件。

关键词：纳米材料；MIL-53；MIL-88A；四氧化三铁；电化学性能

Abstract

Material, energy and information are called the three pillar industries of the 21st century economy and society. Materials, especially new nanomaterials, are closely related to the high development of science and technology. At present, nanotechnology has been widely used in petroleum, new energy, biomedical, optical and sensor fields. Nanotechnology has become an important standard to measure the level of international science and technology. The size, morphology and structure of nanoparticles play an important role in determining the special chemical and physical properties of nanomaterials. There are numerous examples of how the geometrical structure of particles determines the properties of materials, so it is very important to study the formation process of different structures of nanomaterials and to control them effectively.

Two kinds of precursors (MIL-53 and MIL-88A) were prepared, and then the nano-materials of Fe_3O_4 were prepared by calcination under different conditions (400 ℃ nitrogen atmosphere, 400 ℃ argon atmosphere and 500 ℃ argon atmosphere). The preparation of MIL-53: weighing 0.540g $FeCl_3.6H_2O$ and 0.332g terephthalic acid (H_2BDC). The two substances were dissolved in 10 mL DMF, respectively. The two solutions are mixed by magnetic stirring. The mixed solution was ultrasonic treated for 10 minutes. Subsequently, the solution was transferred to the reactor and reacted in an oven at 180 C for 12 hours. The samples were washed twice with DMF and ethanol, and dried in vacuum at 60 ℃. Preparation of MIL-88A: weighing 64 mg of ferric nitrate nine hydrate, 32 mg of fumaric acid, and adding 8 ml DMF. Ultrasound for 30 minutes. Oil bath for 10 minutes. The samples were washed twice with DMF and methanol, and dried in vacuum at 60 ℃.

It was characterized by XRD. The system of three electrode was used to make the active material of the prepared nanomaterial and the pretreated nickel foam into a working electrode. The electrolyte was 2mol/L potassium hydroxide solution, the platinum electrode was adopted for the electrode, and the Hg/HgO electrode was used