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溶液雾化氧化法制备超细 Co_3O_4 粒子及其性能表征

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摘要: 以氯化钴水溶液为原料采用雾化氧化法制备纯度较高的超细 Co_3O_4 粒子。采用热重-差热分析(TGA-DTA)对 $\text{CoCl}_2 \cdot 6\text{H}_2\text{O}$ 在空气中的热行为进行研究。由 $\text{CoCl}_2 \cdot 6\text{H}_2\text{O}$ 配制成原料溶液, 以压缩空气为载气, 采用内混气流式喷嘴雾化溶液, 并直接在竖立管式高温电阻炉内进行氧化反应, 制备的产物通过X线衍射分析(XRD)、傅里叶红外光谱分析(FT-IR)和扫描电镜(SEM)等进行表征, 同时对产物采用电位滴定法定量测定余氯含量, 研究雾化氧化反应温度对产物的影响。实验结果表明: 通过控制一定反应条件, 氯化钴溶液采用直接雾化氧化法可以制备纯度较高的 Co_3O_4 粉体粒子; 在雾化氧化过程中, 反应温度对所得产物纯度、形貌与结晶度产生重要影响。

关键字: 雾化氧化法; Co_3O_4 粒子; 性能表征

Preparation and characterization of ultrafine Co_3O_4 particles by solution spray-oxidation method

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Abstract: The ultrafine Co_3O_4 particles were synthesized using cobalt chloride solution as reactant by solution spray-oxidation method. The thermal behavior of $\text{CoCl}_2 \cdot 6\text{H}_2\text{O}$ in air atmosphere was studied by TGA-DTA analysis. The feed solution prepared by $\text{CoCl}_2 \cdot 6\text{H}_2\text{O}$ was sprayed using inner mixed air-nozzle and oxidized in the pipe high-temperature resistance furnace with compressed air as the carrier gas. The as-prepared products were characterized by X-ray diffraction analysis (XRD), infrared spectroscopy (FT-IR) and scanning electron microscopy (SEM), and the remaining chlorine content of the as-product was determined by potential titration. The reaction temperature of spray-oxidation process was investigated. The results show that by controlling the appropriate conditions, pure Co_3O_4 particles are prepared using cobalt chloride solution by spray-oxidation, and the reaction temperature has an important impact on Co_3O_4 particles purity, micro-structure morphology and crystallization during the process of spray-oxidation.

Key words: spray-oxidation; Co_3O_4 particles; characterization

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