

Chemistry

Effect of Microwave Synthesis Conditions on the Structure of Nickel Hydroxide Nanosheets

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Summary

Nickel hydroxide nanosheets are synthesized by a microwave-assisted hydrothermal reaction. This protocol demonstrates that the reaction temperature and time used for microwave synthesis affects the reaction yield, crystal structure, and local coordination environment.

Abstract

A protocol for rapid, microwave-assisted hydrothermal synthesis of nickel hydroxide nanosheets under mildly acidic conditions is presented, and the effect of reaction temperature and time on the material's structure is examined. All reaction conditions studied result in aggregates of layered α -Ni(OH)₂ nanosheets. The reaction temperature and time strongly influence the structure of the material and product yield. Synthesizing α -Ni(OH)₂ at higher temperatures increases the reaction yield, lowers the interlayer spacing, increases crystalline domain size, shifts the frequencies of interlayer anion vibrational modes, and lowers the pore diameter. Longer reaction times increase reaction yields and result in similar crystalline domain sizes. Monitoring the reaction pressure *in situ* shows that higher pressures are obtained at higher reaction temperatures. This microwave-assisted synthesis route provides a rapid, high-throughput, scalable process that can be applied to the synthesis and production of a variety of transition metal hydroxides used for numerous energy storage, catalytic, and other applications.

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cells⁴, water electrolyzers^{4,5,6,7,8,9}, supercapacitors⁴, photocatalysts⁴, anion exchangers¹⁰, and many other

Protocol

NOTE: The schematic overview of the microwave synthesis process is presented in **Figure 1**.

1. Microwave synthesis of α -Ni(OH)₂ nanosheets

1. Preparation of precursor solution

1. Prepare the precursor solution by mixing 15 mL of ultrapure water (≥ 18 M Ω -cm) and 105 mL of ethylene glycol.

Add 5.0 g of Ni(NO₃)₂ · 6 H₂O and 4.1 g of urea to the solution and cove.....

Representative Results

Influence of reaction temperature and time on the synthesis of α -Ni(OH)₂

Before the reaction, the precursor solution [Ni(NO₃)₂ · 6 H₂O, urea, ethylene glycol, and water] is a transparent green color with a pH of 4.41 ± 0.10 (**Figure 2A** and **Table 1**). The temperature of the microwave reaction (either 120 °C or 180 °C) influences the *in situ* reaction pressure and color of the sol.....

Discussion

Microwave synthesis provides a route to generate Ni(OH)₂ that is significantly faster (13-30 min reaction time) relative to conventional hydrothermal methods (typical reaction times of 4.5 h)³⁸. Using this mildly acidic microwave synthesis route to produce ultrathin α -Ni(OH)₂ nanosheets, it is observed that reaction time and temperature influence the reaction pH, yields, morphology, porosity, and structure of the resulting materials. Using an *in situ* reaction pr.....

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Center for Intelligent Materials Assembly, Award No. 2122041, for analysis of the reaction conditions.

Materials

Name	Company	Catalog Number	Comments
ATR-FTIR	Bruker		Tensor II FT-IR spectrometer equipped with a Harrick Scientific SplitPea ATR micro-sampling accessory
Bath sonicator	Fisher Scientific	15-337-409	--
Ethanol	VWR analytical	AC61509-0040	200 proof
Ethylene Glycol	VWR analytical	BDH1125-4LP	99% purity
Falcon Centrifuge tubes	VWR analytical	21008-940	50 mL
KimWipes	VWR analytical	21905-026	--
Lab Quest 2	Vernier	LABQ2	--
Microwave Reactor	Anton Parr	165741	Monowave 450
Ni(NO ₃) ₂ · 6 H ₂ O	Ward's Science	470301-856	Research lab grade
pH Probe	Vernier	PH-BTA	Calibrated vs standard pH solutions (pH= 4, 7, 11)
Porosometer	Micromeritics	--	ASAP 2020. Analysis software: Micromeritics, version 4.03
Powder x-ray diffractometer	Bruker		AXS Advanced Powder x-ray diffractometer; d-spacing, and crystallite size analyses were performed using Highscore XRD software, and crystal structures were created using VESTA 3 software.
Reaction vial	Anton Parr	82723	30 mL G30 wideneck, 20 mL max fill capacity
Reaction vial locking lid	Anton Parr	161724	G30 Snap Cap
Reaction vial PTFE septum	Anton Parr	161728	Wideneck
Scanning electron microscope	FEI	--	Helios Nanolab 400
Urea	VWR analytical	BDH4602-500G	ACS grade

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