

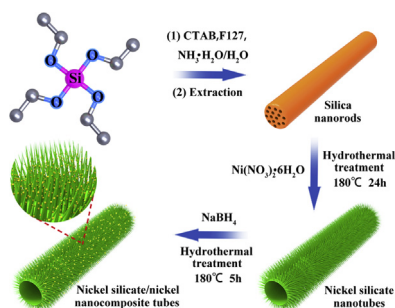
Regular Article

Self-template synthesis of nickel silicate and nickel silicate/nickel composite nanotubes and their applications in wastewater treatment

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GRAPHICAL ABSTRACT

Nickel silicate nanotubes (NiSNTs) prepared via a self-templating process, can serve as the superior candidates for efficiently adsorbing heavy metal ions and organic dyes in wastewater. Moreover, NiSNTs can further be transformed to NiSNTs/Ni composite nanotubes, exhibiting efficient and recyclable catalytic decomposition of organic pollutants.



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ABSTRACT

Hierarchical one-dimensional (1D) hollow nanostructures have attracted intense research attention due to their unique structures that may be capable of many promising applications. Here, we report a facile and effective strategy to prepare nickel silicate nanotubes (NiSNTs). Using this approach, mesoporous SiO₂ nanorods served as sacrificial templates for a simple hydrothermal process to prepare NiSNTs via a self-template route. The obtained NiSNTs preserve the tubular hierarchical morphology and hollow interior structures, which confer the NiSNTs with high surface areas and large pore volumes. These features point to NiSNTs as superior candidates for efficiently adsorbing heavy metal ions and organic dyes in wastewater. Moreover, NiSNTs can also serve as both supports and nickel sources, forming nickel silicate/nickel nanocomposite tubes (NiSNTs/Ni) via *in situ* reduction of NiSNTs under hydrothermal conditions. The addition of nickel precursors and surface pre-activation are therefore unnecessary. The NiSNTs/Ni exhibit efficient and recyclable catalytic properties when used for the reduction of 4-nitrophenol (4-NP). With a rational adaption, we believe this synthetic strategy can be extended to fabricate multi-functional one-dimensional hollow nanostructures with diverse compositions and morphologies, which may be useful for environmental protection, catalysis, energy and many other important domains.

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1. Introduction

Hollow micro/nanostructures with well-controlled sizes, morphologies, and compositions have attracted great attention due to their potential applications in catalysis, biomedicine, optics and electronics, energy storage and conversion, environmental protection, etc. [1–11]. As an important class of hollow micro/nanostructures, hollow 1D nanostructures, i.e., nanotubes, have recently become popular due to their low density, high surface-to-volume ratio and unique physicochemical features in relation to their elongated 1D structures that may be used for a rich variety of promising applications [12–14].

Several synthetic strategies including the well-known template methods [15,16], galvanic replacement reaction [17,18], Kirkendall effect [19], nonequilibrium heat treatment [20,21], and electrospinning techniques [22,23] have been developed to achieve nanotube structures. Among these strategies, the template methods have been widely agreed to be one of the most representative and straightforward ways in which the cavity sizes and the shapes of the voids can be easily controlled [24–28]. However, the template methods often suffer from intrinsic disadvantages, including the difficulty in coating the desirable materials onto template surfaces due to material incompatibility and complicated post-treatments for template removal. These inconveniences may obstruct their practical use. To overcome these inconveniences and retain the inherent convenience of the template method, self-template strategies have been derived and developed from the template methods to prepare hollow nanostructures. Unlike conventional templates employed in the template methods, the selected templates used in the self-template approach can serve not only as the core for the void structures but also as the consumable reactants for shell construction [3,29–31]. In this way, neither additional surface functionalization nor post-template removal is necessary [3,32]. As such, the self-template approach may provide a convenient and efficient avenue for fabricating nanotubes.

Silicate materials have attracted broad interest due to their low cost, excellent thermal and chemical stability, and environmentally benign nature, meaning that they have been applied for a wide variety of areas, including catalyst support, molecular sieving, adsorption and separation [22,33,34]. Recently, the synthesis of silicate colloidal particles with controllable hollow structures has become more attractive than synthesis of their counterpart solid particles because of prospective new applications and enhanced performance [33–36]. Among silicate hollow particles, nickel silicate nanotubes (NiSNTs) have garnered particular attention due to their unique one-dimensional structure, a large specific surface area, and a mesoporous volume, making them potentially useful as catalysts, adsorbents, drug carriers, and lithium battery anode support materials [23,36–38].

In principle, NiSNTs can be synthesized via template methods where nanorods/tubes are employed as the template and are subsequently removed to generate the cavity. The commonly applied templates are polymer nanorods and carbon nanotubes, which can be selectively removed after the formation of NiSNTs [23,36,38]. Despite the recent success in the preparation of NiSNTs using template methods, it is still worth noting that these methods suffer from a complicated process and limited template morphology selections. Thus, it is a significant challenge to develop simple template routes for preparing NiSNTs with tunable structures.

In this work, we develop a facile strategy for preparing of nickel silicate nanotubes (NiSNTs) by employing a self-template process in a hydrothermal environment. Using this method, mesoporous SiO₂ nanorods were prepared via ammonia-catalyzed hydrolyzation of tetraethyl orthosilicate (TEOS) with F127 and CTAB as the

binary surfactant templates and serving as the 1D sacrificial templates. NiSNTs are subsequently synthesized using a hydrothermal treatment in a Ni(NO₃)₂ aqueous solution consisting of SiO₂ nanorods, where post template removal is omitted and the structure of the NiSNTs is tunable using the experimental parameters. The NiSNTs possess tubular hierarchical hollow structures, high surface area, and a large pore volume, which allow them the efficient absorption of heavy metal ions and organic dyes from wastewater. Moreover, the *in situ* reduction of NiSNTs results in the formation of hierarchical Nickel silicate/Ni composite nanotubes (NiSNTs/Ni) that exhibit recyclable catalytic activity during decomposition of organic dyes in water.

2. Experimental section

2.1. Materials

All the chemicals were used as received and were of analytic grade unless otherwise noted. Triblock copolymer Pluronic F-127 (EO₁₀₀PO₇₀EO₁₀₀, EO = ethylene oxide, PO = propylene oxide) was purchased from Sigma-Aldrich. Tetraethyl orthosilicate (TEOS, ≥99.8%), hexadecyltrimethyl-ammonium bromide (CTAB, ≥99.8%), hydrochloric acid (HCl, 36.0–38.0%), nickel nitrate hexahydrate (Ni(NO₃)₂·6H₂O, ≥99.8%), sodium borohydride (NaBH₄, ≥98%), aqueous ammonia (28 wt% aqueous solution), and absolute ethanol were purchased from Sinopharm Chemical Reagent Co., Ltd. (China). Ultrapure water (>17 MΩ cm⁻¹) obtained from a GZY-P10 water system was used throughout experiments.

2.2. Synthesis of nickel silicate nanotubes

First, SiO₂ nanorods were synthesized using the reported procedures [12,39,40]. In brief, TEOS (4.8 mL) was added to a mixture comprising F127 (1.26 g), CTAB aqueous solution (0.04 M, 100 mL), and aqueous ammonia solution (2.5 wt%, 120 mL) using stirring. After 10 min, the stirring was stopped, and the mixture remained stationary for 6 h at room temperature. The mixture gradually turned cloudy. SiO₂ nanorods were collected by centrifugation and were washed with ethanol and deionized water three times. The surfactant molecules (F127 and CTAB) were removed by heating SiO₂ nanorods in an ethanol/hydrochloric acidic solution at 90 °C for 24 h. Finally, SiO₂ nanorods were washed with ethanol and dried in a vacuum oven at room temperature for 24 h.

Second, the NiSNTs were synthesized via a hydrothermal process using SiO₂ nanorods as the sacrificial templates. Typically, Ni(NO₃)₂·6H₂O (1.0 g) was added to a SiO₂ nanorod (0.1 g) aqueous dispersion (3.33 g/L, 30 mL) under sonication. Then, this dispersion was transferred into a 50-mL Teflon-lined stainless steel autoclave and was heated at 180 °C in for 24 h. After the autoclave cooled to room temperature, the NiSNTs were collected by centrifugation and washed with ethanol and ultrapure water several times before drying in a vacuum oven for 24 h. The dry NiSNTs were stored in a glass vial for further use.

2.3. Synthesis of nickel silicate/nickel composite nanotubes

Typically, a dispersion (15 mL) of NiSNTs (0.05 g) and NaBH₄ (0.15 g) was charged into a Teflon-lined stainless steel autoclave, which was heated to 180 °C for 5 h [38]. After the autoclave cooled to room temperature, the NiSNTs/Ni were collected by centrifugation and washed with ethanol and ultrapure water several times prior to drying in a vacuum oven for 24 h.

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