



Propylene oxide assisted sol–gel synthesis of zinc ferrite nanoparticles for solar fuel production

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Received 23 August 2015; received in revised form 9 October 2015; accepted 9 October 2015

Available online 23 October 2015

Abstract

This paper reports the synthesis of phase-pure Zn-ferrite nanoparticles with high specific surface area (SSA) via the propylene oxide (PO) assisted sol–gel method. For the synthesis of Zn-ferrite, metal precursors (ZnCl_2 and $\text{FeCl}_2 \cdot 4\text{H}_2\text{O}$) were first dissolved in ethanol, and then PO was added dropwise for the gel formation. The effects of a variety of synthesis parameters, such as the concentration of PO, the gel aging time, the calcination temperature, and the calcination dwell time, on the phase/chemical composition, SSA, porosity, crystallite size, and morphology of the Zn-ferrite were studied in detail. Different analytical techniques, such as powder X-ray diffraction (PXRD), BET surface area analyzer (BET), electron dispersive spectroscopy (EDS), scanning electron microscopy (SEM), and high resolution transmission electron microscopy (HR-TEM), were used for analyzing the Zn-ferrite samples. The acquired results indicate that the phase/chemical composition of the Zn-ferrite remains unchanged, irrespective of the variation in the experimental conditions. BET analysis further confirms that the SSA of Zn-ferrite increased due to the increase in the concentration of PO and decreased with the upsurge in the calcination temperature and dwell time. The crystallite size of Zn-ferrite was also observed to be higher when the calcination temperature and dwell time were increased. SEM and HR-TEM assessment verify the formation of Zn-ferrite nanoparticles via the sol–gel method employed during the study.

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Keywords: Zn-ferrite; Sol–gel method; Nanoparticles; Solar fuel; Propylene oxide

1. Introduction

Conversion of solar energy into chemical fuels, such as solar H_2 or solar syngas (a mixture of H_2 and CO for the production of liquid transportation fuels), via thermochemical H_2O and CO_2 splitting reaction is one of the favorable possibilities for the fulfillment of the future energy demand. In recent years, researchers have been working towards metal oxide (MO) based solar thermochemical cycles for the production of solar fuels. A variety of MO-based thermochemical cycles, such as zinc oxide cycle, tin oxide cycle, iron oxide cycle, doped iron oxide (ferrite) cycle, and ceria/doped ceria cycle, have been

tested for their performances in the solar H_2O and CO_2 splitting reaction [1–55].

The results of a literature survey indicate that, in recent years, researchers have primarily been focused on the non-volatile ferrite cycle. A range of ferrite materials, e.g., Ni-ferrite, Co-ferrite, Zn-ferrite, Mn-ferrite, Ni–Zn-ferrite, Ni–Mn-ferrite, and Ni–Sn-ferrite, were studied regarding their performance in solar thermochemical fuel production [36–53]. Most of these ferrite materials were synthesized using various approaches, including self-propagation high-temperature synthesis (SHS), solid state synthesis (SSS), oxidation of aqueous metal hydroxide suspension (OAMHS), aerosol spray pyrolysis (ASP), and the sol–gel method [36–54]. Recently, Bhosale et al. [37–39,43,45,47,48] reported sol–gel derived ferrites as being quite capable to produce H_2 via the solar H_2O splitting reaction. The quantities of H_2 reported in the case of sol–gel synthesized ferrites were observed to be higher

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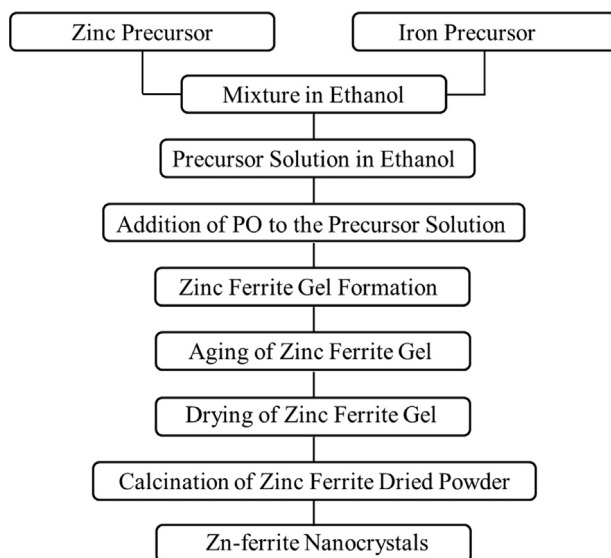


Fig. 1. Sol-gel route for the synthesis of Zn-ferrite.

compared to previously investigated ferrites prepared using SHS, SSS, etc.

Previous investigations reported utilization of Zn-ferrite prepared by ASP [51], SSS, and commercially purchased [55] for solar thermochemical cycles. However, the performance of sol-gel derived Zn-ferrite in the production of solar fuels has not been reported. This study reports the propylene-oxide assisted sol-gel synthesis of Zn-ferrite for solar thermal applications. Physico-chemical characterization of the sol-gel derived Zn-ferrite was performed using different characterization methods. The influence of a variety of synthesis parameters, such as concentration of PO, gel aging time, calcination temperature, and calcination dwell time, on the phase/chemical composition, specific surface area (SSA), porosity, crystallite size, and morphology of the synthesized Zn-ferrite was studied in detail.

2. Materials and methods

2.1. Materials

For the synthesis of Zn-ferrite via the sol-gel method, ZnCl_2 and $\text{FeCl}_2 \cdot 4\text{H}_2\text{O}$ were used as the metal precursors, ethanol ($\text{C}_2\text{H}_5\text{OH}$, 95%) was used as the solvent, and propylene oxide ($\text{CH}_3\text{CHCH}_2\text{O}$, 99%) was used as the gelation agent. All of these chemicals were purchased from Alfa Aesar and Sigma Aldrich and were used without any pre-treatment.

2.2. Sol-gel synthesis of Zn-ferrite

To synthesize Zn-ferrite, ZnCl_2 and $\text{FeCl}_2 \cdot 4\text{H}_2\text{O}$ were added in ethanol in the appropriate weight ratio (Zn: Fe = 1:2). The mixture of metal salts and ethanol was sonicated until a visibly clear solution was obtained. To this mixed solution, propylene oxide (PO) was added drop-wise as the gelation agent, and then the formation of gel was achieved. As-synthesized Zn-ferrite gel was aged for quite a few hours at

room temperature. After aging, the gel was dried at 100 °C for 2 h using a temperature controlled hot plate. The dried gel powder was further calcined at different temperatures (with different dwell times) in air by using a muffle furnace. The obtained calcined powder was stored in a dry atmosphere for further analysis. The sol-gel route employed in this study for the synthesis of the Zn-ferrite is shown in Fig. 1.

2.3. Zn-ferrite characterization

Calcined Zn-ferrite powder was characterized using different analytical methods to determine the phase/chemical composition, crystallite size, specific surface area (SSA), porosity, and particle morphology. A Panalytical XPert MPD/DY636 powder X-ray diffractometer with Cu-K α radiation ($\lambda = 0.15418$ nm, voltage = 45 kV, current = 20 mA, angular range = 20–80° 2θ , steps = 0.05° 2θ , and recording time = 5 s) was used to identify the phase composition and crystallite size of the derived Zn-ferrite. The crystallite sizes were calculated by using the Scherrer equation as follows:

$$\text{crystallite size} = \frac{K\lambda}{\beta \cos\theta} \quad (1)$$

K = dimensionless shape factor (value close to unity, typical value of 0.9)

λ = X-ray wavelength

β = line broadening at half the maximum intensity (FWHM), after subtracting the instrumental line broadening (rad)

θ = Bragg's angle

A BET surface area analyzer, ASAP 2420 from Micromeritics, was used to determine the specific surface area (SSA) and cumulative pore volume (i.e., porosity) (P_v) of the Zn-ferrite material via the adsorption/desorption isotherms obtained after degassing the powders at 200 °C for 12 h.

The material morphology and chemical composition of the derived Zn-ferrite were analyzed using a Zeiss Supra 55VP field-emission scanning electron microscope (SEM) equipped

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