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Selective preparation of nanorods and micro-octahedrons of Fe_2O_3 and their catalytic performances for thermal decomposition of ammonium perchlorate

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Abstract

Nanorods and micro-octahedrons of α-Fe_2O_3 were selectively synthesized through one-step hydrothermal treatment of iron(III) chloride in aqueous formamide solution for different time. The resulting samples were characterized with XRD, FE–SEM, TEM, and FT–IR. It was found that monodispersed nanorods were 10–25 nm in diameter and 50–100 nm in length, while the octahydrons were 100–400 nm in size. These α-Fe_2O_3 nanorods and micro-octahedrons exhibited quite different catalytic performances on thermal decomposition of ammonium perchlorate. The nanorods significantly reduced the decomposition temperature of ammonium perchlorate, but the micro-octahedrons did not. This study provides alternative choice of good burning rate catalysts for composite solid propellants in solid fueled rockets.
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Keywords: Iron oxide; Catalysis; Thermal decomposition; Ammonium Perchlorate

1. Introduction

The synthesis of oxide nanoparticles attracts more and more attention because these nanoparticles exhibit electrical, optical and magnetic properties that are different from their bulk counterparts. The electronic and magnetic properties of Fe_2O_3 nanoparticles are very interesting for the design of new electronic and optical devices and for use as pigments in surface coatings [1]. Moreover, the strong absorption of Fe_2O_3 in the visible range, as well as the low cost, has stimulated considerable interest in its use as photocatalyst and photoelectrode [2,3]. Iron oxides are widely applied in catalysis such as styrene synthesis [4], photocatalytic production of hydrogen and oxygen by water splitting [5], removal of carbon monoxide [6], selective oxidation of NH_3 and reduction of NO by NH_3 [7], reduction of aromatic nitro compounds with hydrazine hydrate [8], catalytic conversion of methane into aromatic hydrocarbons [9], thermal decomposition of ammonium perchlorate [10,11], and so forth. It is well known that the shape and size of catalysts play crucial roles on their catalytic performances. For instance, Wang and coworkers prepared flower-like and quasicubic α-Fe_2O_3 nanoparticles through a solvothermal process and compared catalytic performances of these two kinds of nanoparticles. They found that quasicubic nanoparticles catalyzed oxidation of almost 100% CO at a temperature of 230 °C, much lower than those of nanophases with flowerlike, hollow, or other forms of irregular external morphologies having various crystal planes exposed to the gas [12].

Iron oxides include α-Fe_2O_3, γ-Fe_2O_3 and Fe_3O_4. Among these iron oxides, α-Fe_2O_3 has the corundum structure, while the other two have the spinel structure. γ-Fe_2O_3 and Fe_3O_4 are metastable in the oxidative atmosphere. They will be oxidized to α-Fe_2O_3 by heating above 400 °C [11]. γ-Fe_2O_3 particles have cubic unit cells with both octahedrally and tetrahedrally coordinated Fe^{3+} sites (defect spinel structure) whereas the unit cell of α-Fe_2O_3 is hexagonal and contains only octrahedrally coordinated Fe^{3+} atoms (corundum structure) [12].

Various methods have been reported for the synthesis of iron oxide nanoparticles. These methods include reduction of iron salts in micelles [13,14], thermal reactions [15–17] using the electrochemical method [18], sonochemical decomposition of iron pentacarbonyl in solutions with certain polymers as protective agent [19–21], or in aqueous solutions without any

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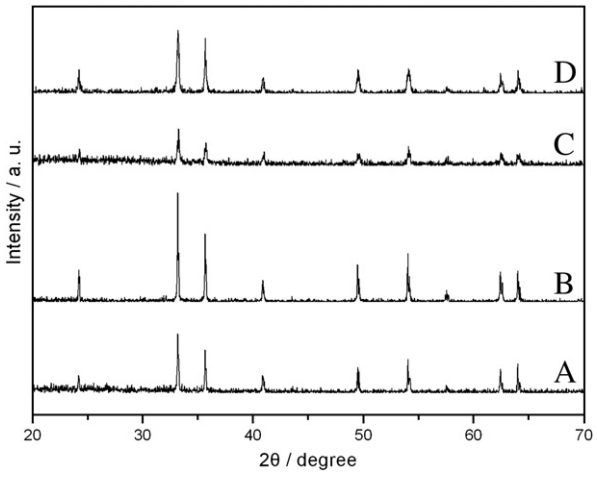

Fig. 1. XRD patterns of the resulting samples synthesized under different conditions. (A) 160 °C, 12 h, in the presence of formamide; (B) 160 °C, 24 h, in the presence of formamide; (C) 160 °C, 12 h, in the absence of formamide; (D) 160 °C, 24 h, in the absence of formamide.

surfactants[22], and so on. Recently, there are several reports on controlled synthesis of various α-Fe_2O_3 nano-microstructures [23–26]. In this work, we report the selective synthesis of nanorods and micro-octahedrons of α-Fe_2O_3 through one-step hydrothermal reactions.

Solid fueled rockets are used for space, ballistic, tactical and assist propulsion. Solid composite propellants in use today are mixtures of prepolymer (binder), aluminum fuel and oxidizer salts like ammonium perchlorate (AP). Burning rate catalysts are always of great interest in order to improve the ballistics of the rocket design. Fe_2O_3 is considered as one of potential burning-rate catalysts for composite solid propellants. Recently, it was found that the catalytic performance of nanometer-sized Fe_2O_3 particles was superior to that of micrometer-sized Fe_2O_3 particles on the thermal decomposition of AP [10,11]. In this study, we also compare catalytic performances of α-Fe_2O_3 nanorods and micro-octahedrons for thermal decomposition of ammonium perchlorate. To the best of our knowledge, this is the first example to report catalytic performances of these two kinds of α-Fe_2O_3.

2. Experimental

2.1. Synthesis

Synthesis of Fe_2O_3 nanorods. 0.540 g of $FeCl_3 \cdot 6H_2O$ (AR) was dissolved in 18 mL of 1 mol/L aqueous formamide solution. The resulting solution was transferred to a 22-ml Teflon-sealed autoclave and stored at 160 °C for 12 h, then was air-cooled to room temperature. The product (denoted as A) was washed several times with deionized water, and finally dried at 60 °C in a vacuum-oven.

Synthesis of Fe_2O_3 micro-octahedrons. The procedure for the synthesis of Fe_2O_3 micro-octahedrons was similar to that for Fe_2O_3 nanorods. The only difference is to increase reaction time from 12 h to 24 h. This sample was denoted as B.

For comparison, we synthesized two samples with the similar procedures to those for nanorods and micro-octahedrons in the absence of formamide. The resulting samples for 12 h and 24 h were denoted as C1 and C2, respectively.

2.2. Characterization

X-ray powder diffraction (XRD) patterns were obtained using a Philips MPD 18801 diffractometer using Cu-Kα radiation. Scanning electron microscopy (SEM) measurements were performed used a JSM-5600 SEM. Transmission electron microscopy (TEM) study was carried out on a Philips CM-120 electron microscopy instrument. The samples for TEM were prepared by dispersing the final powders in ethanol; the dispersion was then dropped on carbon-copper grids. Fourier transform infrared (FTIR) spectrum was detected by a Shimadzu IR-400 spectrometer as KBr methods.

2.3. Catalytic performance on thermal decomposition of AP

0.010 g of catalyst was added into 10 mL of ethanol. After sonication for 30 min, the suspension was mixed with 1.0 g of ammonium perchlorate. The new suspension was then sonicated

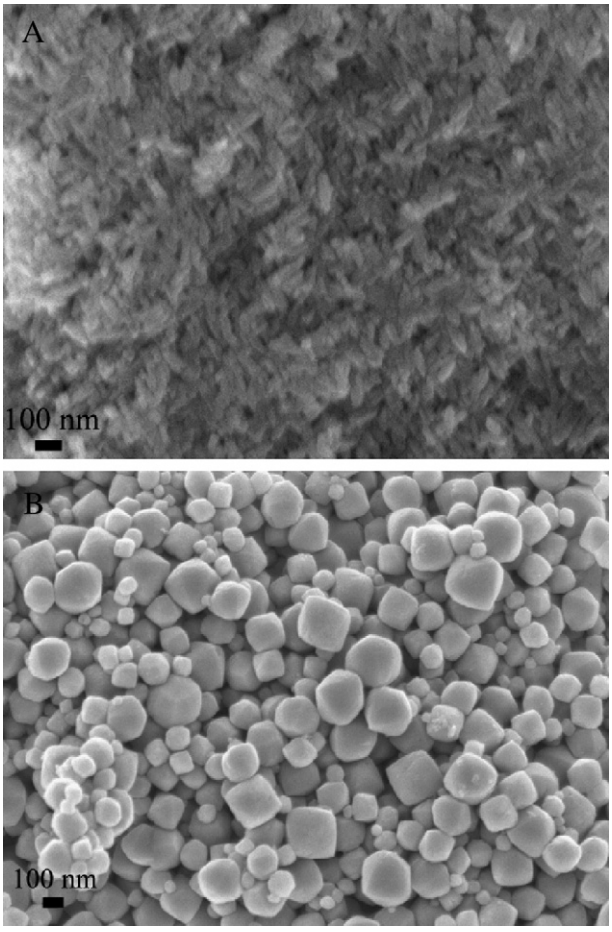

Fig. 2. SEM images of α-Fe_2O_3 samples synthesized in the presence of formamide. (A) 160 °C, 12 h, in the presence of formamide; (B) 160 °C, 24 h, in the presence of formamide.

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