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A novel approach for the preparation of nanosized Gd_2O_3 structure: The influence of surface force on the morphology of ball milled particles

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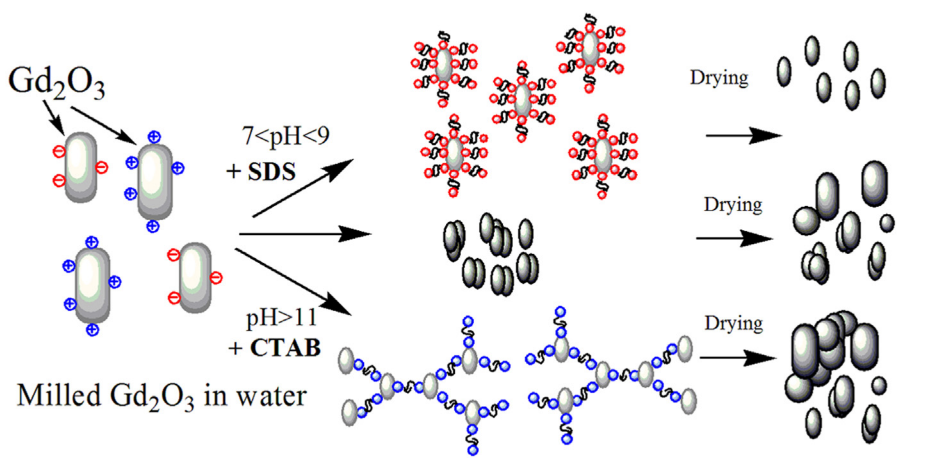
HIGHLIGHTS

- Without surfactants, milled Gd_2O_3 are rough (submicron) particles of various shapes.
- High sodium dodecyl sulfate (SDS) adsorption on milled grains forms Gd_2O_3 nanowires.
- Low cetyltrimethyl ammonium bromide (CTAB) adsorption forms coarser Gd_2O_3 particles.
- Surface force arising from surfactants adsorption varies the shape of milled Gd_2O_3.
- High SDS adsorbability stabilises the particles via steric and charge balance.

GRAPHICAL ABSTRACT



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ABSTRACT

This work investigates the effect of inter-particle forces arising from adsorbed typical cationic and anionic surfactants on the morphology of ball milled gadolinium oxide (Gd_2O_3). The experimental outcomes are interpreted in terms of stabilization and interaction mechanisms of fine washed Gd_2O_3 particles (size diameter <1 μm) in aqueous medium under the variation of surface forces arising of adsorbed surfactant. After ball milling and washing, the point of zero charge or isoelectric point (IEP) of Gd_2O_3 particles suspension is at pH 11 where its maximum yield stress is observed. Because of hydrophobic interaction, the maximum yield stress increases by 30 times by adsorbed sodium dodecyl sulfate (SDS) and its IEP shifts slightly to a lower pH. By cetyl trimethyl ammonium bromide (CTAB), the yield stress also increases by a much smaller extent (3 times) and shifts to a higher pH of ~12.5. Without surfactants, the microstructure of dried Gd_2O_3 displays the coarse particles of various shapes, i.e. rod, spherical and cubic shapes. This indicates that the milled particles remain agglomerated in dispersion. In the presence

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of adsorbed anionic SDS, the particles are refined together with numerous 2D nanowire or nano-rod particles at pH ~ 8. In contrast, coarser particles with absence of nano-rods are found when cationic CTAB is used to modify the Gd_2O_3 surface at a pH of about 12.5. The SDS-modified suspension exhibits a much higher yield stress, which results from finer particles in suspension. This is invoked from an organic shell formed by the high adsorbability of negatively charged heads of SDS into the bare positive charge density of the particle. The organic SDS shell prevents the fine particles from re-welding during the dispersing and annealing route. This work develops an inexpensive ball-milling approach with assisted SDS surfactant for mass production of nanosized Gd_2O_3 from bulky gadolinium material.

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1. Introduction

Recently, nanoscale and submicronsized structures of gadolinium (Gd) based compounds, especially Gd_2O_3, have attracted considerable attentions from both scientists and manufacturers due to their special optical and magnetic characteristics [1–3]. In application, they are used as fluorescent, luminescent materials for optical glass, plasma display panel of television tube and as radiation sensors and detectors [2,4–6]. Gd_2O_3 can also be used as a clean cooling source for magnetic refrigerators or as a data storage place of computer bubble memory owing to its excellent magneto caloric properties [7–9]. The most important applications of nanosized gadolinium oxide are in medical areas. Because of its safe properties in clinical uses, gadolinium oxides can be used as a gamma absorber and a source in cancer radiation therapy, magnetic resonance imaging contrast agents, magnetic targeting drug carrying, and recently proposed as radiation shielding material for X-ray diagnosis [10–18].

Wet chemical synthesis is the most popular liquid state method to synthesize nanosize Gd_2O_3 particles [19–24]. In this method, Gd compounds with solid state structure are generated from chemical reactions and the reaction yield is controlled by multiple factors such as the concentration and purity of reactants, pH value, temperature, pressure and so on. The product particle size is determined by the parameters that control the nuclei growth such as the external surfactants, thermal decomposition or hydrothermal supporting techniques [9,19–28]. Therefore, wet chemical synthesis is a complicated method with low yield, which prevents mass production. Mechanochemical approach employing solid state reaction during high energy ball milling process is also used to produce fine Gd_2O_3 particles. Tsuzuki, et al. successfully synthesised nano- and submicron-sized cubic and platelet Gd_2O_3 particles via the reactions between gadolinium chloride ($GdCl_3$) and sodium hydroxide (NaOH) or calcium oxide (CaO) powder followed by post heat treatment [29,30]. Unfortunately, there exist many by-products in the reaction from the stoichiometry such as NaCl, $CaCl_2$ or residual amount of reactants such as NaOH, $GdCl_3$, CaO, which result in impure substances of the final products [29,30]. In contrast, the top-down mechanical technique without chemical reactions, such as ball milling, is an effective and low cost method of producing relatively large quantity of fine particles from bulky brittle materials [31–35]. However, this approach has experienced difficultly in producing nanosized gadolinium compounds particles because it completely depends on the nature of material used including physical, mechanical and chemical characteristics [36–40].

Gadolinium oxide, being malleable and magnetic, is difficult to mill to nanoscale just simply by ball milling [41–45]. This work reports that an addition of inert NaCl can reduce the size of Gd_2O_3 by attrition and prevent the resulting refined particles from aggregation during the milling process. The inert NaCl could be removed by repetitively washing with distilled water, settling the flocculated particles out and decanting the supernatant NaCl solution. The control of particle-particle interaction and flocculation morphology by using specific polyelectrotrolyte additives at a distinct surface chemistry condition can stabilize ultrafine dispersion and also support the reproduction of ultrafine grained powder [46]. Although, many studies have used surfactants to control the nuclei growth from reaction, no study has incorporated milling process and colloidal phase study to form Gd_2O_3 nanopowder [19–24]. Anionic sodium dodecyl sulfate (SDS) and cationic cetyltrimethylamonium bromide (CTAB) polyelectrolyte, which are the most widely used in both fundamental scientific studies and industrial products, were employed in this work for investigating their influence on the morphology of Gd_2O_3 in aqueous medium. Additionally, a model is developed to clarify the variation of Gd_2O_3 morphology due to the effects of these surfactants.

2. Materials and method

2.1. Materials

Premilled gadolinium (III) oxide (Gd_2O_3) powder with a purity of 99.99% with size distribution of d_{10} = 0.28 μm, d_{50} = 4.34 μm and d_{90} = 10.6 μm, was purchased from Alfa Aesar (United Kingdom). Sodium chloride crystals (NaCl, 99%), sodium dodecyl sulfate (SDS, 99%), cetyltrimetyllammonium bromides (CTAB, 99%) and potassium hydroxide pellets (KOH) (85%) were supplied by Aldrich Sigma. Nitric acid solution HNO_3 (40%) was from Chem Alert.

2.2. Method

A mixture with 1:1.5 wt ratio of Gd_2O_3:NaCl was loaded together with harden steel balls (12.5 mm in diameter) in a SPEX 8000M mixer/mill model. The milling with the ball-to-powder weight ratio of 2.8:1 was performed for 70 min at room temperature. The washing procedure using deionized water was conducted repetitively for 4 times by dissolution of 100 g mixture in 1000 g water in a 2-L beaker. An amount of 2 g NaOH pellets was added for the following two reasons. First, NaOH at this dilute level provided mild washing of the particle surface from any adsorbed anionic impurities [47–50]. Second, the alkali pH of this solution enhanced the flocculation of the Gd_2O_3 particles as in this pH region the charge density of these particles is low. The upper water layer of cleaned slurry at the end of washing procedure was less than 0.5 mS/cm or the concentration of remained NaCl was less than 0.005 M [47]. To increase the particle concentration to solid volume fractions (ϕ_s) of 0.30, the washed slurry was separated in a Sigma 2–6 centrifuge (Germany) at 2000 rpm for 15 min. The solid concentration was determined by evaporating a small sample (2 g) of the slurry in an oven for 5 h at 110 °C

Distilled water was added into centrifuged pristine slurry to create 260 ml Gd_2O_3 suspension at solid volume fraction to 0.003 for zeta potential-pH characterization (Colloidal Dynamic Zeta Probe). A Branson sonifier was used to disperse this suspension for 1 min set at 70% amplitude prior to zeta potential measurements. The

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