



Azeotropic distillation-assisted preparation of nanoscale gamma-alumina powder from waste oil shale ash

Baichao An, Guijuan Ji, Wenying Wang, Shucaï Gan*, Jijing Xu, Guimei Gao, Guanghuan Li

College of Chemistry, Jilin University, 6 Ximinzhu street, Changchun 130026, PR China

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ABSTRACT

A combined process was proposed for the utilization of waste oil shale ash (OSA) in the production of gamma-alumina nanoparticles. The process consisted of two stages, leaching and sintering. The ultrasonic technique followed by a heterogeneous azeotropic distillation process in the presence of polyethylene glycol (PEG) was carried to ensure complete elimination of the residual water in the precipitate. The structural and morphological properties of the calcined nanocrystalline powders were characterized by X-ray diffractometer (XRD), transmission electron microscope (TEM), Brunauer–Emmett–Teller nitrogen-gas adsorption method (BET). The as-prepared precursor hydroxides were analyzed using thermogravimetric–differential thermal analysis (TG–DTA), Fourier transform infrared spectroscopy (FT-IR), and X-ray photoelectron spectroscopy (XPS). The results indicated that the gamma-alumina powder with a uniform particle is well dispersed and the particle size is 20–40 nm; the waste OSA can be utilized to produce gamma-alumina nanoparticles.

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1. Introduction

Today, there is a growing interest in developing low-cost energy resource in the world. As a possible alternative, oil shale, which is a natural kerogenous rock, has been used to generate electricity and extract oil for almost a century. A by-product fly ash from oil shale processing is OSA, which is considered as a serious environmental pollutant [1]. Therefore, it is necessary to develop a new approach to OSA management. So far, most research focused on the aspect of building materials [2–7] and adsorbent [8–16]. Syntheses of tobermorite [17] and calcium-alumino-silicate hydrate [18] have been also reported as possible ways for the utilization of OSA.

Nano-sized alumina has extensive potentiality for being used in chemical absorbent, gas sensor, catalysts, and ceramic raw material. Considerable effort has been put into develop wet chemical synthesis methods because they offer convenient routes for ultra-fine and high-purity alumina nanopowder. However, the alumina particles were often found to be agglomerated on drying and calcination. To solve this problem, various methods for the reduction of the particle size have been reported, including sonochemical synthesis [19,20], azeotropic distillation [21,22], and supercritical-water processing [23] and so on.

In this work, we aim to synthesize gamma-alumina nanopowder using OSA as alumina source via the ultrasonic technique in the

hydrolysis–condensation stage followed by azeotropic distillation. The dispersion mechanism of gamma-alumina nanopowder is also proposed based on the present experimental facts.

2. Materials and methods

2.1. Raw materials and reagents

Samples of OSA were collected from the factory in Huadian city of Jilin Province. The organic contents (loss-on-ignition, LOI) and metal contents of oil shale are shown in Table 1. The carbon dioxide gas, with a purity of 99.99%, was purchased from Changchun Xinguang Gas Manufacturing Co., Ltd. Other reagents were purchased from Beijing Chemical Reagent Research Institute. All chemical reagents were analytical grade.

2.2. Ultrasonic irradiation facility and parameters

The low powder ultrasonic radiation was performed in an ultrasonic cleaner (KQ-100DB, Kunshan Inc., China) that produced a 40 kHz ultrasound at its bottom. Bath capacity of the sonicator is 3 L. Its power can be changed from 40 to 100 W.

2.3. Experimental procedure

The preparation of the nanoscale alumina from OSA is schematically sketched in the flowchart of Fig. 1 and involves two stages.

* Corresponding author. Tel.: +86 431 88502259.

E-mail address: gansc@jlu.edu.cn (S. Gan).

Table 1
Chemical component of oil shale and oil shale ash.

Component	% Raw oil shale	% Raw oil shale ash
Silicon dioxide	31.68	63.58
Titanium dioxide	0.39	0.75
Ferric oxide	4.23	5.47
Aluminum oxide	10.77	16.69
Phosphorous pentoxide	0.26	0.26
Manganese oxide	0.05	0.13
Calcium oxide	1.63	5.12
Magnesium oxide	1.84	2.51
Potassium oxide	0.84	1.68
Sodium oxide	0.40	1.02
LOI	47.85	2.24

2.3.1. Leaching of aluminium

A porcelain boat, containing 20 g 120-mesh OSA and 8 g sodium chloride was heated at 700 °C for 3 h and then air-dried. Subsequently, the leaching experiments were performed in a 1000 mL three-necked flask heated in a hemispherical mantle kept constant within ± 0.5 °C and connected to a mechanical agitator, equipped with twin-bladed impeller and shaft coated by Teflon. For minimizing aqueous loss, a reflux condenser was applied. After 5 h, the leach liquor was separated from the residue by vacuum filtration. Then, 6 M sodium hydroxide solution was added to the leaching liquor to adjust the pH to 13. After filtration, the supersaturated sodium aluminate solution was obtained, and the aluminum concentration is shown in Table 2.

2.3.2. Synthesis of gamma-alumina nanoparticles

Approximately 0.6 g surfactant (polyethylene glycol, PEG) was added to the supersaturated sodium aluminate solution. To this solution, carbon dioxide was injected until the pH value of the mixture was 9. Under ultrasonic radiation (for 30 min, 60 °C), the reaction mixture was further aged about 11.5 h. The resulting white precipitate was recovered and washed with anhydrous ethanol several times to obtain the as-synthesized product (marked as S₁).

Table 2
Chemical composition of leaching and purified solution of elements.

Element	Leaching solution of OSA ($\mu\text{g mL}^{-1}$)	Purified solution of OSA ($\mu\text{g mL}^{-1}$)
Aluminum	985.400	983.900
Calcium	10.170	1.077
Manganese	37.140	7.435
Iron	1.942	0.000
Titanium	0.131	0.000

Subsequently, S₁ was transferred into a rockered flask containing 30 mL of n-butanol. After distilling at 117 °C (the boiling point of n-butanol) for 35 min, the remaining n-butanol was removed through oven-drying (100 °C for 12 h) to obtain loose powder (marked as S₂). This powder was calcined at 550 °C for 2 h to obtain nano-sized gamma-alumina powder.

2.4. Characterization

The chemical compositions of the OSA were determined by X-ray fluorescence (XRF) analysis (PW1404/10, Philips, Holland). The mineral leachates and their purified solution were analyzed using inductively coupled plasma mass spectrometry (ICP-MS), model X Series II, USA. X-ray diffraction (XRD) analysis for powders was carried out by D/Max-IIIC (Rigaku, Japan) with Cu K α radiation. Powder morphology and dispersity were observed on transmission electron microscope (TEM, JEM-2000EX, Japan). The N₂ adsorption measurement was examined by a surface analyzer (ASAP 2010, USA) and the BET surface area, and the pore volumes were calculated. The pore size distributions were estimated by the Barrette–Joynere–Halenda (BJH) method. Thermogravimetric–differential thermal analysis (TG–DTA) curves of the samples were recorded on a TG-8120 (Rigaku, Japan) using alpha-alumina as the standard materials. Infrared spectra of the samples were investigated on a FT-IR (Nicolet Impact 410, USA) by

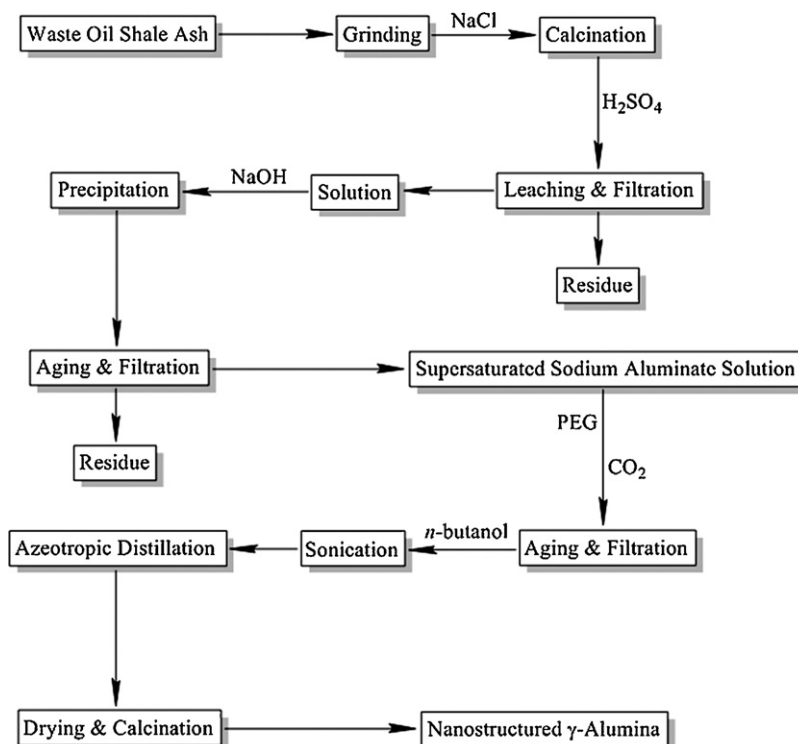


Fig. 1. Flowchart for the synthesis of gamma-alumina nanoparticle.

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