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Preparation of size-controlled tungsten oxide nanoparticles and evaluation of their adsorption performance

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ABSTRACT

The present study investigated the effects of particle size on the adsorption performance of tungsten oxide nanoparticles. Nanoparticles 18–73 nm in diameter were prepared by evaporation of bulk tungsten oxide particles using a flame spray process. Annealing plasma-made tungsten oxide nanoparticles produced particles with diameters of 7–19 nm. The mechanism of nanoparticle formation for each synthetic route was examined. The low-cost, solid-fed flame process readily produced highly crystalline tungsten oxide nanoparticles with controllable size and a remarkably high adsorption capability. These nanoparticles are comparable to those prepared using the more expensive plasma process.

1. Introduction

Tungsten oxide (WO_3) is an n-type semiconductor metal oxide with an optical band gap of approximately 2.5 eV, and thus is light-activated by irradiation with visible light at a wavelength of approximately 500 nm [1]. Due to its distinctive photocatalytic, electrochromic, and photochromic properties, tungsten oxide is widely used in environmentally friendly and energy-renewal applications, such as solar-powered water splitting, smart-windows, gas-sensing, and dye photodegradation [2–5]. Optimal performance of these technologies is dependent on tungsten oxide surface adsorption.

A better understanding of surface adsorption on nanosized tungsten oxide particles might lead to improved applications, especially in the fields of gas-sensing and catalyst activity. Compared with the bulk form, tungsten oxide nanoparticles exhibit considerably enhanced properties. For example, electrochromic performance is enhanced by reduction of particle size, which allows for fast metal-ion-insertion kinetics. In addition, the greater surface area of nanosized tungsten oxide allows for greater adsorption and light harvesting, which might enhance photocatalytic performance. Meanwhile, highly crystalline tungsten oxide nanoparticles are valuable because amorphous tungsten oxide suffers from high dissolution in acidic solutions, which limits its use in applications despite superior photochromic properties [6,7]. Moreover, controlling the size of tungsten oxide nanoparticles enhances visible light photodegradation, which is suitable for indoor photodegradation or when ultraviolet radiation is unavailable [8,9]. For this reason, efficient methods that allow preparation of size-controlled tungsten oxide nanoparticles are desirable.

Currently, several methods are available for the preparation of tungsten oxide nanoparticles: combustion synthesis, thermal evaporation, hydrothermal processes, and sol–gel [10–12]. However, these methods do not offer the advantage of controllable size, which is required for investigation of the size-dependence of tungsten oxide nanoparticle adsorption performance. In addition, these processes are energy-intensive and time-consuming, which limit their use in industrial applications.

A potential alternative method for the synthesis of nanoparticles with controllable size, morphology and crystallinity is the flame spray technique. Using this process, our group has successfully prepared nanoparticles with enhanced properties from various materials, including yttrium aluminum garnet, barium titanate, and yttria synthesized from liquid precursors [13–15]. In addition, we also prepared non-crystalline silica nanoparticles via solid evaporation, which is also known as solid-fed flame synthesis [16]. In this route, bulk materials, such as micron- or submicron-sized particles, are used as the nanoparticle precursor, which offers tremendous advantages, such as reduced material and process costs, short-processing time, and continuous operation.

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Hence, the primary purpose of the present study was to prepare crystalline tungsten oxide nanoparticles of various sizes via a solid-fed flame process to investigate the size-dependence of adsorption. Nanoparticle size and crystallinity were controlled by manipulation of either the carrier- or the fuel-gas flow rate. In addition, commercially available tungsten oxide nanoparticles that were produced using a plasma process were used as the raw material. The commercially available nanoparticles were annealed in a furnace at various temperatures for the preparation of nanoparticles less than 20 nm in diameter. The effects of particle size on adsorption performance were evaluated.

2. Experimental

2.1. Preparation of nanoparticles with controlled sizes

The experimental setup used for the flame process, depicted in Fig. 1, has been described in detail elsewhere [16]. In the present study, we used methane flow rates in the range of 0.5–2.0 L/min with an oxygen–methane volume ratio of 2.5 to ensure complete combustion in the flame. Oxygen was used as the carrier gas and the flow rate was varied between 2 and 10 L/min. A commercially available tungsten oxide powder with a mean particle size, d_p, of 716 nm and a deviation, σ_p, of 1.5 (Nittan Co., Japan) was used as the precursor—yellow in color, non-spherical in morphology and used with no additional treatment. The powder was fed into the flame reactor using an RGB-1000 solid particle disperser (Palas GmbH., Germany) to produce size-controlled nanoparticles via the solid evaporation route [16]. The plasma-synthesized precursor was annealed using a furnace (KDF 1700, Denko, Japan). Tungsten oxide nanoparticles with a diameter of 7 nm (σ_p of 1.3), synthesized using the plasma method, were annealed at various

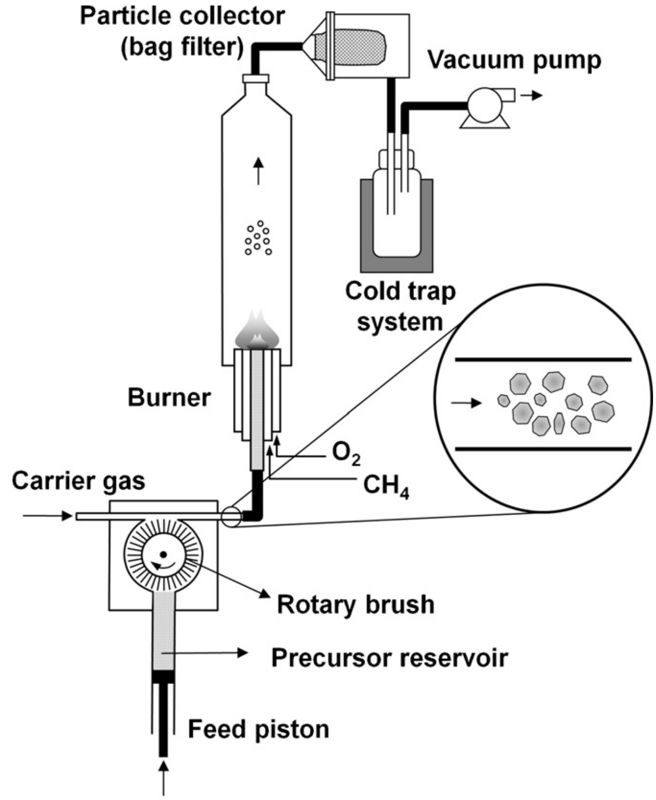

Fig. 1. Experimental setup for the preparation of tungsten oxide nanoparticles via a flame-based, solid evaporation route.

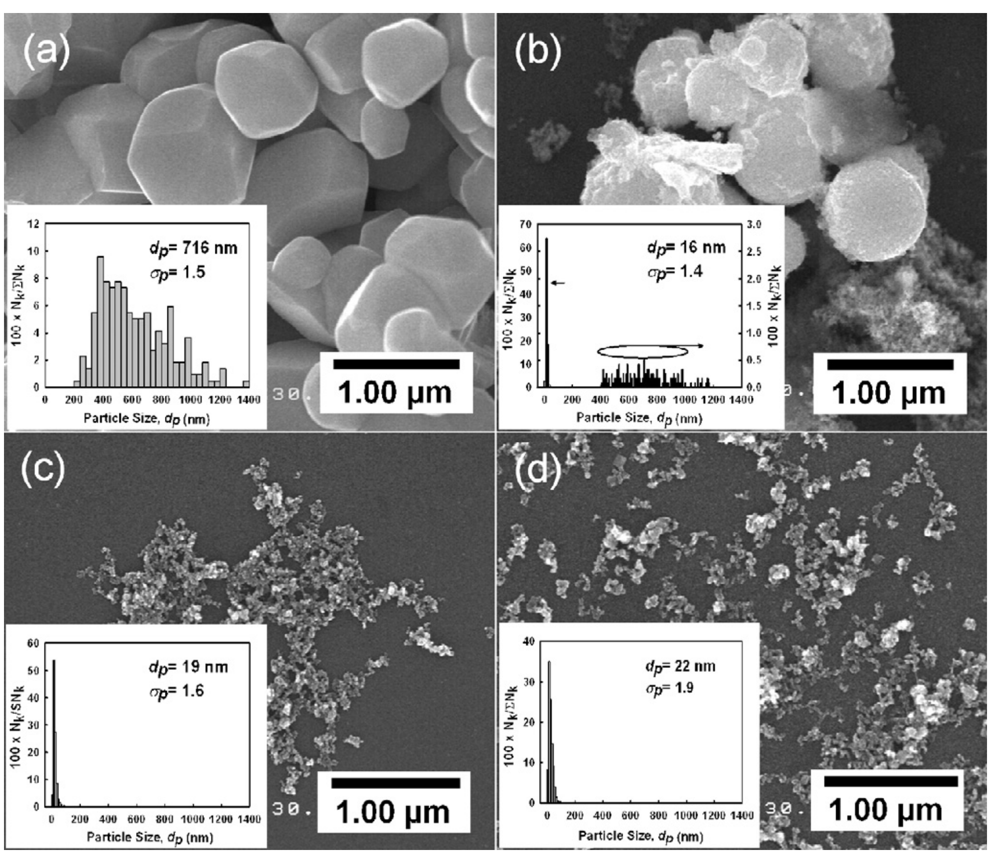

Fig. 2. FE-SEM images of tungsten oxide nanoparticles (a) initial precursor with a size of 716 nm. Tungsten oxide nanoparticles prepared at carrier gas flow rate of 10 L/min and methane flow rates of (b) 0.5 L/min, (c) 0.8 L/min, or (d) 1.0 L/min.