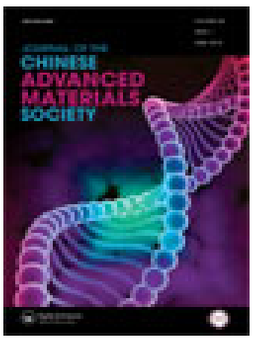

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Ultrafine yttria nanoparticles: synthesis and characterization

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Yttrium oxide nanoparticles (Y_2O_3) have been synthesized by a low temperature co-precipitation method. The precursor material used in this research was yttrium nitrate hexahydrate (as a basic material), ammonium hydroxide (as a precipitating agent) without surfactant. The resultant precipitate $Y(OH)_3$ was washed with deionized water several times and then heat treated at 450 °C for 1 hr. The prepared Y_2O_3 nanoparticles were characterized by TG-DTA, XRD, dynamic light scattering, Zetasizer and TEM. From the results obtained, Y_2O_3 ultrafine nanoparticles with particle size of 7.78 nm were successfully prepared as promise for wide range of applications.

Keywords: yttrium oxide nanoparticles; co-precipitation; ultrafine; characterization

1. Introduction

Despite the broad role of oxidative stress as causative agent of many diseases, medicine has been limited in its development of treatments that counteract free radical damage. In contrast, in the field of engineering, considerable effort has been developed to counter the effects of oxidative stress at the materials science level what is called nanotechnology-based therapeutical treatments for oxidative stress-induced diseases.[1] Nanotechnology can be defined as design, characterization, production and application of structures, devices and systems by controlling shape and size at nanometer scale or creation of materials and devices (as rings or caps) by controlling the size of material.[2] The most studied nanoparticle redox reagents at the cellular level are oxide nanoparticles (particularly cerium and yttrium oxide), fullerenes and carbon nanotubes.

Cerium oxide or yttrium oxide-based nanoparicles possess a number of advantages over other antioxidants; first, these nanoparticles act as catalysts to mimic the superoxide dismutase activity. Second, these nanopaticles are not consumed as they detoxify free radicals because they reconstitute their catalytic function by moving spontaneously between oxidized and reduced states. As result, they remain resident in the tissue and active for extended periods of the time. Third, when nanoparticles administered systemically, they cross the blood brain barrier.[3] Also, it was concluded that the supplementation of neuronal cultures with a single dose of the ceria nanoparticles has a significant synergistic effect in a realistic model system of spinal cord injury.[4,5] Yttrium oxide is noteworthy because the free energy of oxide formation from elemental yttrium is among the highest known.[6] Yttrium

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oxide is one of the 17 rare earth oxides and it exists in three structural phases such as cubic, hexagonal and monoclinic. The cubic phase of the yttria is highly stable at ambient conditions and it withstands even at high temperature because it has a melting temperature around 2400 °C. Most of the properties of yttria depend on the crystalline structure of the material. [7] Yttria nanoparticles can be synthesized by different methods such as sol-gel,[8] hydrothermal,[9]combustion [10]and precipitation method.[11,12] Among these methods, precipitation has been verified to be effective method for synthesis of yttria powders with favorable properties due to its feasibility, facility and green.[13] The objective of this study is the synthesis of ultrafine Y_2O_3 nanoparticles by low temperature co-precipitation method.

2. Material and methods

2.1. Preparation

Yttria nanoparticles were synthesized using co-precipitation method using yttrium nitrate hexahydrate ($Y(NO_3)_3.6H_2O$) as precursor material and ammonium hydroxide (NH_4OH) as precipitating agents. Initially, 8M of $Y(NO_3)_3{\cdot}6H_2O$ in 500 ml of distilled water was prepared and NH_4OH solution was added drop wise with stirring (1000 rpm) after reaching pH of 10.5 for 1 hr at room temperature. The precipitate was washed with deionized water several times to remove impurities and collected using high-speed centrifuge then dried at 75 °C till dryness. Finally, the precipitate precursor was annealed at 450 °C /1 hr. Figure 1 shows a schematic illustration of the synthesis method.

2.2. Characterization

Thermogravimetrical studies of the precursor material was performed with SI EXSTAR 6000 TG/DTA 6300 instrument between 25 °C and 800 °C with a heating rate of 10 °C min^{-1} in a nitrogen atmosphere. Phase identification and crystalline structure of as-prepared and annealed powders were performed by using X-Ray Diffractometer, XRD (SHIMADZU, XRD-6000) with Cu Kα radiation. The morphological investigation of annealed yttria nanoparticles was carried out by HRTEM (JEM-2100, JEOL electron microscope). Surface charge (Zetapotential) and particle size distribution were measured using Zetasizer and dynamic light scattering (DLS), respectively.

3. Results and discussion

3.1. TGA/DTA studies

Thermal decomposition process of the as-prepared precipitate precursor ($Y(OH)_3$) was studied by simultaneous DTA-TGA analysis and the results are shown in Figure 2. The broad endothermic peak centered at 324.06 °C was assigned to the removal of molecular water. The exothermic peak at 598.16 °C was caused by the decomposition of yttrium hydroxide and crystallization of yttrium oxide (Y_2O_3). It was found that thermal decomposition of the hydroxide precipitate into yttrium oxide was almost completed at~ 700 °C since there was no significant weight loss at temperature higher than 700 °C.[13]

3.2. XRD studies

Figure 3 showed the XRD pattern of YNPs with cubic phase and crystalline size of 5 nm. Although it was mentioned that few numbers of $Y(OH)_3$ peaks in addition to the Y_2O_3 peaks are obtained in as-prepared nanocrystalline yttria prepared by co-precipitation

Figure 1. Synthesis of Y2O3 nanoparticles by chemical precipitation.

method using NaOH as precipitating agent and those peaks transformed to cubic yttrium oxide when annealed at 500 °C [8], it was reported that the precursor transforms completely to cubic yttria at 600 °C.[14] Our research was able to prepare yttrium oxide nanoparticles at annealing temperature 450 °C. The crystalline size of samples, dXRD, was estimated by applying full-width-half-maximum of characteristic peak to

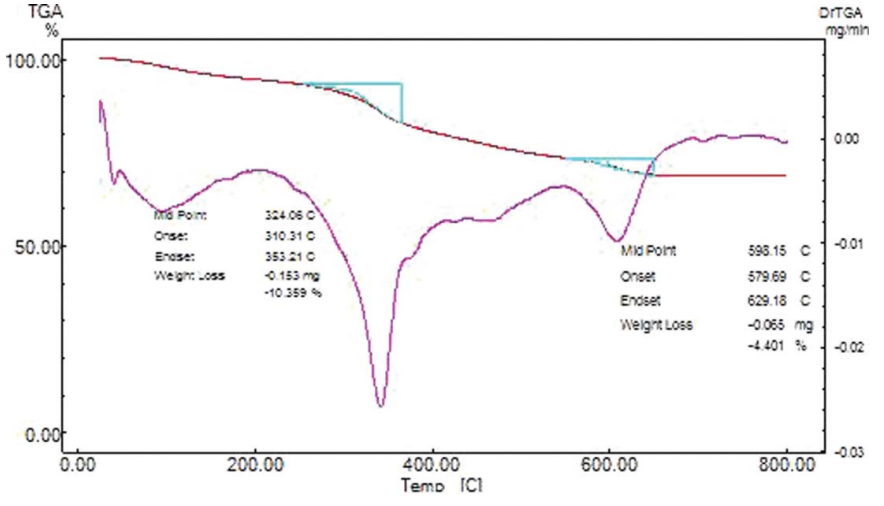

Figure 2. DTA-TGA analysis of the yttria precursor.

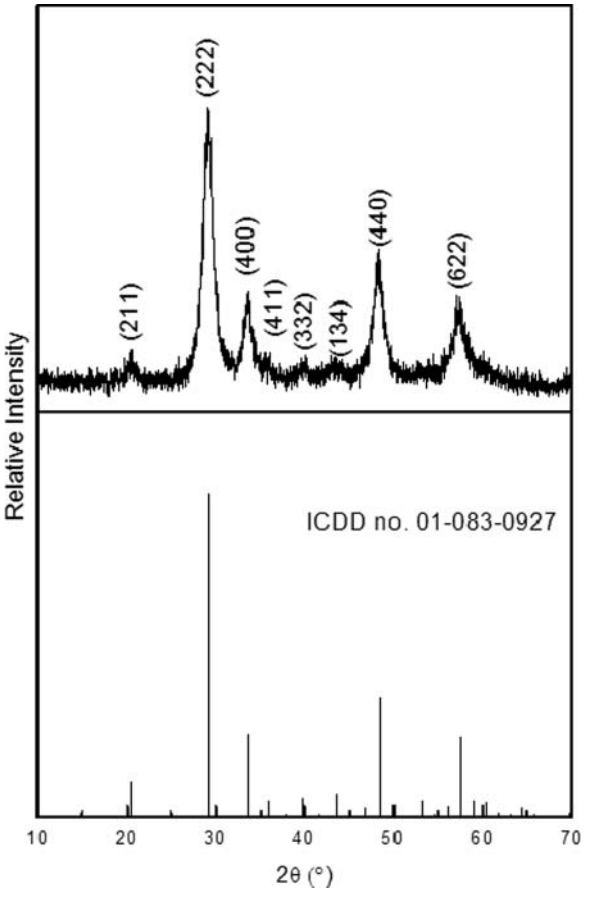

Figure 3. XRD pattern of yttria nanoparticles (YNPs)

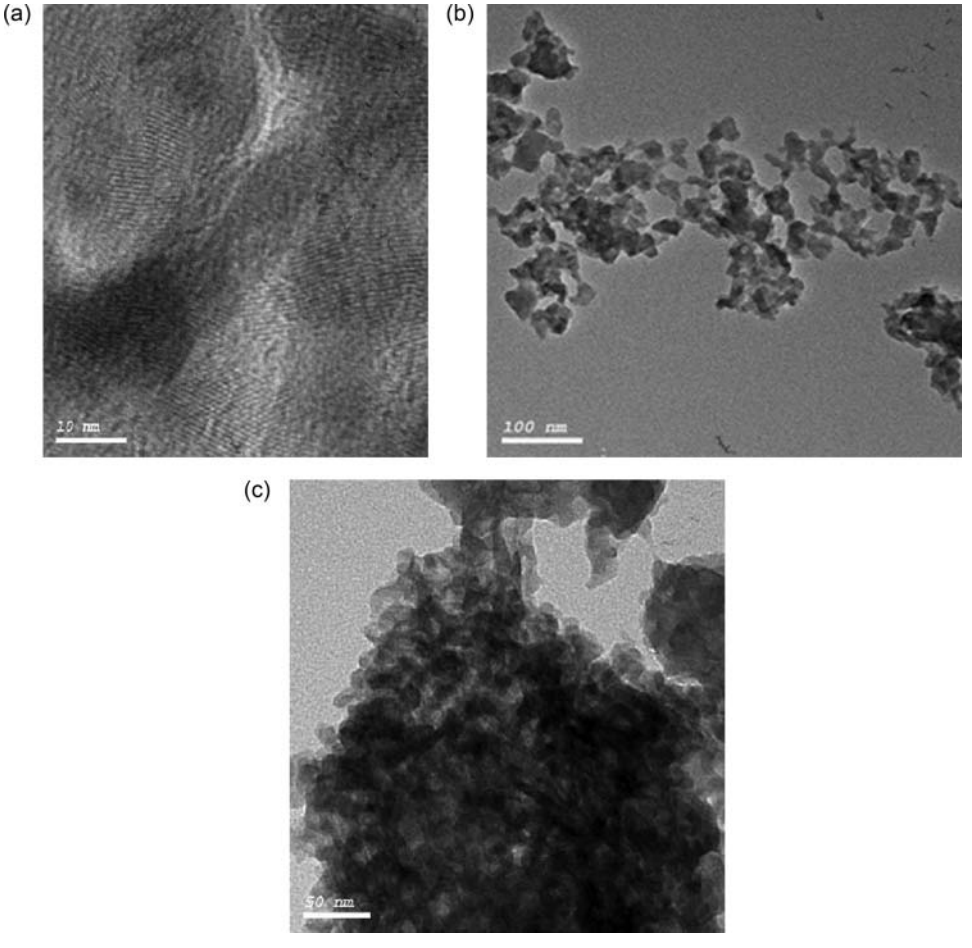

Figure 4. (a) The TEM image shows crystallinity of the yttria nanoparticles annealed at 450 °C. (b) The TEM image shows morphological shape of the yttria nanoparticles. (c) The TEM image shows high degree of agglomeration of the yttria nanoparticles.

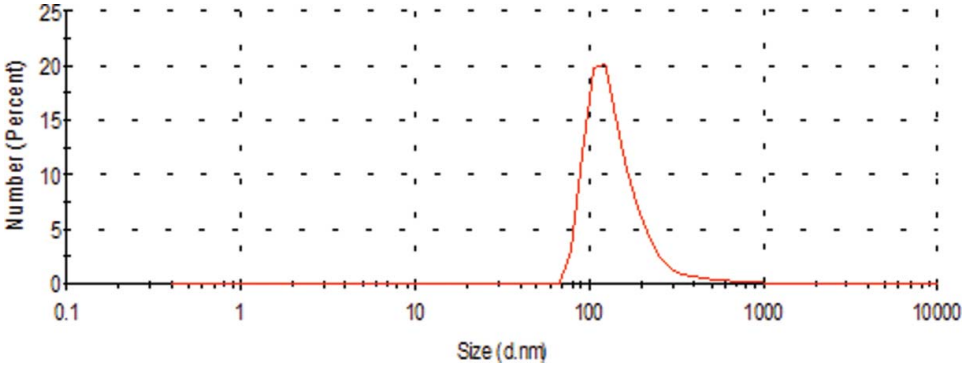

Figure 5. Pattern of DLS of yttria nanoparticles.

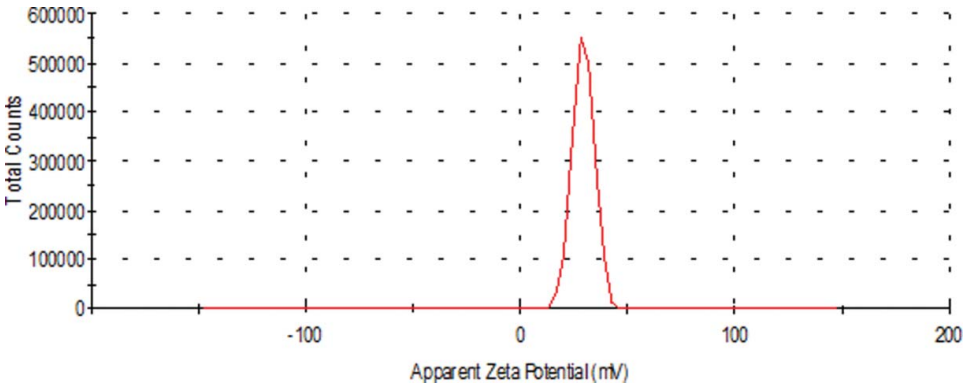

Figure 6. Pattern of Zetapotential of yttria nanoparticles.

Scherrer equation [15]

$$\mathrm{dXRD} = \frac{0.9\lambda}{\beta \cos\theta}$$

where λ is the wave length (1.5418 Å) of the incident X-ray, θ is the diffraction angle and β is the width of the diffraction line at half intensity, in radians.

3.3. Morphological characters

TEM images of prepared sample showed crystalline spherical shape nanoparticles with size 7.78 nm as shown in Figure 4(a) and 4(b). The results of DLS showed that the average particle size is 149.5 nm with surface charge of +29.4 (Figures 5 and 6). The sample showed high degree of agglomeration as shown in Figure 4 (c); that is, may refer to small size of particles and several attempts will be done to reduce agglomeration of yttria nanoparticles and enhance stability of particles over time.

4. Conclusion

Chemical precipitation of Y_2O_3 nanoparticles using yttrium nitrate hexahydrate and ammonium hydroxide has been considered. From the TGA/DTA results, it is clear that all the organics present in the precursor were released around 598.16 °C but the annealing temperature was reduced to 450 °C to reduce agglomeration approved by DLS results of average particle size 149.5 nm. The XRD data obtained on Y_2O_3 powder is in good agreement with the standard reported data. TEM results showed that Y_2O_3 ultrafine nanoparticles with particle size of 7.78 nm were successfully prepared as a promise for wide range of biomedical applications. In the literature, it is shown that yttria nanoparticles are toxic, unless they can have antioxidant properties that promote cell survival under conditions of oxidative stress. More catalytic, cytotoxicological investigations as well as molecular engineering of these nanoparticles for therapeutic purposes will be conducted soon.

Disclosure statement

No potential conflict of interest was reported by the authors.

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