

# Succinic acid as a precursor for synthesis of nano cerium oxide

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## **Abstract:**

Applying succinic acid as a versatile bidentate ligand to coordinate to cerium, resulted to a complex. The obtained crystalline complex was characterized by IR and melting point, then calcined at a certain temperature which was found by TGA method. Since in the first step, altering organic ligand type will affect on shape of precursor and as a result on the nano oxide, and in the next step, calcination time and temperature results in different morphologies of cerium oxide, we examine all these changes and conclude the optimum conditions and the best ligand to be succinic acid. Characterization of the nanorods of CeO<sub>2</sub> was performed by XRD, SEM and IR. Cerium oxide in nano-scale has many applications such as oxygen reservation capacity, conductivity, high UV absorption, high hardness, catalysis, fuel cells, sensors.

**Keywords:** Succinic acid, Cerium oxide, Nanorods

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## ***1. Introduction***

Ceria, CeO<sub>2</sub>, is a well-known functional rare earth oxide and nanostructured ceria has been extensively used in many areas including high-storage capacitor devices, buffer layer for conductors, fuel cells, polishing materials, UV blocks and optical devices [1-3]. In addition, CeO<sub>2</sub> has been widely used in automotive catalysts [1-2] due to its unique characteristics of facile Ce(IV) and Ce(III) switching. Stimulated by both their promising application and their fantastic properties, much attention has been directed to the controlled synthesis of CeO<sub>2</sub> nanostructured materials. Over the past few years, a remarkable process has been developed for the synthesis of CeO<sub>2</sub> with different morphologies and sizes due to its size/shape-dependent properties.

The physical/chemical properties of cerium oxides are strongly dependent on their microstructures, including size, morphology, and specific surface area [3]; therefore, the preparation of nanocrystalline ceria has been drawing much attention by the material scientists and chemists. A variety of chemical techniques have been utilized to prepare ceria nanomaterials, including hydrothermal [4-5], microemulsion, precipitation, sol-gel, and solvothermal synthesis [6-7].

Recently, Taniguchi et al. reported the synthesis of organophilic CeO<sub>2</sub> nanoparticles by a single step reaction of cerium oleate complex with NH<sub>4</sub>OH [8]. The reaction was carried out at room temperature in an aqueous medium. The authors claimed that the oleate links to the surface of CeO<sub>2</sub> particles by chemical bonding and render the particles' surface an organophilic nature.

The same research group performed supercritical hydrothermal synthesis of hydrophilic polymer-modified CeO<sub>2</sub> nanoparticles.

## **2. Experimental**

## ***2.1 Materials***

All reagents used in this work were of analytical grade, purchased from Merck Chemical Co. and used without further purification.

## ***2.2 Synthesis method***

Cerium nitrate hexahydrate and cerium chloride heptahydrate was used as the metal source. In a typical procedure, 1 mmol of  $\text{Ce}(\text{NO}_3)_3 \cdot 6\text{H}_2\text{O}$  was dissolved in deionized water under stirring to form a homogeneous solution. Subsequently, 1 mmol of succinic acid was dissolved in 30 ml of deionized water with a few drops 0.01 M NaOH solution. The obtained solution was introduced into the above homogeneous solution under continuous stirring. After 30 minutes, the solution was transferred into a Teflon-lined stainless steel autoclave, sealed, and maintained for 36 h. The supernatant liquid was discarded and the remaining product was washed with deionized water and ethanol in sequence several times, and then separated by centrifugation. and then the solution was poured into a Teflon-lined autoclave. The product was dried in a vacuum oven.

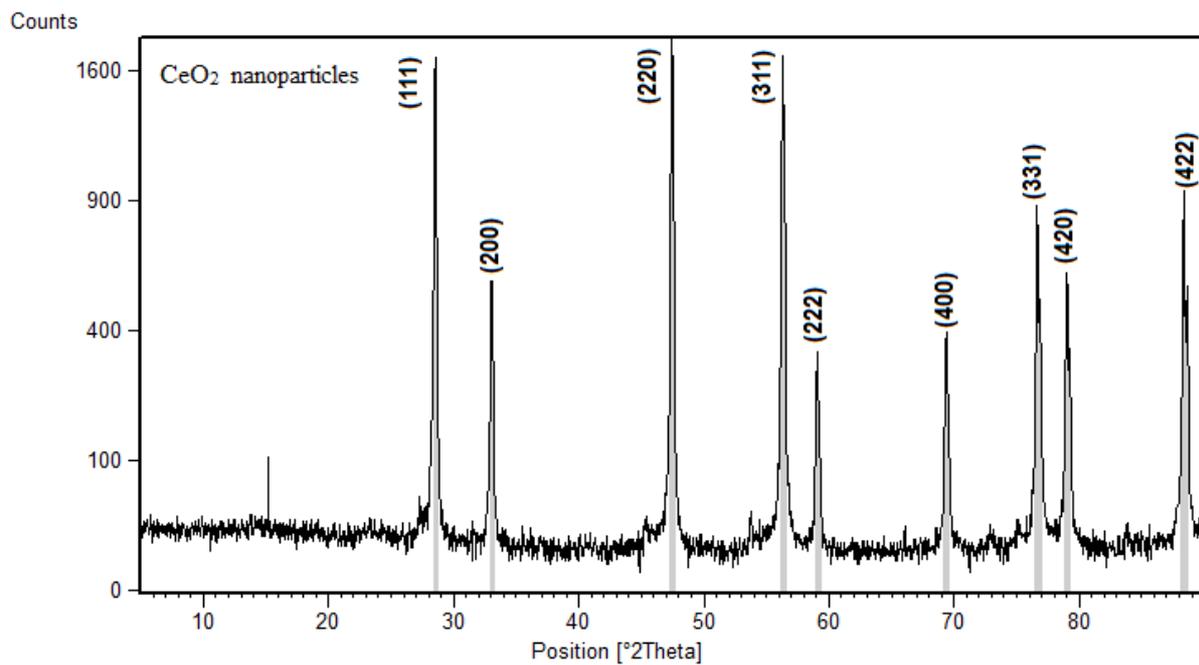
## ***2.3 Characterization***

The powder X-ray diffraction (XRD) patterns of products were obtained on a JEOL diffractometer with monochromatized  $\text{CuK}\alpha$  radiation ( $\lambda = 1.5418 \text{ \AA}$ ). Fourier transform infrared (FT-IR) spectra were recorded on a Shimadzu-8400S spectrometer in the range of  $400\text{--}4000 \text{ cm}^{-1}$  using KBr pellets. Scanning electron microscopy (SEM) images were taken on a Philips XL-30 with gold coating and finally thermogravimetric analysis (TGA) were on a 931 TA Instruments analyzer.

## **3. Results and discussion**

### 3.1 XRD pattern

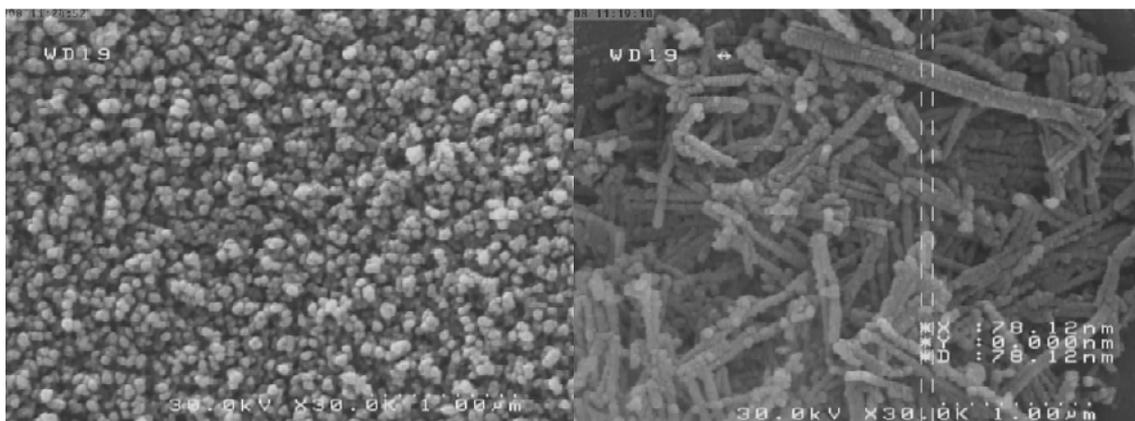
Chemical compositions of generated nanoparticles are verified by powder X-ray diffraction analysis. Figure 1a, b shows the XRD pattern for the fcc  $\text{CeO}_2$ . Diffraction peaks along (111), (200), (220), (311) and (222) planes confirm the cubic fluorite structure (JCPDS:34-0394) of  $\text{CeO}_2$ .



**Fig. 1** The XRD pattern of the obtained  $\text{CeO}_2$  particles

### 3.2 SEM studies

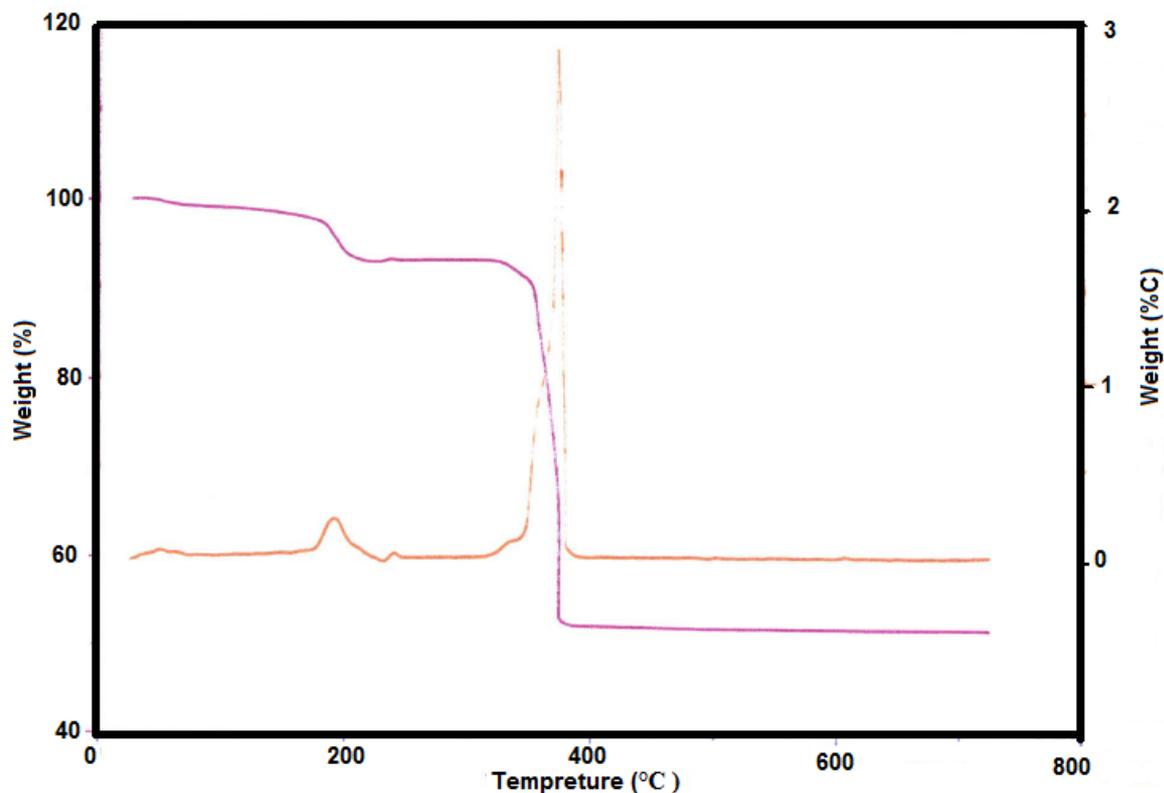
The morphological studies of the synthesized cerium oxide have been carried out by scanning electron microscopy. Fig. 2 displays the SEM image of the product. As it can be observed, the product is consisted of agglomerated nanoparticles constructing nanostructures in the range of 1-100nm.



**Fig. 2** The SEM image of the obtained CeO<sub>2</sub> particles

### **3.3 TGA**

Thermal behavior of the prepared CeO<sub>2</sub> nanomaterial was monitored by thermogravimetric analyses. As can be seen in Fig. 3, the TGA curve shows a two step process and there is 6.7% weight loss below 200 °C due to the removal of associated water which is in good agreement with the results of FT-IR spectroscopy. The second step of weight loss about 400 °C is ascribed to the thermal decomposition of organic compounds in the DTA curve; an exothermic peak at 400 °C confirms this explanation.



**Fig. 3** The TGA analysis of CeO<sub>2</sub> particles

#### 4. Conclusion

In a summary, our novel method of synthesis based on succinic acid was proven to be facile, highly economical and environmentally friendly. CeO<sub>2</sub> nanostructure has been synthesized by hydrothermal decomposition reaction with succinic acid as an available and affordable ligand. The XRD spectrum shows the presence of CeO<sub>2</sub>. It is expected that as-prepared ceria nanostructure have potential applications in several fields as catalysts, catalyst supports, chemical sensors, storage hydrogen devices and optical or electrical materials.

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