



# Innovative solvo-combustion route for the rapid synthesis of MoO<sub>3</sub> and Sm<sub>2</sub>O<sub>3</sub> materials

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## Abstract

This paper reports the development of a solvo-combustion synthesis route using acetylacetone as the novel fuel and ethanol as the solvent, with water excluded from the system. MoO<sub>3</sub> and Sm<sub>2</sub>O<sub>3</sub> materials synthesized by this route were characterized by various physicochemical techniques. X-ray diffraction (XRD) analysis revealed that the as-prepared materials contained  $\alpha$ -MoO<sub>3</sub> and cubic Sm<sub>2</sub>O<sub>3</sub> began to crystallize at 500 °C. Scanning electron microscopy (SEM) analysis indicated that MoO<sub>3</sub> particles possessed a plate shape, whereas Sm<sub>2</sub>O<sub>3</sub> particles formed foam-like agglomerates. The Sm<sub>2</sub>O<sub>3</sub> materials exhibited surface areas at least 2 times greater than those of the MoO<sub>3</sub> materials. The nature of each oxide and the reagents used in the synthesis played important roles in determining the resulting morphology and surface area. This solvo-combustion route is expected to be suitable for the synthesis of complex oxides with attractive specific surface areas.

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## 1. Introduction

The development of alternative chemical processing methods allows for the preparation of materials with the desired particle shapes and sizes and enables a decrease in the synthesis temperature, improving the homogeneity of the resulting materials. These factors in turn determine the overall physicochemical properties of the material [1–5]. Recently, innovation in synthesis strategies for the production of oxide nanocrystals has been one of the most important issues in research on advanced materials. Several soft chemical methods such as the sol–gel, hydrothermal and solvothermal methods as well as solution combustion have been employed to synthesize finer and more homogeneous powder materials at low temperature [6–10]. Because the aqueous phase in bulk solution promotes rapid growth of oxide

nanoparticles, the nonaqueous sol–gel and solvothermal routes are attractive for the synthesis of well-defined oxide particles under mild conditions [5,11–13].

Thus, solution combustion syntheses have garnered much interest because of their versatility, simplicity and ability to rapidly synthesize a wide variety of advanced materials. This method generally involves a self-sustained reaction mainly featuring an aqueous solution containing a fuel and an oxidizer. The particle sizes and morphologies of oxide materials have been reported to depend to a significant degree on fuel type and the steps of the reaction. Organic compounds such as urea, glycine, citric acid, alanine, ethylene glycol, hexamine, hexamethylenetetramine, methylcellulose and sucrose are commonly used as fuels in combustion processes. Therefore, the influence of fuel type in the combustion reaction process is a key parameter in advanced materials research [9,10,14–25]. Kondawar et al. recently obtained ZnFe<sub>2</sub>O<sub>4</sub> oxide by a method referred to as solvo-combustion, however, water was used as the principal dissolvent during the synthesis [26]. To the best of our knowledge, MoO<sub>3</sub> and Sm<sub>2</sub>O<sub>3</sub>

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have not been prepared by a solution combustion synthesis system of acetylacetone and ethanol without water.

MoO<sub>3</sub> and Sm<sub>2</sub>O<sub>3</sub> are promising materials for modern technological applications in many fields. MoO<sub>3</sub> exhibits good catalytic properties for hydrocarbon oxidation and hydrodesulfurization, and it is an important material for the development of sensors as well as electrochromic and photochromic devices [27–32]. Sm<sub>2</sub>O<sub>3</sub> is one of the most attractive rare-earth oxides due to its potential applications in solar cells, nanoelectronics, semiconductor glass, biochemical sensors and nanomagnets [12,33–36]. The conventional soft chemistry synthesis methods used to prepare these oxides include sol–gel, hydrothermal and templating processes [12,27,35]. The development of new methodologies to produce oxides from simple and readily available starting materials in a few synthesis steps, while ensuring high yield and good physicochemical properties, is a significant challenge for synthetic chemists.

In this study, a new solvo-combustion route was explored for the synthesis of MoO<sub>3</sub> and Sm<sub>2</sub>O<sub>3</sub> powders, using acetylacetone as the fuel and ethanol as the solvent and excluding water. The physicochemical characteristics of the oxides were examined by several analytical techniques. The results are discussed in terms of single phase formation, morphology and surface area. Furthermore, this approach is compared with other conventional methods of synthesis.

## 2. Experimental

### 2.1. Solvo-combustion synthesis

Ammonium molybdate tetrahydrate and samarium (III) acetate hydrate (99.9% Sigma-Aldrich) were used as chemical reagents to obtain MoO<sub>3</sub> and Sm<sub>2</sub>O<sub>3</sub>, respectively. For MoO<sub>3</sub> synthesis in particular, 0.99 mmol of ammonium molybdate tetrahydrate was placed in a flask and dissolved in 0.15 mol of acetylacetone with 0.26 mol of ethanol. The mixture was stirred and refluxed until the temperature reached 70 °C. Then, 1 mL of nitric acid was added and the flask was immediately placed onto a hot plate at 180 °C to rapidly evaporate the solvents. During evaporation, the sample spontaneously ignited, and in a few minutes, a black and spongy powder was obtained. Sm<sub>2</sub>O<sub>3</sub> was synthesized by the same process, but using 5.7 mmol of samarium (III) acetate hydrate as a chemical reagent. The as-prepared powders were ground and thermally treated in air for 6 h at different temperatures.

### 2.2. Characterization

Phase crystallization and complete removal of organic material were determined by differential thermal analysis and thermogravimetric (DTA/TGA) analysis using a SDT Q600, TA Instruments apparatus at a heating rate of 10 °C min<sup>-1</sup> under air flow.

The crystal structure was analyzed by XRD using a Bruker D8 Advance diffractometer with CuK $\alpha$  radiation ( $\lambda=1.5406$  Å). XRD data were collected at room temperature from 10° to 70°, with a step interval of 0.02° and a counting time of 0.5 s step<sup>-1</sup>.

The presence of organic functional groups was determined by Fourier transform infrared spectroscopy (FTIR) analysis using Nicolet 380 FTIR Thermo Electron Corporation equipment. The spectra were recorded in the range of 400–4000 cm<sup>-1</sup>.

Sample morphology was observed using a JEOL 6490 LV scanning electron microscope. All samples were attached to an aluminum sample holder using carbon tape and placed in the SEM chamber.

The specific surface area was determined by the Brunauer–Emmett–Teller method ( $S_{BET}$ ) using nitrogen adsorption isotherms with Quantachrome NOVA 2000e equipment; samples were degassed for 1 h at 300 °C prior to the analysis.

## 3. Results and discussion

### 3.1. Thermal analysis

Fig. 1 shows typical DTA/TGA curves for as-prepared MoO<sub>3</sub> and Sm<sub>2</sub>O<sub>3</sub> samples. The MoO<sub>3</sub> DTA curve presents two exothermic peaks, a small peak at 260 °C and a broad asymmetrical peak from 300 to 570 °C with a maximum at 480 °C, which was attributed principally to the decomposition of remaining organic precursors. This asymmetric peak also exhibits a shoulder at 415 °C that could be related to the crystallization of  $\alpha$ -MoO<sub>3</sub> according to previous work [29]. However, it can be assumed that the crystallization of MoO<sub>3</sub> takes place as a gradual process from 415 °C covering a range of around 150 °C. Finally, an

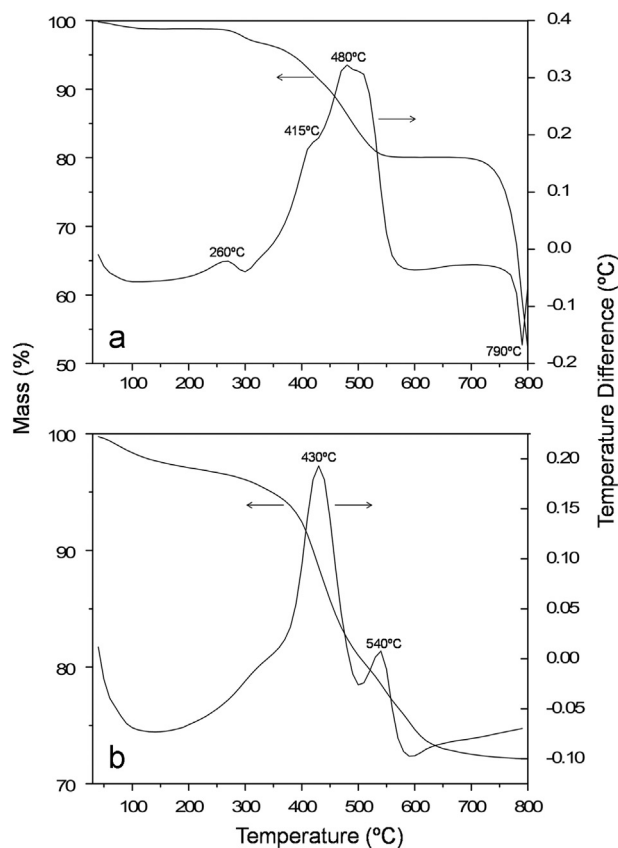


Fig. 1. DTA/TGA profiles of as-prepared samples: (a) MoO<sub>3</sub> and (b) Sm<sub>2</sub>O<sub>3</sub>.

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