



# Facile synthesis of gallium oxide hydroxide by ultrasonic irradiation of molten gallium in water



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## ABSTRACT

This work describes the single-step synthesis of GaO(OH) by ultrasonic irradiation of molten gallium in warm water. The ultrasonic energy causes dispersion of the liquid gallium into micrometric spheres, as well as decomposition of some of the water into H<sup>•</sup> and OH<sup>•</sup> radicals. The OH<sup>•</sup> radicals and the dissolved oxygen react on the surface of the gallium spheres to form crystallites of GaO(OH). These crystallites prevent the re-coalescence of the gallium spheres, and as the reaction proceeds all the gallium is converted into crystalline GaO(OH).

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## 1. Introduction

Gallium oxide hydroxide [GaO(OH)], named also gallium oxyhydroxide, is a precursor for the preparation of the oxide Ga<sub>2</sub>O<sub>3</sub> which has various applications in gas sensing [1,2], catalysis [3] optoelectric devices [4,5] and as an insulating barrier for spin-dependent tunneling junctions [6]. The conversion of GaO(OH) into Ga<sub>2</sub>O<sub>3</sub> is done by dehydration at elevated temperatures. Several routes of synthesis of GaO(OH) were reported in the literature. Sato et al. [7] prepared GaO(OH) by the addition of various bases (NaOH, KOH, etc.) to aqueous solutions of GaCl<sub>3</sub>. The precipitates were all amorphous and were converted spontaneously into GaO(OH) after one day in the basic solutions. Tas et al. [8] precipitated GaO(OH) from solutions of Ga(NO<sub>3</sub>)<sub>3</sub> with or without the presence of urea. Decomposition of urea by reaction with water at 80 °C produced NH<sub>4</sub>OH which raised the pH of the solution and caused precipitation of GaO(OH). In the absence of urea, forced hydrolysis occurred at 90 °C when gallium ions reacted with water to give GaO(OH). A similar reaction of forced hydrolysis was reported by Hamada et al. [9] in the presence of sulfate, yielding

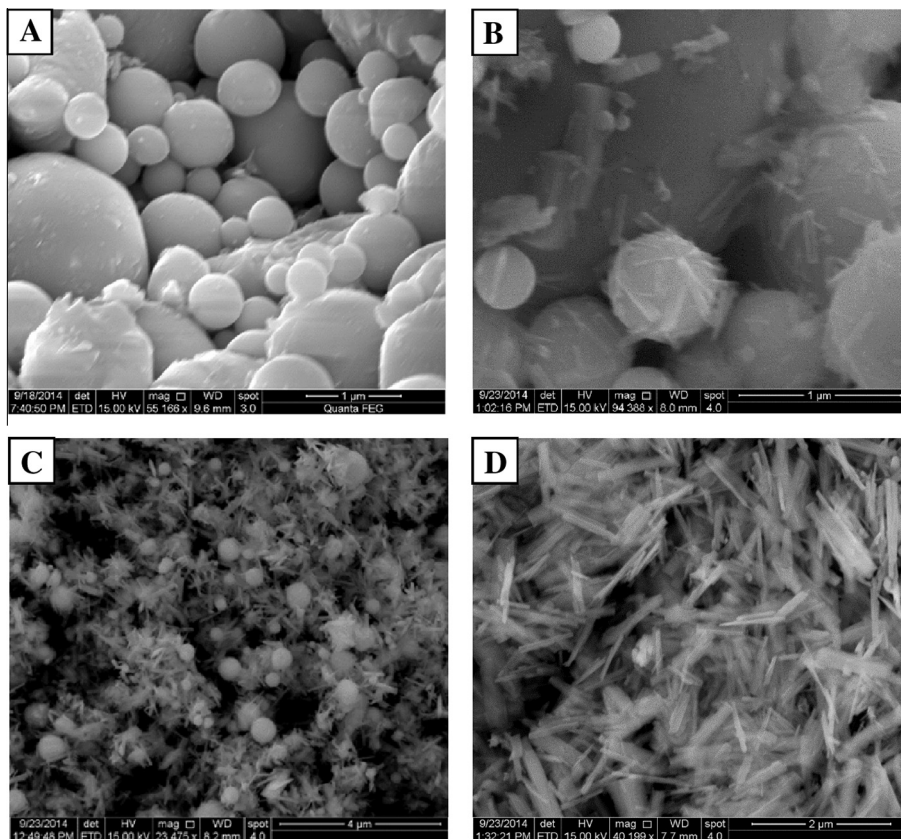
particles of 100 nm in size. Patra et al. [10] reported the formation of sub-micrometer rod-shaped particles by refluxing an aqueous solution of Ga(NO<sub>3</sub>)<sub>3</sub> and NH<sub>4</sub>OH in a microwave oven. Ristič et al. [11] prepared GaO(OH) by the sol-gel method, hydrolyzing aqueous Ga ions in the presence of (CH<sub>3</sub>)<sub>4</sub>NOH. Zhao et al. [5] synthesized micro- to nano-size GaO(OH) particles from solutions of Ga(NO<sub>3</sub>)<sub>3</sub> and NaOH under hydrothermal conditions. No surfactants were needed for stabilization of the particles. A hydrothermal process at different temperatures was used also by Muruganandham et al. [12] for the formation of α-GaO(OH) using oxalic acid. Huang and Yeh [13] utilized a combined procedure of laser ablation followed by refluxing, in the presence of CTAB surfactant or PVP, to prepare nanowires of GaO(OH) and Ga<sub>2</sub>O<sub>3</sub>.

It should be mentioned that Ga<sub>2</sub>O<sub>3</sub> can be synthesized also by direct calcination of Ga(NO<sub>3</sub>)<sub>3</sub> solutions. This method enables the formation of microcomposites of Ga<sub>2</sub>O<sub>3</sub> with other materials. For example, Bagheri et al. [14] prepared gallia-SnO<sub>2</sub> nanocomposite gas-sensors, containing up to 50% Ga<sub>2</sub>O<sub>3</sub> by co-precipitation of various proportions of SnCl<sub>4</sub> and Ga(NO<sub>3</sub>)<sub>3</sub> and calcination at elevated temperatures. In a similar manner they synthesized CoO-doped Ga<sub>2</sub>O<sub>3</sub> nanostructures that can act as photocatalysts [15].

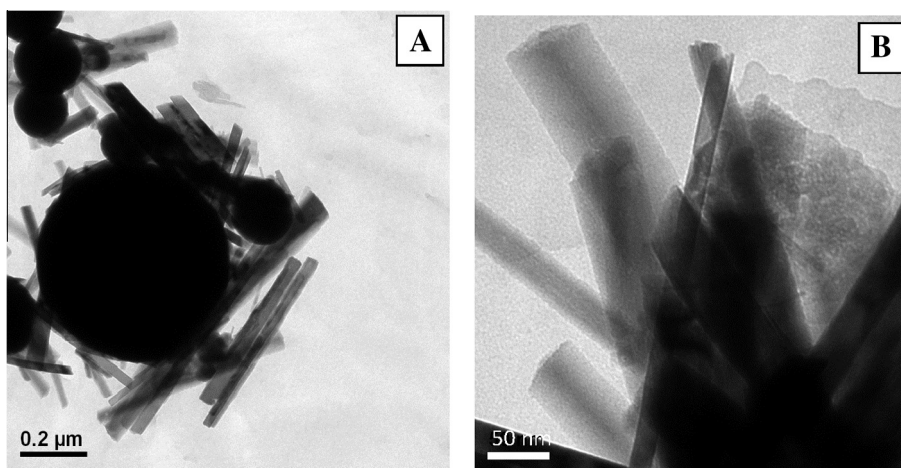
Preparation of GaO(OH) by a sonochemical reaction was done in our laboratory in the past [16]. Ultrasonic irradiation of an aqueous solution of GaCl<sub>3</sub> yielded rolled-up tubular particles, 80–120 nm in diameter and 500–600 nm in length. Small amounts of metallic Ga were incorporated with these tubes. Recently, we reported on the

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**Fig. 1.** SEM micrograph of the products of ultrasonic irradiation of molten Ga in warm water for various durations. (A) 30 s, mainly bare Ga spheres, some of which have merged. (B) 2 min, Ga spheres partially covered with crystallites of GaO(OH) spheres (C) 30 min, Ga spheres together with a considerable portion of GaO(OH). (D) 120 min, GaO(OH) only.



**Fig. 2.** TEM images of the Ga particles and the GaO(OH) crystallites (A) 30 min sonication, (B) 120 min sonication of molten Ga.

formation of gallium spheres in the micrometric to sub-micrometric scale by ultrasonic irradiation of molten gallium in liquid media such as water, n-hexane and n-heptane [17,18]. The dispersion of the molten gallium into microscopic spheres was induced by the acoustic cavitation phenomenon that occurs in liquids upon applying a powerful ultrasonic field. These particles did not recombine into bulk gallium after the sonication ceased, although the temperature of the medium was still higher than the melting point of gallium. For the water-formed particles this was explained by the formation small crystallites on the surface (observed by SEM)

that acted as an isolation layer, whereas the particles formed in organic liquids were found to be coated with a carbon layer (detected by Raman spectroscopy). The composition of the crystallites was identified by X-ray diffraction as GaO(OH).

Ultrasonic-aided syntheses of nanostructured materials were reported in the literature, and a comprehensive review on this topic was published by Bang and Suslick [19]. A recent example for the sonosynthesis of complex porous structures of Zn(II)-based metal-organic framework (MOF) was reported by Masoomi et al. [20]. They found that sonication time and the concentrations of

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