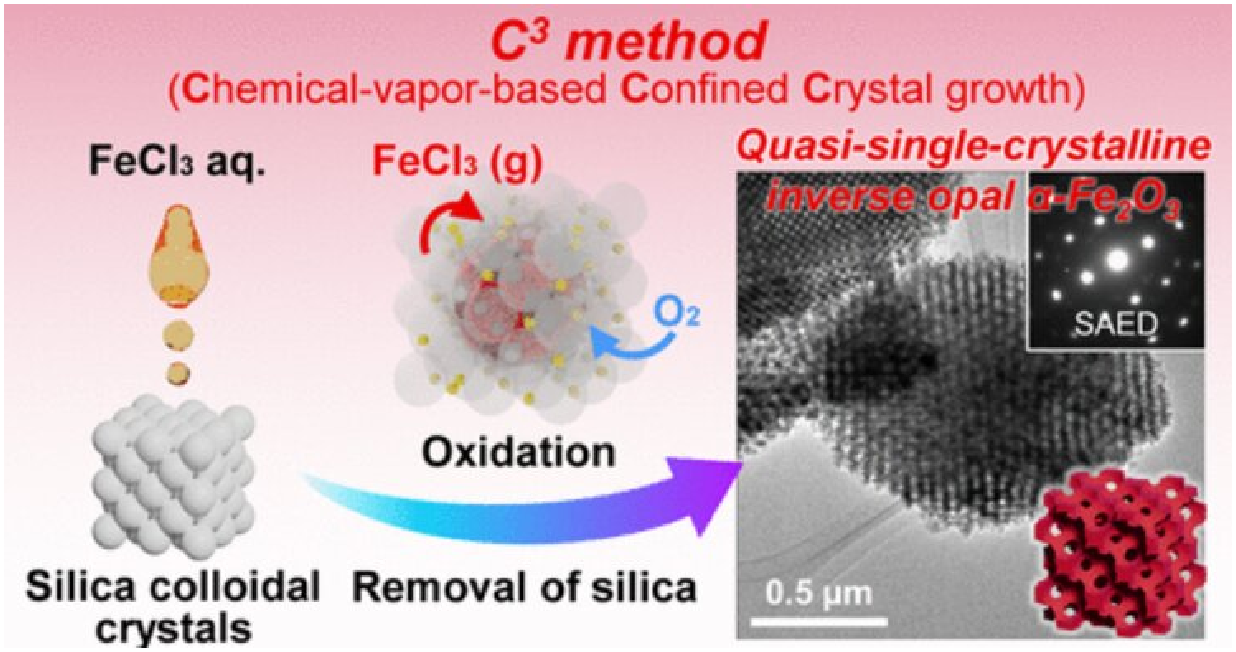
Vapor-based method controls crystallinity of pore walls in nanospaces

August 18 2025



Credit: *Chemistry of Materials* (2025). DOI: 10.1021/acs.chemmater.5c00155

Metal oxide materials with nanoscale pores have been applied and studied in a wide range of fields, including as catalysts, adsorption and separation materials, and energy materials. Among them, single-crystalline nanoporous metal oxides—with interconnected nanopores in a single crystal—are especially lucrative. They have recently attracted attention as unique materials that combine the desirable properties of nanoporous materials, such as high specific surface area and large pore volume, with those of single crystals.

While metal oxide nanoporous structures have been conventionally synthesized by replicating the nanostructure of surfactant micelles, silica, and carbon as templates, the preparation of single-crystalline nanoporous materials is generally difficult, with limited successful approaches. There are still unresolved issues, such as difficulty in controlling the nucleation and crystal growth process inside templates, as well as limited compositions that can be synthesized.

Now, a team of researchers from the Department of Applied Chemistry, Faculty of Science and Engineering, Waseda University, Japan, has developed a novel method for synthesizing difficult-to-prepare single-crystalline nanoporous metal oxides with simultaneous control of their composition, porous structure, and crystal size.

The team was led by Assistant Professor Takamichi Matsuno, including Daichi Oka, Kohei Takaoka, and Professor Atsushi Shimojima. Their findings were published in the journal *Chemistry of Materials*.

Matsuno says, "Iron is an abundant metal on Earth, and iron oxides are used in various applications, including catalysts, electrodes, magnetic devices, and sensors. Therefore, we focused on α-Fe_2O_3, a type of iron oxide, and achieved simultaneous control of its pore structure and crystallite size through the chemical-vapor-based confined crystal growth (C^3) method."

Herein, the team impregnated an $FeCl_3$ precursor aqueous solution and dried it in a porous material composed of silica nanospheres. As a result, an oxide was formed by heating it in the air. After that, the researchers dissolved the silica template in a basic aqueous solution to obtain three-dimensionally ordered nanoporous quasi-single-crystalline α-Fe_2O_3 with an ellipsoidal morphology and average particle dimensions of ~1.1 μm (minor axis) and ~1.6 μm (major axis).

"Heating iron chlorides inside the template causes nucleation and crystal growth of α-Fe_2O_3 via FeOCl by vapor phase transport. As a result, we obtained nanoporous α-Fe_2O_3 with a larger and more uniform crystallite size than that prepared using the previously reported $Fe(NO_3)_3$ hydrate precursor," says Matsuno.

Furthermore, the prepared nanoporous material exhibited high heat resistance as well as high catalytic activity in the photo-Fenton reaction compared to the conventional nanoporous analogs, showcasing the usefulness of nanoporous structures with single-crystalline porous walls.

In this way, the results of this research provide a new way to enhance the performance of nanomaterials and are expected to have a ripple effect in a wide range of fields, including catalysts and energy conversion materials that contribute to the realization of carbon neutrality. Moreover, this work addresses a significant challenge in materials synthetic chemistry, thereby paving the way for the creation of a series of materials with excellent properties through precise and flexible control.

Overall, the proposed C^3 method of volatilizing and oxidizing metal chlorides within nanoporous materials potentially offers a universal approach to controlling the factors that affect their properties.

More information: Daichi Oka et al, Quasi-Single-Crystalline Inverse Opal α-Fe_2O_3 Prepared via Diffusion and Oxidation of the $FeCl_3$ Precursor in Nanospaces, *Chemistry of Materials* (2025). DOI: 10.1021/acs.chemmater.5c00155

Provided by Waseda University

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