

Supporting information

A facile one-step hydrothermal synthesis of rhombohedral CuFeO₂ crystals with antivirus property

Xiaoqing Qiu, Min Liu, Kayano Sunada, Masahiro Miyauchi* and Kazuhito Hashimoto*

Experimental Details

Sample Preparation

All chemical include Fe(NO₃)₃·9H₂O, Cu(NO₃)₂·3H₂O, NaOH, and propionaldehyde were purchased from Aldrich and Wako and used as received. The experiment procedure of CuFeO₂ synthesis is illustrated in Fig. S1.

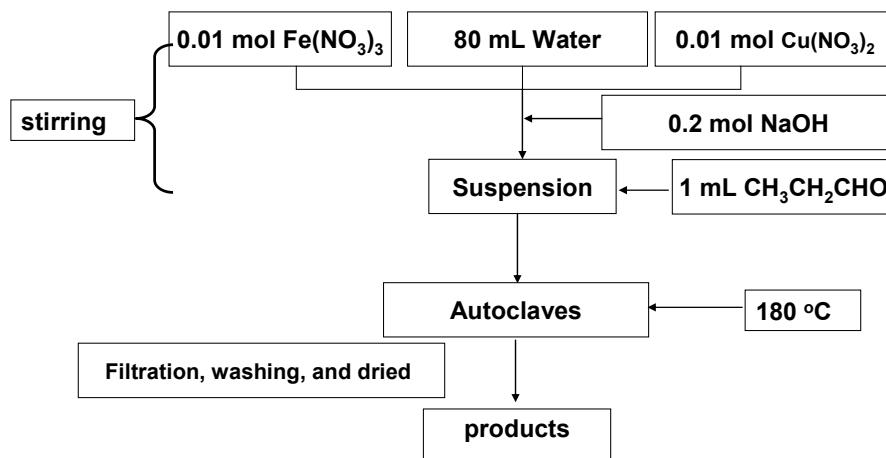


Fig. S1 The experiment procedure of CuFeO₂ synthesis via hydrothermal route

Sample Characterization

The structural characteristics of the samples were measured by powder X-ray diffraction (XRD) at room temperature on a Rigaku D/MAX25000 diffractometer with a copper target ($\lambda = 1.54178 \text{ \AA}$). The data were collected from $2\theta = 10\text{--}80^\circ$ in a step-scan mode (step, 0.02° , counting time 5s). The lattice parameters for the samples

were refined by the least-squares methods using the Rietica program. The morphologies of the samples were investigated by a field-emission scanning electron microscopy (SEM) using a JEOL JSM-6700 apparatus. The absorption spectra of the supernatant after acid etching were recorded using a UV-2550 spectrophotometer (Shimadzu). The ionic characteristics and surface composition were studied by X-ray photoelectron spectroscopy (XPS, Perkin-Elmer, 5600). The binding energy data are calibrated with the C 1s signal at 284.6 eV.

Evaluation of antivirus effect

CuFeO₂ deposited glass (2.5 cm × 2.5 cm) was prepared by simply spreading ethanol suspension (150 µL) of CuFeO₂ (1 mg/mL) for antivirus evaluation. The deposited amount of CuFeO₂ was 0.15 mg/6.25 cm², equal to 0.24 g/m². The glass samples were dried up and sterilized at 120 °C for 3 h. Qβ bacteriophage (NBRC20012) and *Escheichia coli* (NBRC 13965) as the host bacteria were used in the evaluation experiment. For stock suspension of Qβ bacteriophage ($\sim 1.2 \times 10^{11}$ PFU/mL), the bacteriophage infected to the *E. coli* at 35 °C for 10 min was incubated on the double layer plate, which was prepared with nutrient broth (DifcoTM) and agar (DifcoTM) by adjusting agar concentration of bottom layer to 1.5 % and the top layer to 0.5 %. After the plates were incubated at 35 °C overnight, the top agar layer including the bacteriophage, was collected and eluted in 2 mL/plate SM buffer (0.1 M NaCl, 8 mM MgSO₄, 50 mM Tris-HCl pH 7.5 and 0.1% gelatin) at 4 °C overnight. Then the bacteriophage elution was centrifuged (8000 × g, 4°C, 20 min) and the supernatant was collected, filtered (0.22 µm, Millipore, MA) and stocked. The bacteriophage was diluted with PBS to give approximately 2.5×10^9 plaque forming units. Then, the dilution (50µL) was pipetted onto the CuFeO₂ deposited glass. The glass was then kept in the dark. After a period of contact time, the bacteriophage suspension was collected into SM buffer (10mL). An appropriate dilution of the collected suspension was infected to the *E. coli*, and then plated onto an agar medium by above double layer method to determine the number of plaque.

XRD

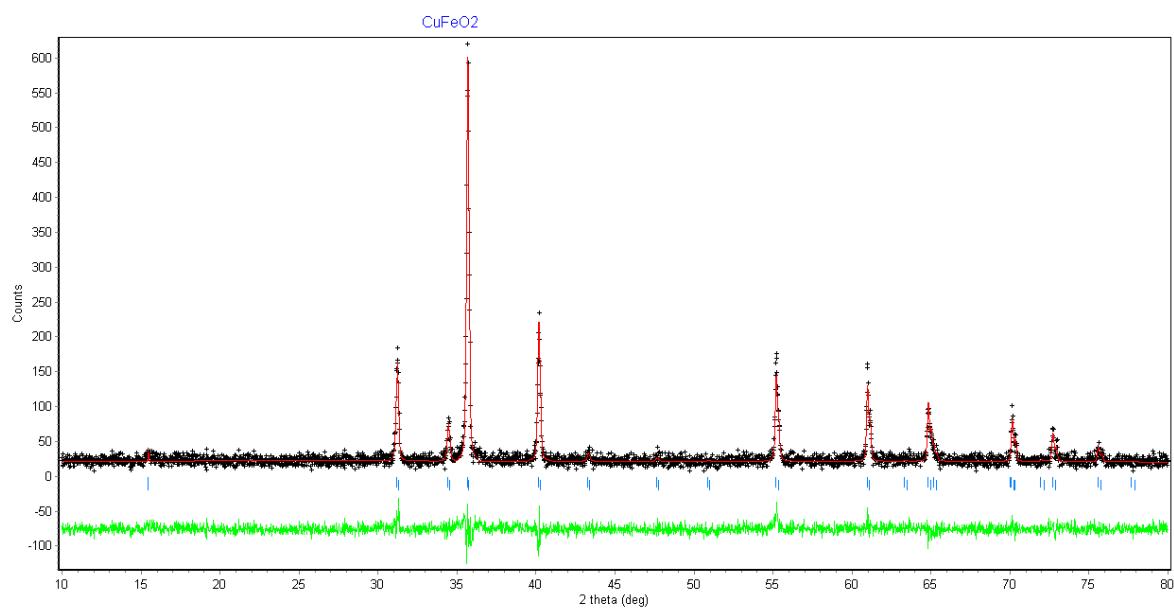


Fig. S2 XRD pattern of as-prepared CuFeO₂ samples ($t = 24$ h) refined with the Rietica Rietveld program, where the black line is for the raw data, the red line is for the calculated values, the green line is the difference. Refined structural parameters ($a = 3.0351\text{\AA}$, $c = 17.1562\text{ \AA}$, $V = 136.8668\text{ \AA}^3$) match the bulk CuFeO₂ data.

SEM

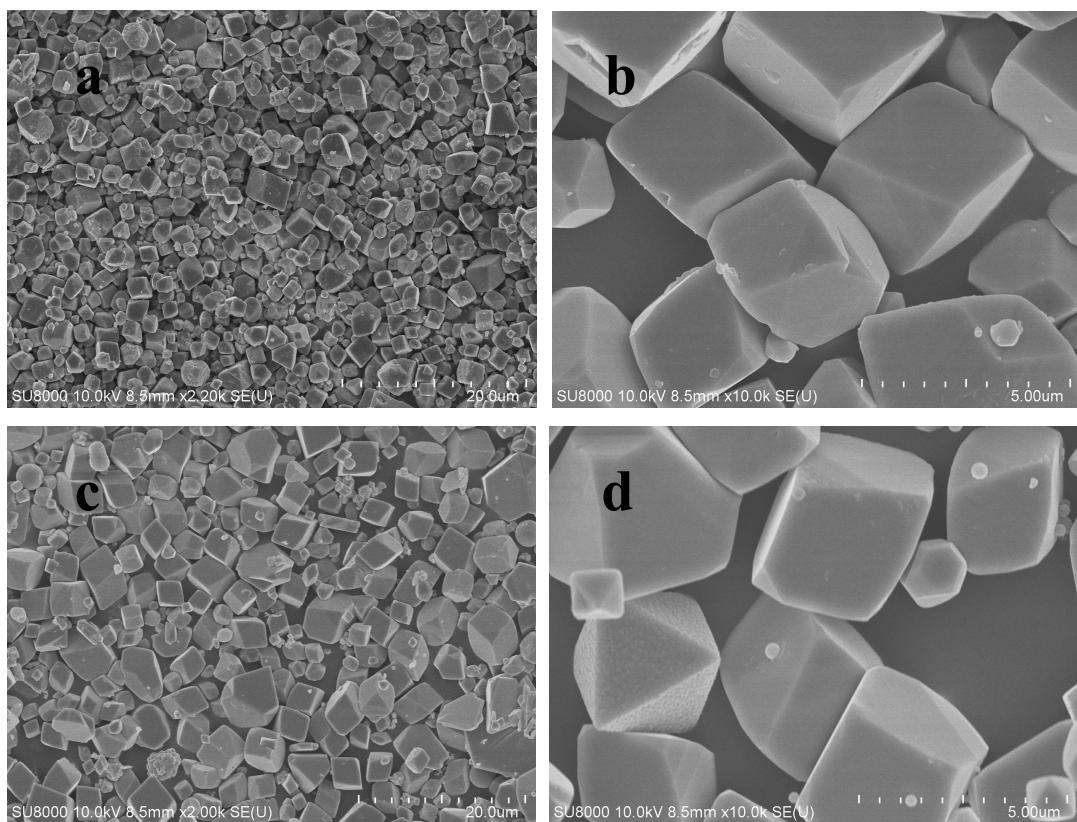


Fig. S3 SEM images. a and b for the sample obtained at 180 °C for 48 h; c and d for the sample obtained at 180 °C for 64 h.