

Supplementary Material (ESI) for Chemical Communications

Electronic Supporting Information for the article:

Green one-pot assembly of iron-based nanomaterials for the rational design of structure

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Experimental Section

Synthesis of low dimensional iron-based nanomaterials. All chemicals used were analytical grade and used without further purification. 1-Butyl-3-methylimidazolium chloride (C_4MimCl , high purity 99.9%) was supplied by C-TRI. Ferric chloride hexahydrate ($FeCl_3 \cdot 6H_2O$, Aldrich, 99.9%) was used as an iron precursor without further purification. Hydrochloric acid (HCl) was supplied by Aldrich.

1.222 g of C_4MimCl was mixed with 1 mL of deionized water. After 1.89 g of ferric chloride hexahydrate was added to the mixture at the molar ratio of $FeCl_3 \cdot 6H_2O$ to C_4MimCl of 1 to 1, 3 ml of 0.1 M HCl was added to the resulting mixture dropwise to control pH 3. The resultant mixture was stirred at room temperature for 120 min in order to obtain a homogeneous solution. To induce the formation of nanostructure via a green ionothermal process, the mixture was heated to 120 °C for 8 hours at ambient pressure.

Characterization. Elemental analysis was performed within the accuracy of 0.3% by elemental analyzer, which is EA1110-FISONS, manufactured by ThermoQuest Italia S.P.A (CE INSTRUMENTS). C, H, O, and N elements of all samples were analyzed quantitatively in the range of 0.01 – 100%. TEM images were collected on an E.M. 912Ω Energy-Filtering Transmission Electron Microscope (EF TEM, 120 kV) and a JEM-3010 High-Resolution Transmission Electron Microscope (HR-TEM, 300 kV). X-ray Diffraction (XRD) data were obtained on a Rigaku D/max IIIc (3 kW) with a θ/θ goniometer equipped with a $CuK\alpha$ radiation generator. The diffraction angle of the diffractograms was in the range of $2\theta=20 - 80^\circ$. FT-IR ATR spectra were collected on a JASCO FT-IR 470 plus as attenuated total reflection. Each spectrum, which was recorded as the average of 12 scans with a resolution of 4 cm^{-1} , was collected from 4000 cm^{-1} to 650 cm^{-1} . Thermogravimetric analyses (TGA) were carried out using a Dupont 2200 thermal analysis station. Every sample was heated from 30 to 900 °C at a rate of $10\text{ }^\circ\text{Cmin}^{-1}$ under a nitrogen atmosphere. N_2 sorption/desorption data was obtained using a gas sorption analyzer (NOVA 4200 Ver. 7.10). The specific surface area was calculated by the BET equation and pore size was determined by the BJH model.

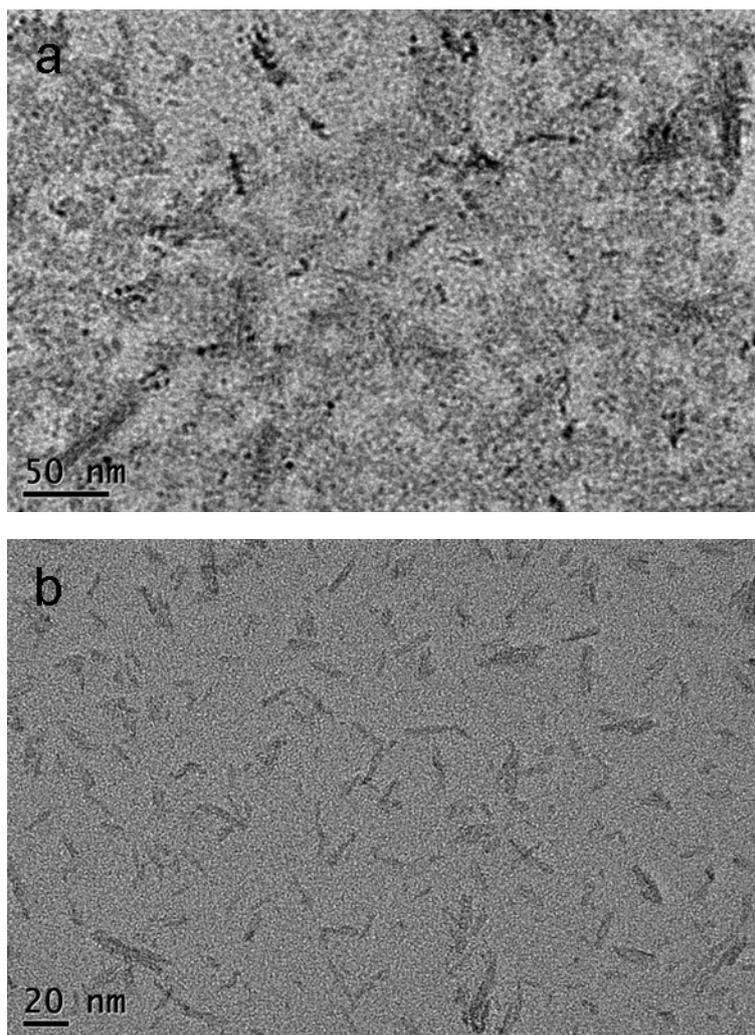


Fig. S1 TEM images of β -FeOOH nanoparticles prepared by ionothermal treatment for (a) 2h and (b) 4h.

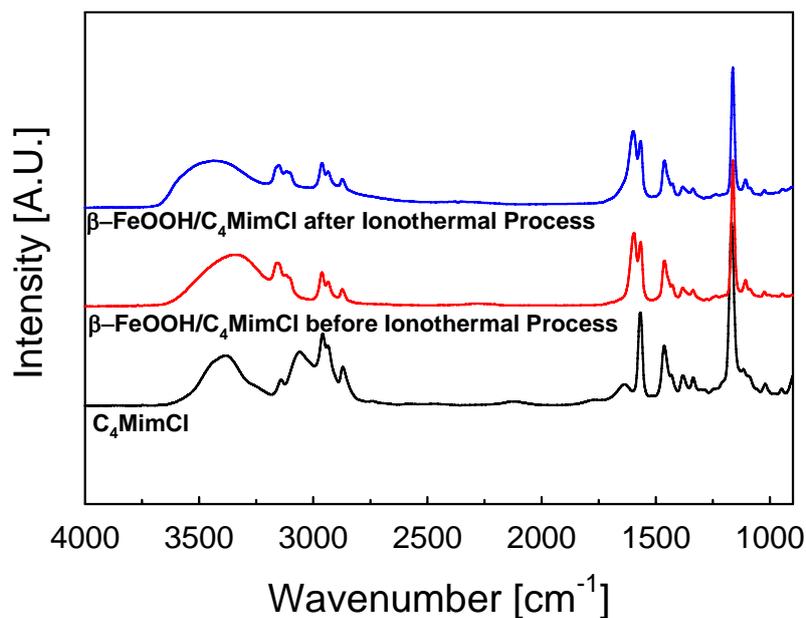


Fig. S2 FT-IR spectra of pristine C₄MimCl, β -FeOOH/C₄MimCl nanorods before ionothermal treatment, and β -FeOOH/C₄MimCl nanorods after ionothermal treatment.

Two peaks above 3000 cm⁻¹ in IR spectra of C₄MimCl are regarded as diagnostic of the changes in the CH...Cl hydrogen bonding between the Cl⁻ anion and the 4, 5-hydrogens of the imidazolium ring.¹ In hybrid system, hydrogen bonding interactions between C₄MimCl and FeOOH induced to shift these two peaks. In addition, the characteristic peaks of imidazolium ring around 1570 cm⁻¹, 1460 cm⁻¹, and 1170 cm⁻¹ were also broadened, split, or shifted as both results of the stacking of imidazolium ring and hydrogen bonding between anion and FeOOH.

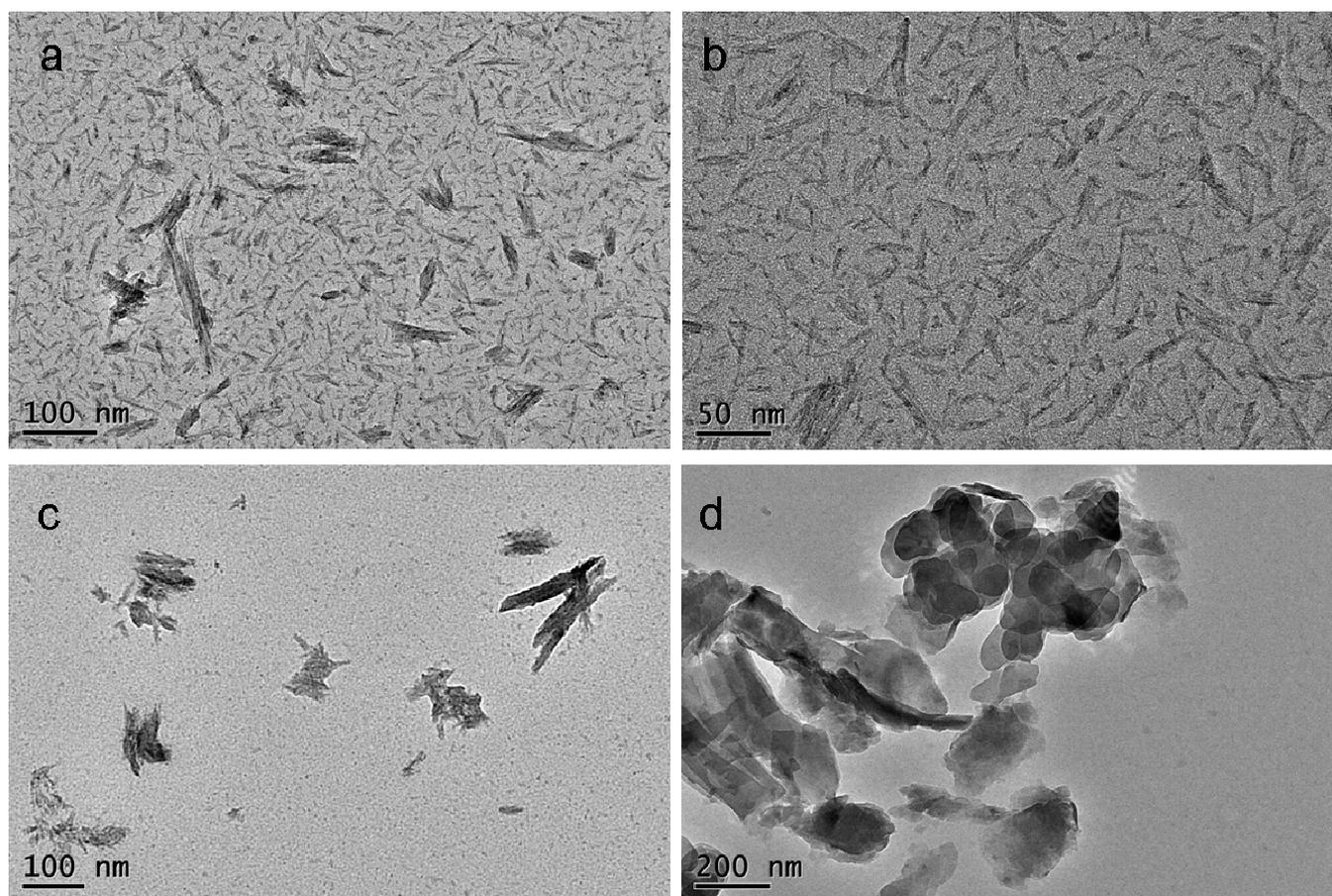


Fig. S3 TEM images of β -FeOOH nanoparticles prepared by ionothermal treatment at (a) 80 °C, (b) 120 °C, (c) 160 °C, and (d) 200 °C.

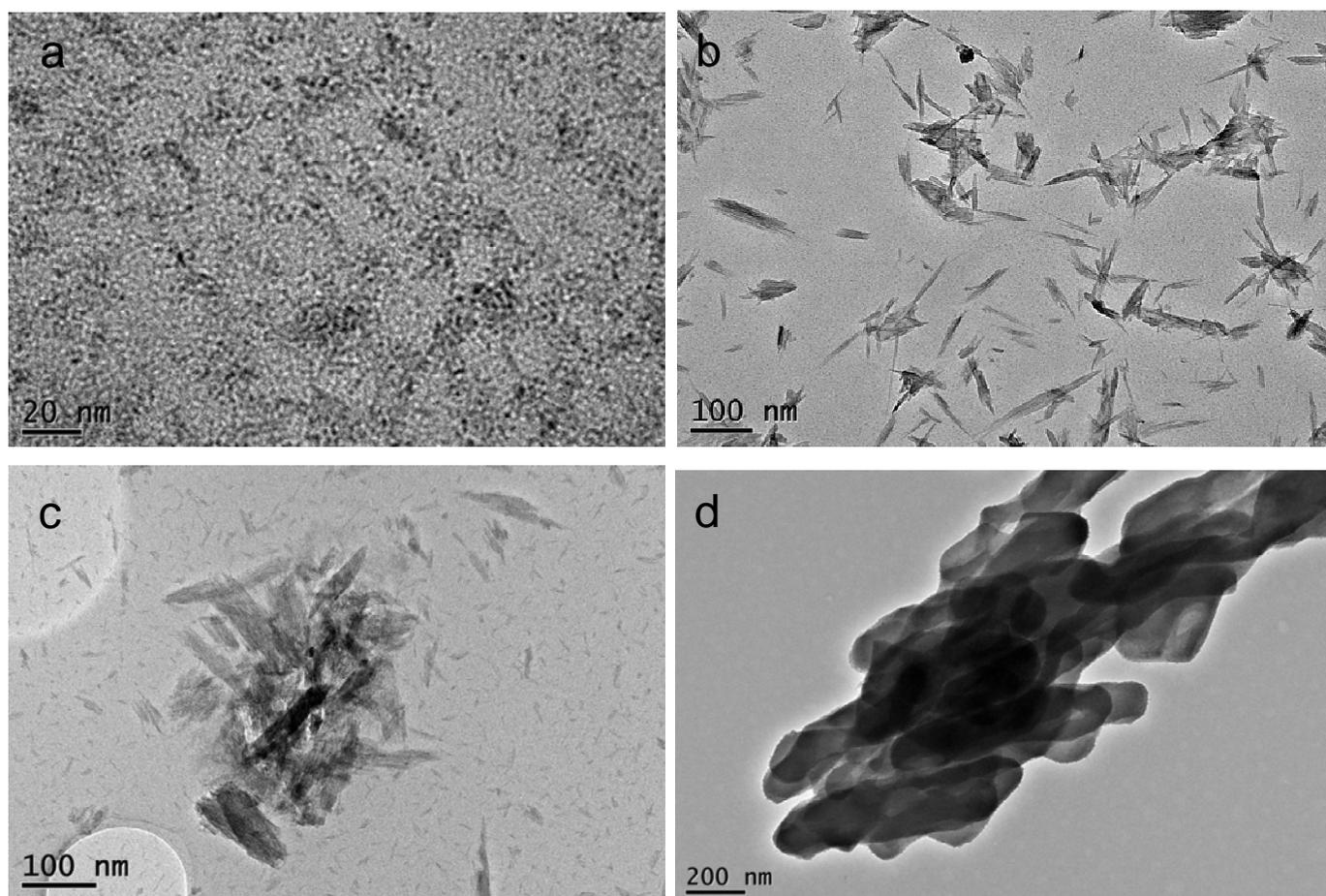


Fig. S4 TEM images of β -FeOOH nanoparticles prepared at a molar ratio of $\text{FeCl}_3 \cdot 6\text{H}_2\text{O}$ to C_4MimCl of (a) 2 : 1, (b) 1 : 2, (c) 1 : 4, and (d) 1 : 8.

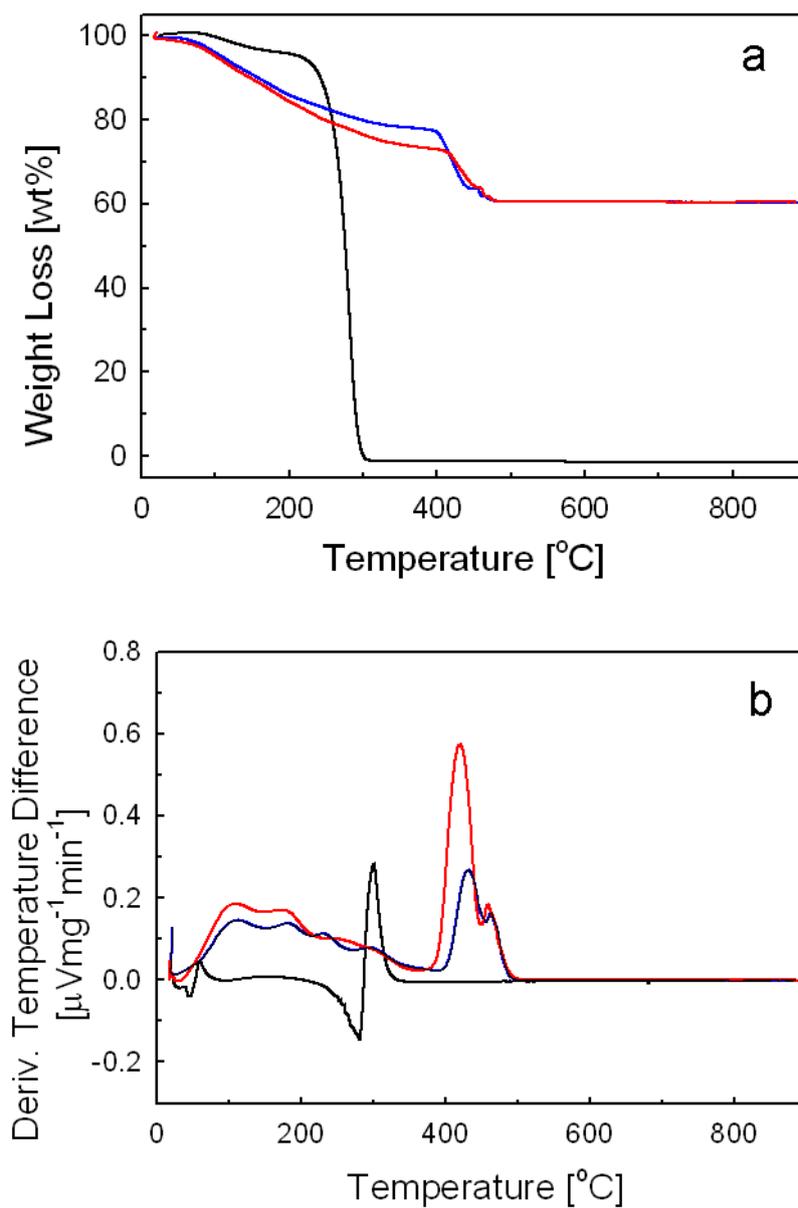


Fig. S5 (a) TGA and (b) DTG curves of pristine C₄MimCl (black line), β -FeOOH nanorods (blue line), and β -FeOOH/C₄MimCl nanorods (red line) under air.

The content of C₄MimCl in β -FeOOH/C₄MimCl nanorods was calculated by subtracting wt% of β -FeOOH from β -FeOOH/C₄MimCl at 400 °C.