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Supporting Information

Chemicals. Triblock copolymer poly(ethylene glycol)-*block*-poly-(propylene glycol)-*block*-poly(ethylene glycol) (Pluronic F127, $M_{av} = 12600$, PEO₁₀₀PPO₆₅PEO₁₀₀), anhydrous iron nitrate (Fe(NO₃)₃), and nickel nitrate hexahydrate (Ni(NO₃)₂·6H₂O) were purchased from Sigma-Aldrich. Orthophosphoric acid (H₃PO₄), hydrochloric acid (HCl), ethanol, methanol, acetonitrile, propanol and tetrahydrofuran (THF) were purchased from Wako. The chemicals were used without further purification.

Experimental Section. In a typical synthesis of mesoporous FeP plates, 25 ml ethanol was acidified with 1.65 g (35 wt %) hydrochloric acid solution. Then, 1 g triblock copolymer (Pluronic F127) was further added to the acidified ethanolic solution and magnetically stirred for 2 h at room temperature (298 K). Then, 5 mmol H₃PO₄ (85 wt %) was added under stirring. In another beaker, 5 mmol FeCl₃ was dissolved in 10 ml ethanol separately. Then, the two solutions were mixed, and the stirring was continued for 10 hours at 298 K. After that, the solution was kept inside an oven at 333 K until the solution was dried completely. The as-prepared sample was collected by washing with water and diethyl ether three times. Finally, the samples were calcined at 723 K for 6 hours under aerobic conditions to obtain mesoporous FeP materials with crystallized walls. Similarly, the synthesis of mesoporous FeP rods was carried out by the same synthetic procedure. The molar ratios of the chemicals were exactly the same, but THF was used as the solvent instead of ethanol.

Characterizations. Scanning electron microscope (SEM) images were collected with a Hitachi SU-8000 SEM at an accelerating voltage of 15 kV. Transmission electron microscope (TEM) observation was performed using a JEM-2100 TEM system that was operated at 200 kV. Wide-angle powder X-ray diffraction (XRD) patterns were obtained with a Rigaku RINT 2500X diffractometer using monochromated Cu K α radiation (40 kV, 40 mA) at a scanning rate of 0.1°·min-1. Nitrogen adsorption-desorption analysis was performed using a Belsorpmini II Sorption System at 77 K. The specific surface areas were calculated by the Multipoint Brunauer-Emmett-Teller (BET) method in a relative pressure, P/P₀, range from 0.05 to 0.30, while the total pore volumes were calculated by the Barrett-Joyner-Halenda (BJH) method. X-ray photo-electron spectroscopy (XPS) spectra were recorded at room temperature by using a JPS-9010TR (JEOL) instrument with an Mg K α X-ray source. All binding energies were calibrated by referencing to C 1*s* (285.0 eV).



Fig. S1 SEM images of the various FeP materials prepared with different solvents [(a) ethanol:water (1:1), (b) THF:water (1:1), (c) water, (d) acetonitrile, (e) methanol, and (f) propanol.

Notes on Fig. S1: Here we have prepared various FeP materials in various solvents. When we use EtOH:H₂O (1:1) or THF:H₂O (1:1) as mixed solvent instead of only EtOH or THF, the plate width and rod diameter increases due to the increment of polarity and H-bonding ability of the medium. In case of EtOH:H₂O (1:1), although the material has 2D-plate like morphology, the width of plates is increased to 35 nm (**Fig. S1a**) compared to the plate width of 22 nm when synthesis was carried out in only EtOH. In case of THF:H₂O (1:1), the diameter of the rods is increased to 47 nm (**Fig. S1b**) compared to the rod diameter of 34 nm when synthesis was carried out in only THF. Apart from these mixed solvents, we prepared the FeP materials in water, acetonitrile, methanol, and propanol, respectively. Due to the high polarity and H-bonding ability (like THF), the obtained FeP material has also rod-like morphology with the diameter of around 60 nm (**Fig. S1c**). In case of acetonitrile, due to the moderate polarity and no-hydrogen bonding ability (like THF), the obtained FeP material has also rod-like morphology. The average widths are 25 nm and 22 nm, respectively (**Fig. S1e-f**). From these controlled experiments, we can conclude that all the FeP materials have well-defined morphologies which are mostly controlled by the polarity and H-bonding ability of the synthetic medium.



Fig. S2 Elemental mapping with EDS spectra (bottom) of (a) mesoporous FeP plates and (b) mesoporous FeP rods, respectively.

Figure S3



Fig. S3 Wide-angle X-ray diffraction patterns of as-synthesized FeP plates (black line) and FeP rods (red line).



Fig. S4 XPS spectra of mesoporous FeP plates after calcination: (a) survey, (b) Fe 2*p*, (c) P 2*p*, and (d) O 1*s* spectra.

Notes on Fig. S4: X-ray photoelectron spectroscopy (XPS) is a powerful tool that is used to determine not only the electronic structure of solids, but also is very important for measuring the chemical composition and local structure of materials. The XPS survey spectrum of the mesoporous FeP plates is shown in **Fig. S4a**. From the survey spectrum, the elements iron (Fe), phosphorus (P), and oxygen (O) are present in the material. A high resolution XPS spectrum with Fe $2p_{3/2}$ and Fe $2p_{1/2}$ is presented in **Fig. S4b**. In the mesoporous FeP plates, the binding energies (BE) associated with Fe $2p_{3/2}$ and Fe $2p_{1/2}$ are 711.38 eV and 725.30 eV, respectively. The line shapes and peak positions are in good agreement with the previously reported iron phosphate material (G. K. Marasinghe *et al., J. Non-Cryst. Solids*, 2000, **263-264**, 146). The P 2p peak at 133.66 eV is deconvoluted into two components with the energy levels for P $2p_{1/2}$ (134.11 eV) and P $2p_{3/2}$ (133.24 eV) (**Fig. S4c**). The main part (P 2p) with binding energy 134.14-133.11 eV is attributed to pentavalent tetra-coordinated phosphorous surrounded by different chemical environments. The peak at 531.63 eV is attributed to the O 1*s* peak of the FePO₄-like structure (**Fig. S4d**).



Fig. S5 (a, b) SEM images of the FeP materials prepared in the absence of F127 in (a) ethanol medium and (b) in THF medium. (c, d) SEM images of the FeP materials prepared under mixed solvent conditions (ethanol and THF).

Figure S6



Fig. S6 The relationship between the length ratios and the solvent compositions. The lengths in two different directions (as indicated by arrows) are measured for mesostructured/mesoporous plates and rods at prepared from various solvent compositions. Then, the length ratios (y/x, y>x) are calculated and plotted.



Fig. S7 SEM images of as-prepared (a) NiP plates, (b) NiP rods synthesized in EtOH and THF medium, respectively, (c) N_2 adsorption-desorption isotherms of mesoporous NiP plates and NiP rods, (d) Pore size distribution curves of mesoporous NiP plates and NiP rods.

Notes on Fig. S7: We have extended our concept to formation of mesoporous nickel phosphate (NiP) with plate- and rod-like morphologies. The experimental conditions are exactly the same as the synthesis of mesoporous FeP materials. Instead of iron nitrate, nickel nitrate was used as an inorganic salt. The corresponding SEM images of as-prepared NiP-plates and NiP-rods are shown in **Fig. S7a-b**. The corresponding N₂ adsorption-desorption isotherms of NiP-plates and NiP-rods after removal of templates are also shown in **Fig. S7c**. The Brunauer-Emmett-Teller (BET) surface areas are 65 m²·g⁻¹ (for mesoporous NiP plates), and 43 m²·g⁻¹ (for mesoporous NiP rods), respectively. The pore size distribution curves obtained by the Barrett-Joyner-Halenda (BJH) method is 5.2 nm (**Fig. S7d**).