

Supporting Information

One-pot synthesis of 5-hydroxymethylfurfural from carbohydrates using an inexpensive FePO₄ catalyst

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To further understand the “phase-transfer” phenomenon, we carried out a series of control experiments, as shown in Table 1. As can be seen, the aqueous solutions which were obtained by immediately filtered upon cooling to the room temperature, contain high concentration FePO₄ (2.13 mg/mL dissolved at 160 °C for 30 min, Table 1, entry 5) before the occurrence of precipitation because the precipitation process is a very slow process. After the storage of 48 h, the dissolved FePO₄ would be slowly precipitated to get the final solution (the Fe³⁺ concentration was less than 0.009 mg/mL, Table 1, entry 5). So, the “phase-transfer” behavior refers to a part of insoluble solid FePO₄ will begin to dissolve in aqueous solution upon elevating the reaction temperature, i.e. the transfer from solid phase to aqueous phase. The Fe³⁺ ions are not dissolved in THF phase.

Table 1 The solubility of FePO₄ at different conditions and the final Fe³⁺ concentration^a

Entry	Time/min	T/°C	Insoluble solid/mg	Solubility /mg.mL ⁻¹ ^b	Final Fe ³⁺ concentration/mg.mL ⁻¹ ^c
1	15	140	135.0	1.65	0.0043
2	30	140	136.0	1.70	0.0071
3	60	140	132.0	1.73	0.0074
4	15	160	123.0	1.98	0.0060
5	30	160	123.0	2.13	0.0090

^aThe experimental condition: 0.2 g FePO₄, 10 mL distilled water, 30 mL THF, 3.5 g NaCl

^bThe solubility was calculated by the dissolved FePO₄ mass divided into the volume of water phase. After the temperature was cooled to room temperature, the reaction solution was immediately taken out and then was filtered to obtain the aqueous phase and THF phase. Subsequently, the water phase was obtained by separating THF phase.

^cThe filtrate was stored for 48 h to fully precipitate the soluble FePO₄ and then Fe³⁺ concentration was determined by atom absorbance spectroscopy (AAS)