

Supporting Information

Preparation of superparamagnetic $\text{Mn}_x\text{Fe}_{1-x}\text{O}$ nanoparticles from
low-index-facet cubes to high-index-facet concaves and the catalytic
performance in aqueous solution

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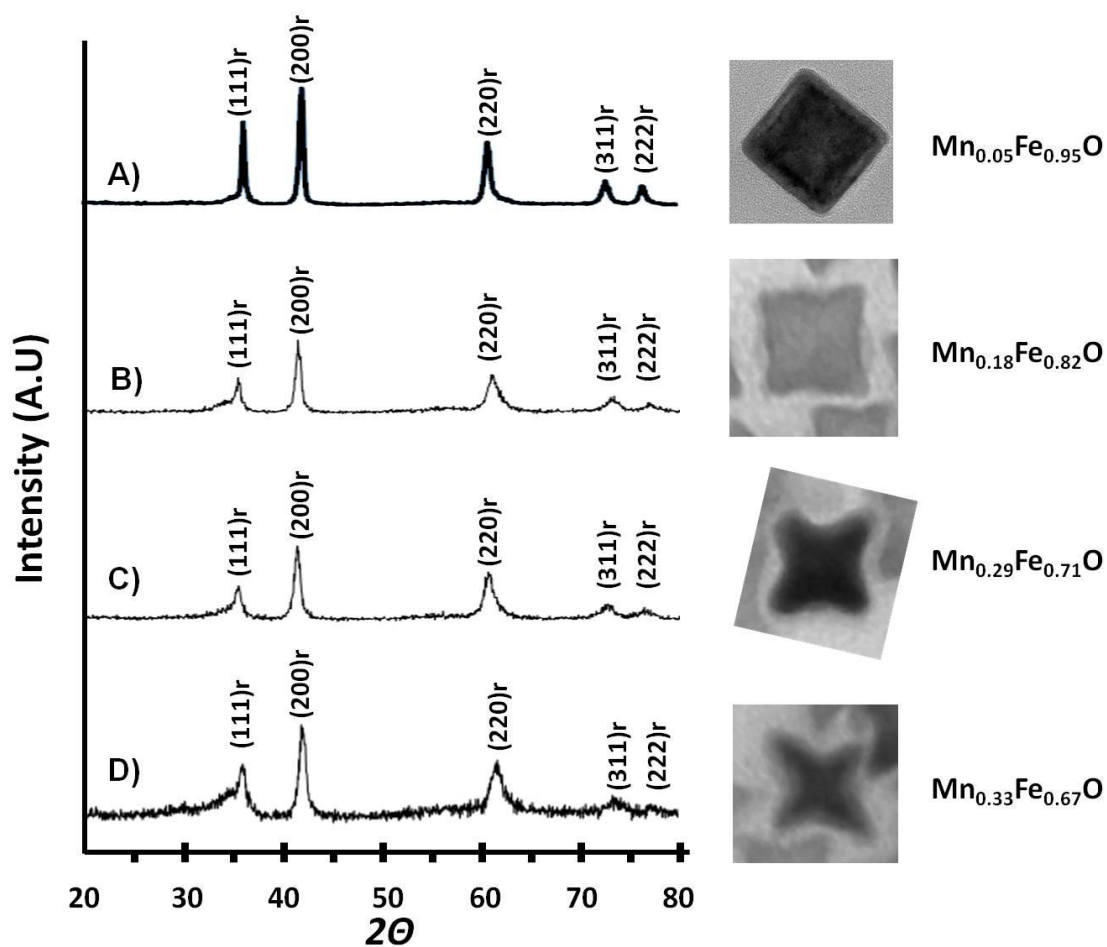


Figure S1. XRD patterns of A) $\text{Mn}_{0.05}\text{Fe}_{0.95}\text{O}$ obtained from pure argon, B) $\text{Mn}_{0.18}\text{Fe}_{0.82}\text{O}$ obtained by the exposure of air for 1.5 min, C) $\text{Mn}_{0.29}\text{Fe}_{0.71}\text{O}$ obtained by the exposure of air for 2 min, and D) $\text{Mn}_{0.33}\text{Fe}_{0.67}\text{O}$ obtained by the exposure of for air 3.5 min. The subscript label “r” indicates NaCl-type (rock-salt).

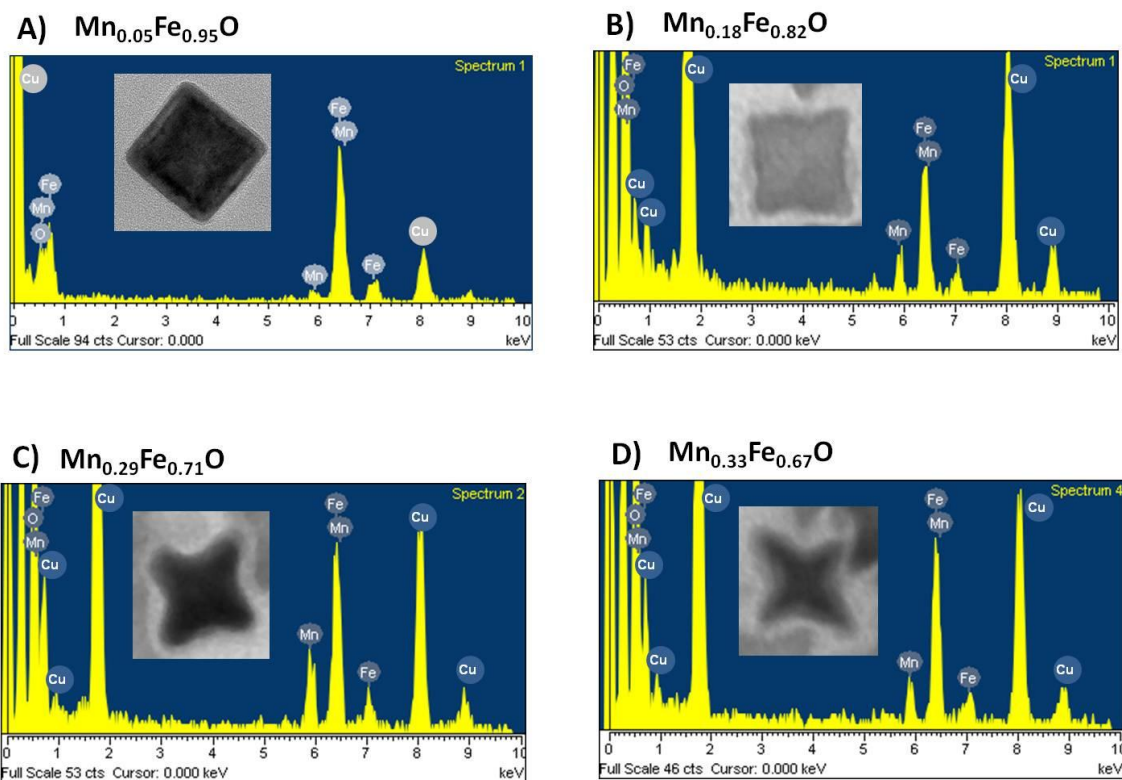


Figure S2. EDS analysis of A) $\text{Mn}_{0.05}\text{Fe}_{0.95}\text{O}$ obtained from pure argon, B) $\text{Mn}_{0.18}\text{Fe}_{0.82}\text{O}$ obtained by the exposure of air for 1.5 min, C) $\text{Mn}_{0.29}\text{Fe}_{0.71}\text{O}$ obtained by the exposure of air for 2 min, and D) $\text{Mn}_{0.33}\text{Fe}_{0.67}\text{O}$ obtained by the exposure of air for 3.5 min. The Cu signal is derived from Cu mesh substrate.

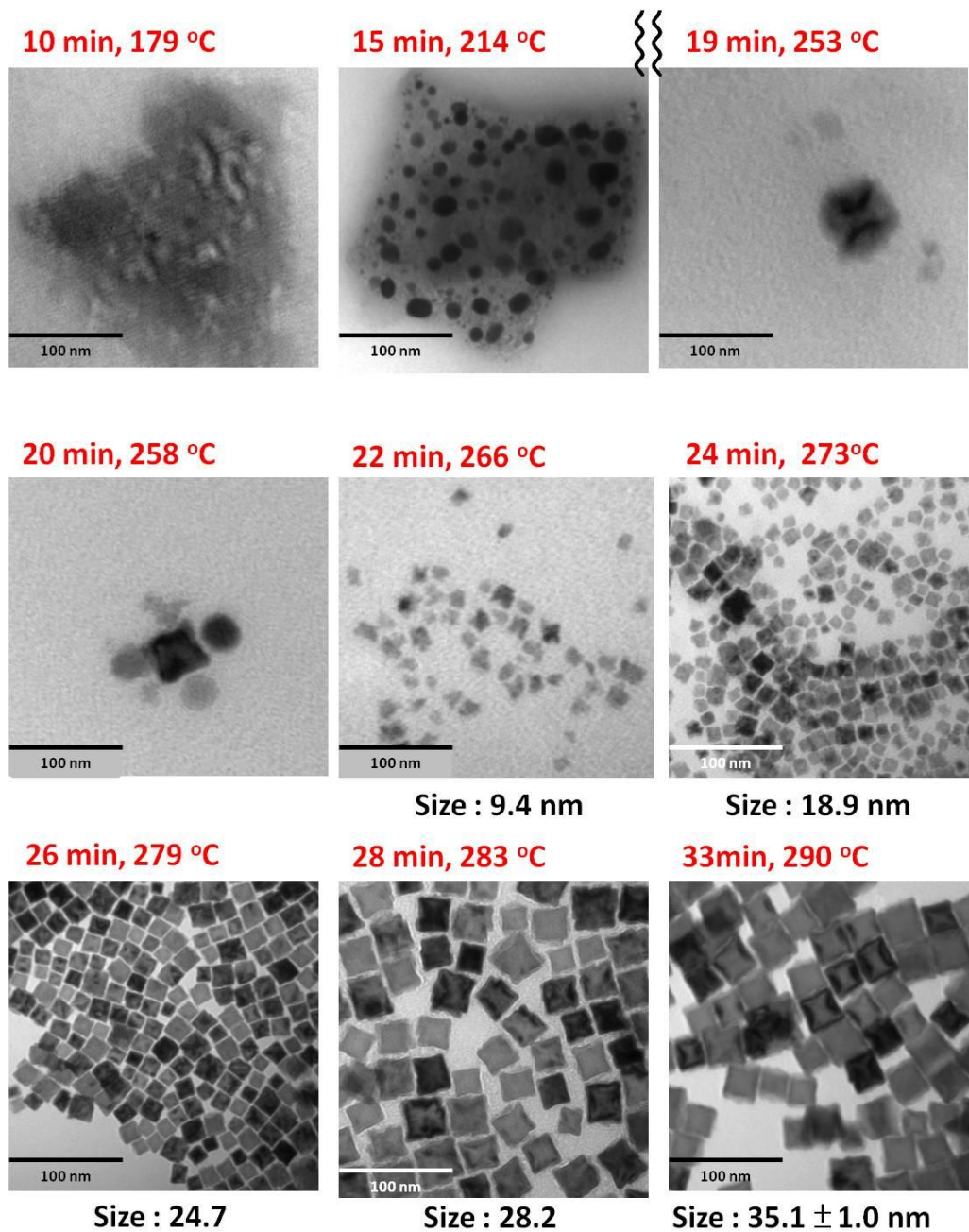


Figure S3. Time-dependent TEM images over the course of reaction time for $\text{Mn}_{0.18}\text{Fe}_{0.82}\text{O}$ concave nanoparticles obtained by the exposure of air 1.5 min. The respective TEM images were taken from reaction time of 10 min, 15 min, 19 min, 20 min, 22 min, 24 min, 26 min, 28 min, and 33 min. The symbol { { represents air exposure of reaction mixture.

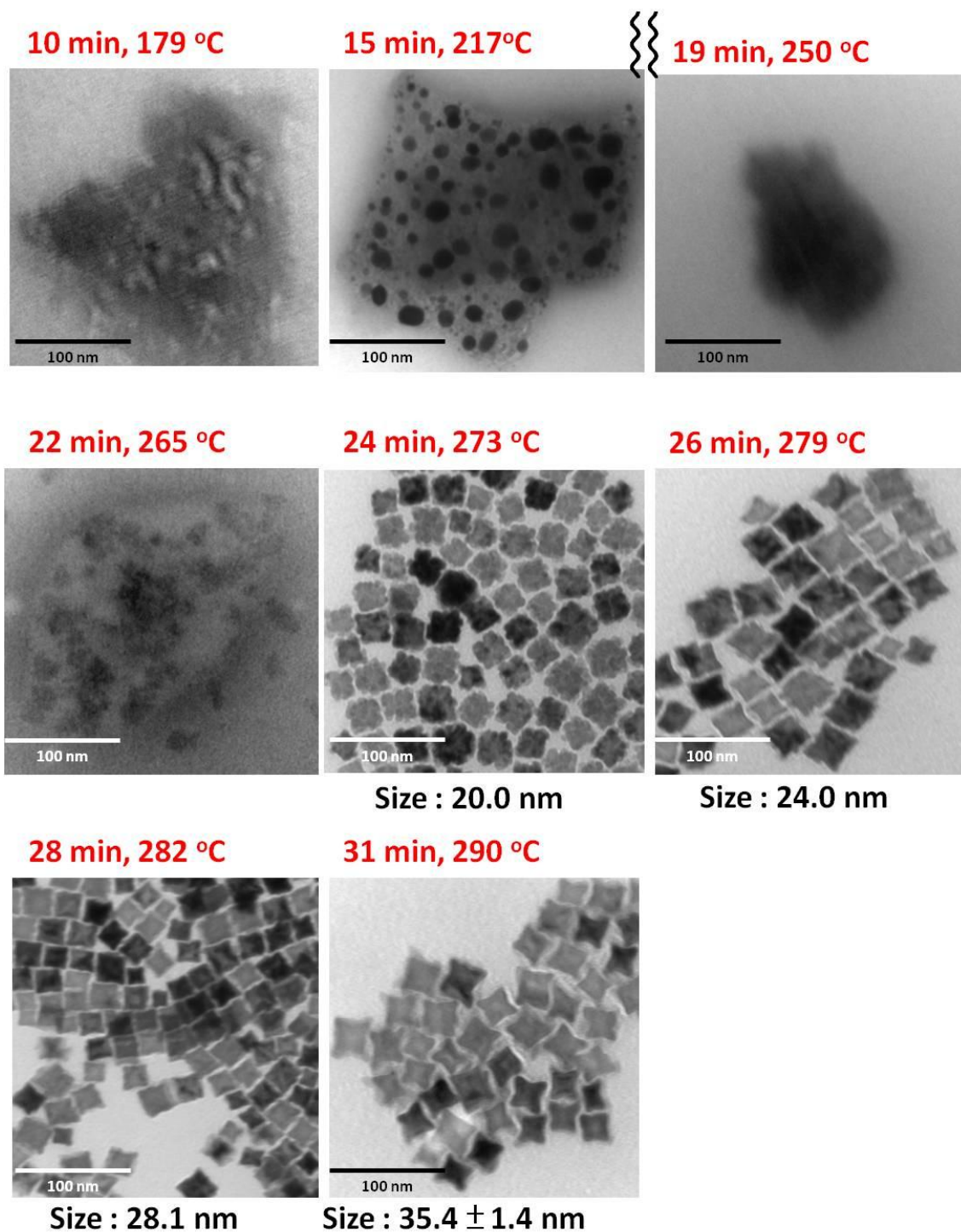


Figure S4. Time-dependent TEM images over the course of reaction time for $\text{Mn}_{0.29}\text{Fe}_{0.71}\text{O}$ concave nanoparticles obtained by the exposure of air 2 min. The respective TEM images were taken from reaction time of 10 min, 15 min, 19 min, 20 min, 22 min, 24 min, 26 min, 28 min, and 31 min. The symbol { { represents air exposure of reaction mixture.

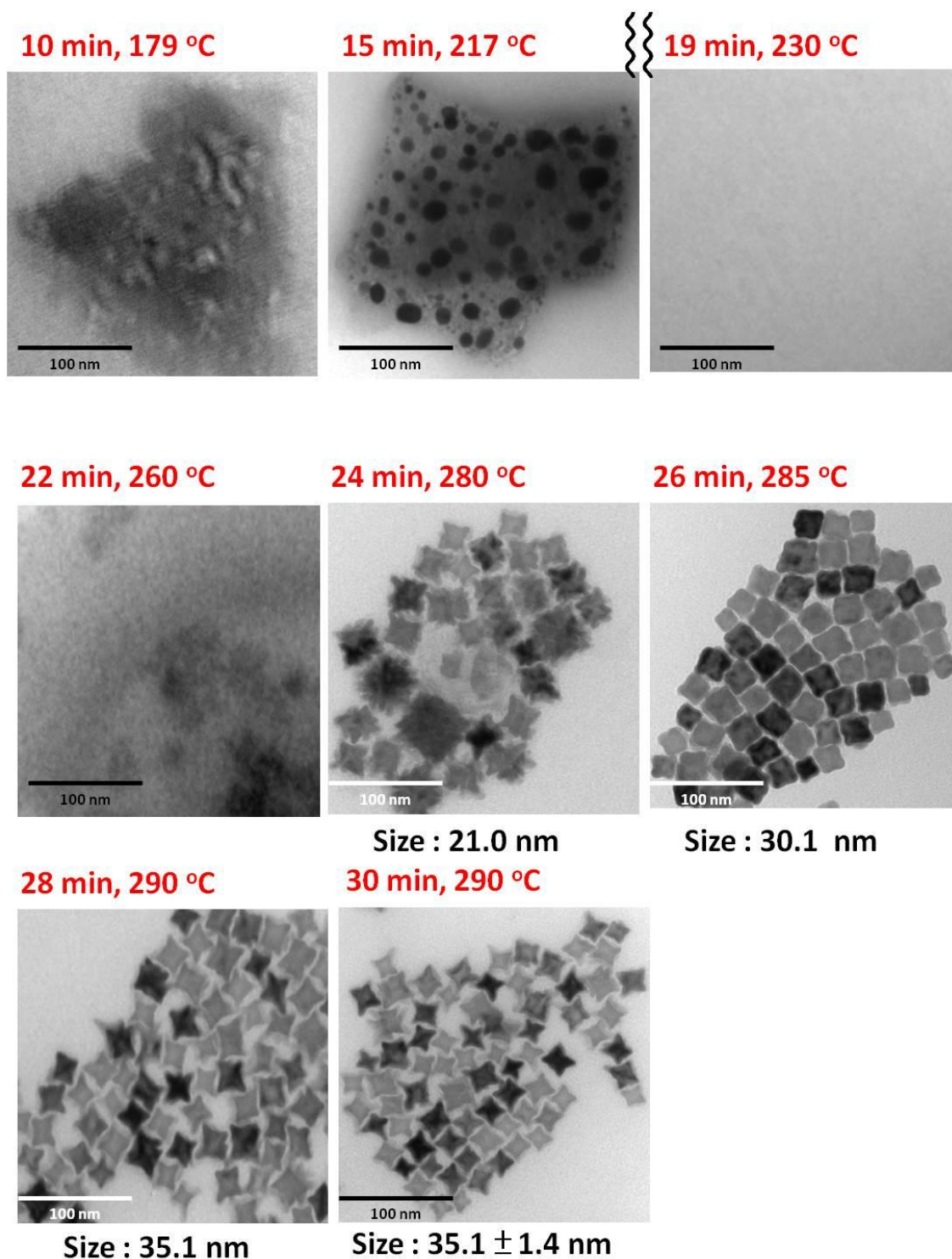


Figure S5. Time-dependent TEM images over the course of reaction time for $\text{Mn}_{0.33}\text{Fe}_{0.67}\text{O}$ concave nanoparticles obtained by the exposure of air 3.5 min. The respective TEM images were taken from reaction time of 10 min, 15 min, 19 min, 20 min, 22 min, 24 min, 26 min, 28 min, and 30 min. The symbol { { represents air exposure of reaction mixture.

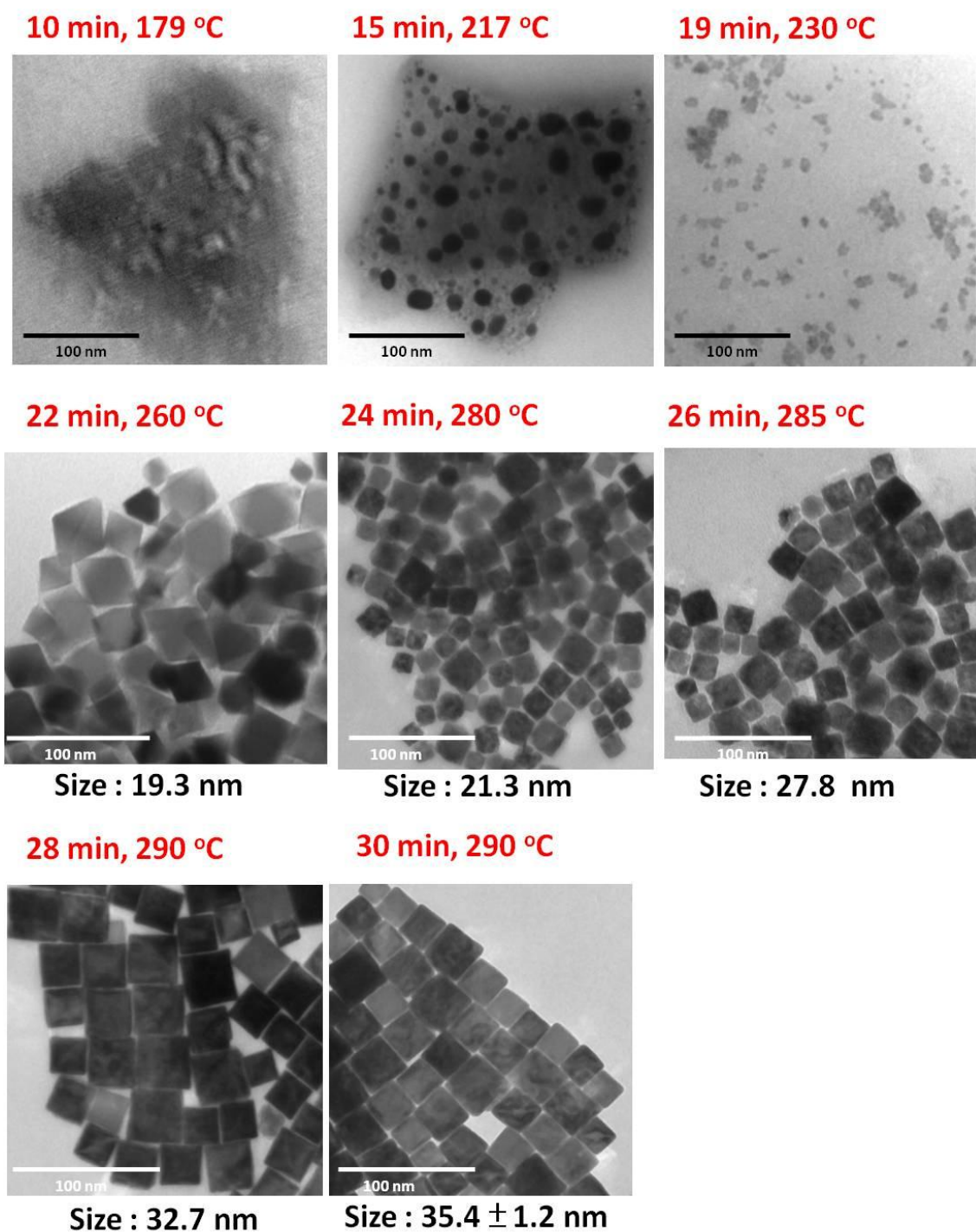


Figure S6. Time-dependent TEM images over the course of reaction time for $\text{Mn}_{0.05}\text{Fe}_{0.95}\text{O}$ cubic nanoparticles obtained in pure argon. The respective TEM images were taken from reaction time of 10 min, 15 min, 19 min, 20 min, 22 min, 24 min, 26 min, 28 min, and 30 min.

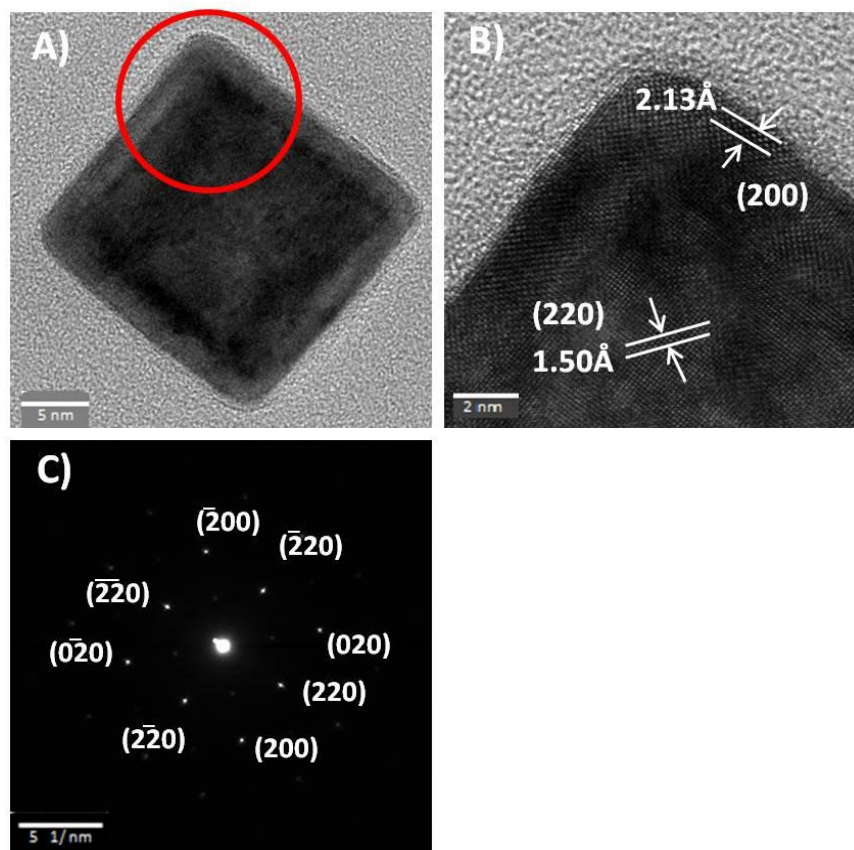


Figure S7. A) HRTEM image of the single cubic $\text{Mn}_{0.05}\text{Fe}_{0.95}\text{O}$ nanoparticle prepared under Ar condition. B) HRTEM image corresponding to the red circle in A. The fringe spacings display ca. 2.13 Å and 1.5 Å that correspond to the {200} and {220} basal planes of the NaCl-type FeO structure, respectively. C) Electron diffraction pattern of the single nanoparticle shown in A along the [001] zone axis.

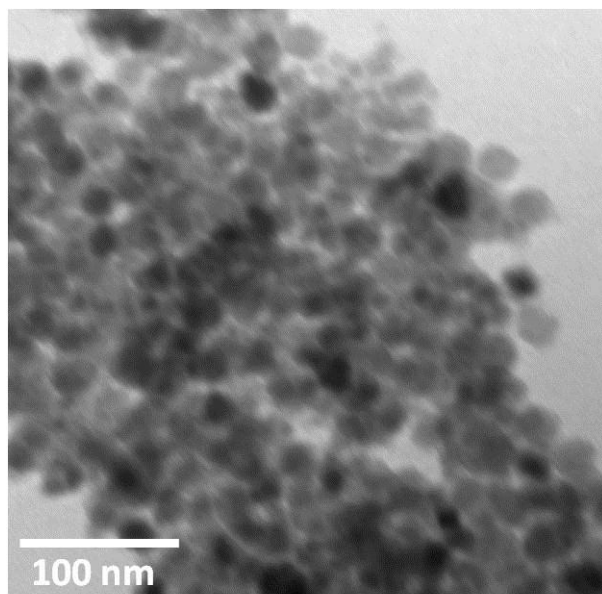


Figure 8. TEM images of the $\text{Mn}_x\text{Fe}_{1-x}\text{O}$ nanostructures synthesized under Ar without oleic acid.

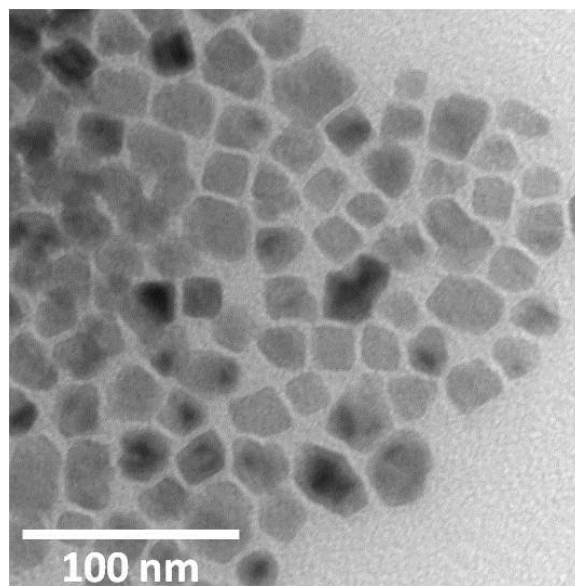


Figure 9. TEM images of the $\text{Mn}_x\text{Fe}_{1-x}\text{O}$ nanostructures obtained by an exposure of air for 5 min. Most of particles have size less than 30 nm.

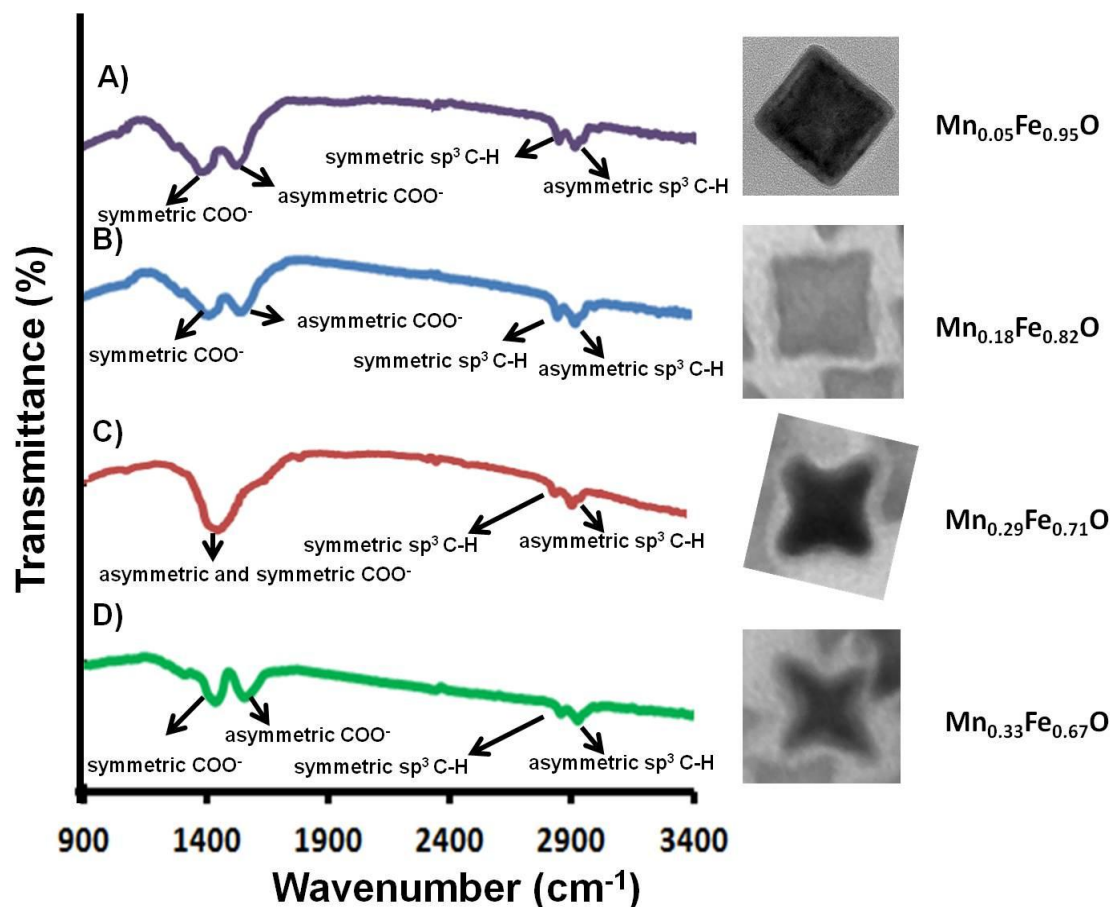


Figure S10. FT-IR spectra of A) Mn_{0.05}Fe_{0.95}O obtained from pure argon, B) Mn_{0.18}Fe_{0.82}O obtained by the exposure of air for 1.5 min, C) Mn_{0.29}Fe_{0.71}O obtained by the exposure of air for 2 min, and D) Mn_{0.33}Fe_{0.67}O obtained by the exposure of air 3.5 min. The absorption bands of ~1420 cm⁻¹ and ~1550 cm⁻¹ correspond to symmetric and asymmetric COO⁻ vibration, respectively, originating from oleic acid.¹

[1] Guilherme V. M. Jacintho, Alexandre G. Brolo, Paola Corio, Paulo A. Z. Suarez, and Joel C. Rubim *J. Phys. Chem. C* **2009**, *113*, 7684.

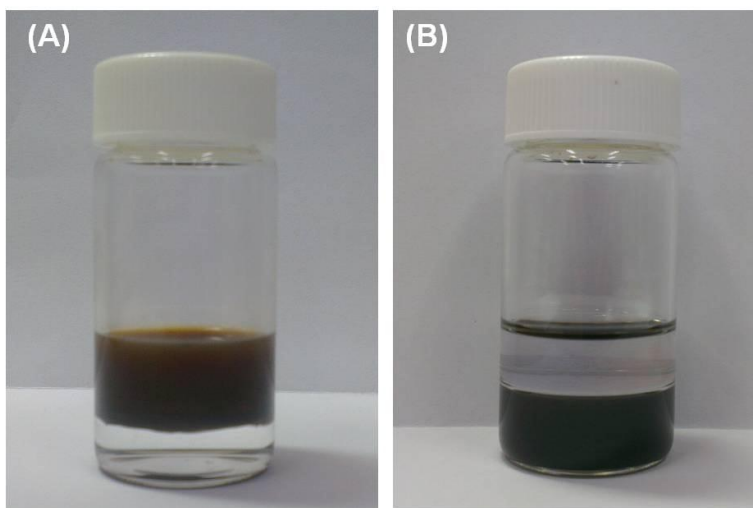


Figure S11. Photographs showing (A) oleic acid capped particles in brown color dispersed in toluene (upper layer) and (B) CTAB-modified particles dispersed in H₂O (bottom) as brown colloid.

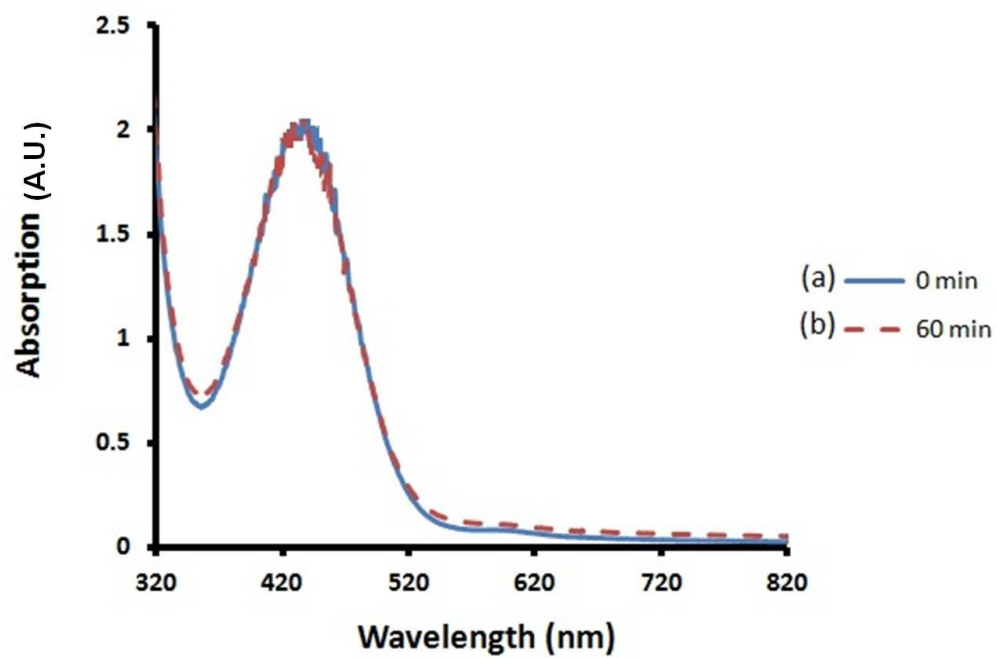


Figure S12. UV-vis spectra of xylenol orange oxidized by 30% H₂O₂ without catalysts measured at (a) beginning and (b) 60 min reaction.

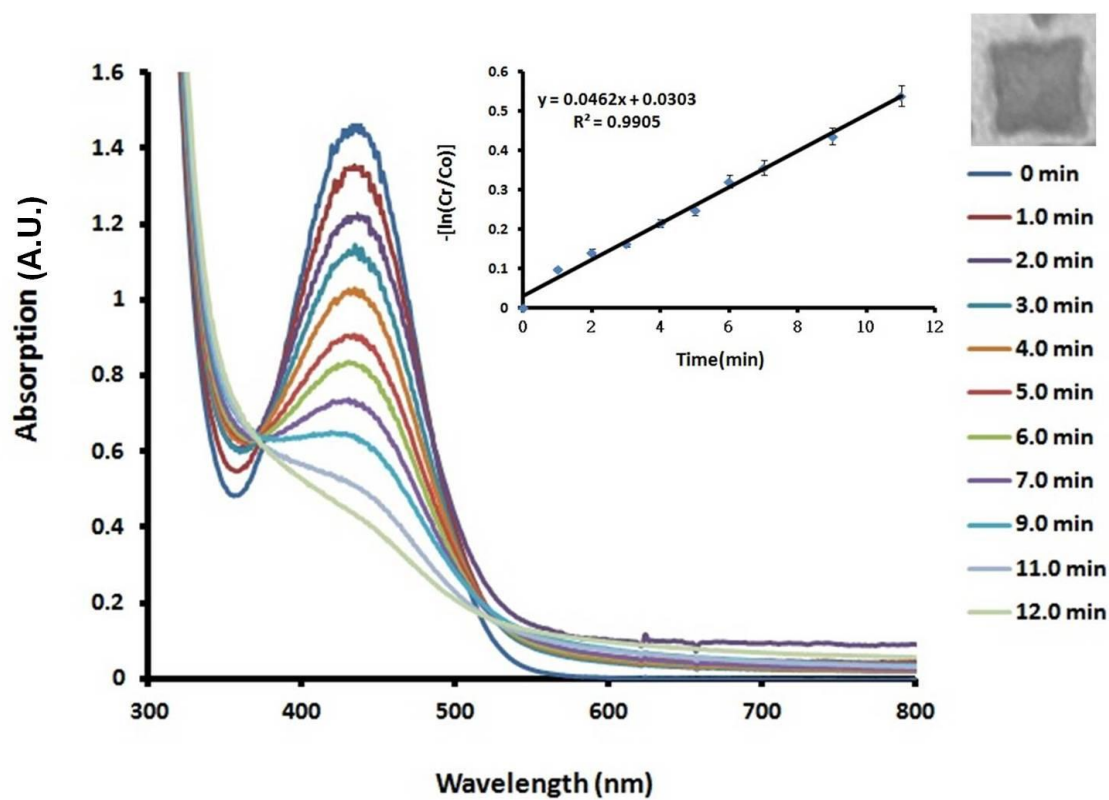


Figure S13. UV-vis spectra of xylol orange oxidized by 30% H_2O_2 at different reaction time interval using $Mn_{0.18}Fe_{0.82}O$ prepared under 1.5 min air exposure as catalyst. The inset shows the logarithmic plot of concentration of xylol orange as a function of degradation time.

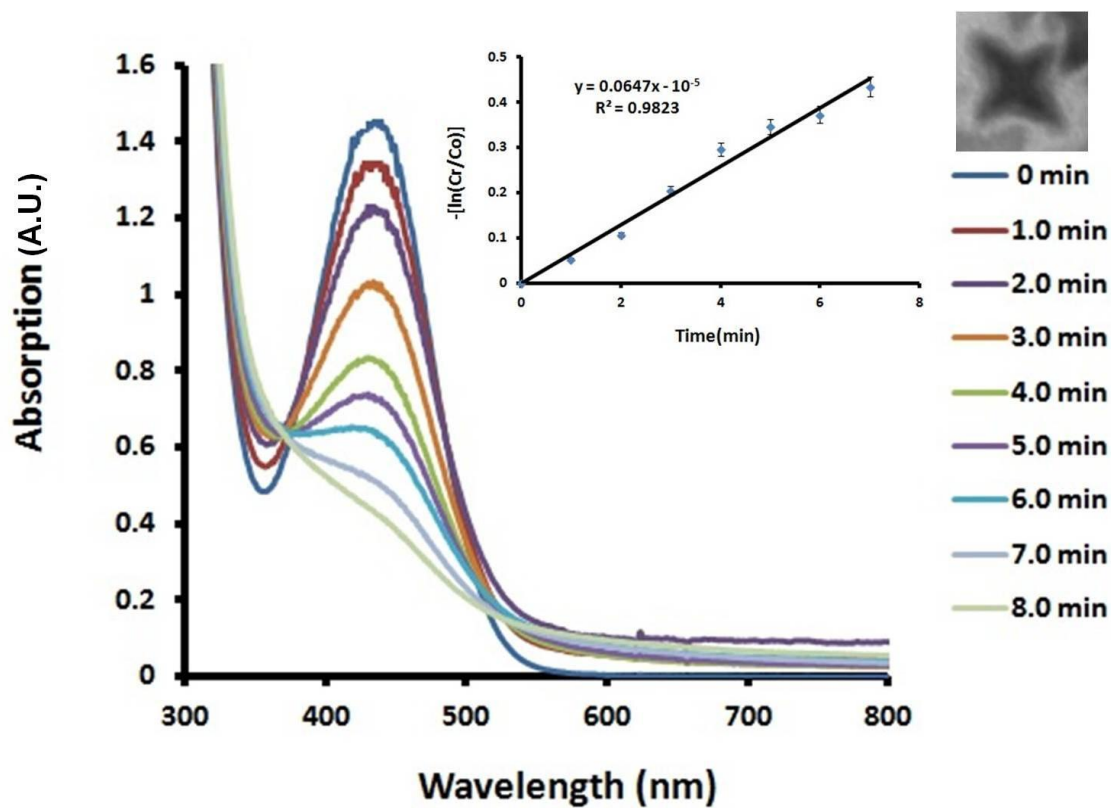


Figure S14. UV-vis spectra of xylol orange oxidized by 30% H_2O_2 at different reaction time interval using $\text{Mn}_{0.33}\text{Fe}_{0.67}\text{O}$ prepared under 3.5 min air exposure as catalyst. The inset shows the logarithmic plot of concentration of xylol orange as a function of degradation time.

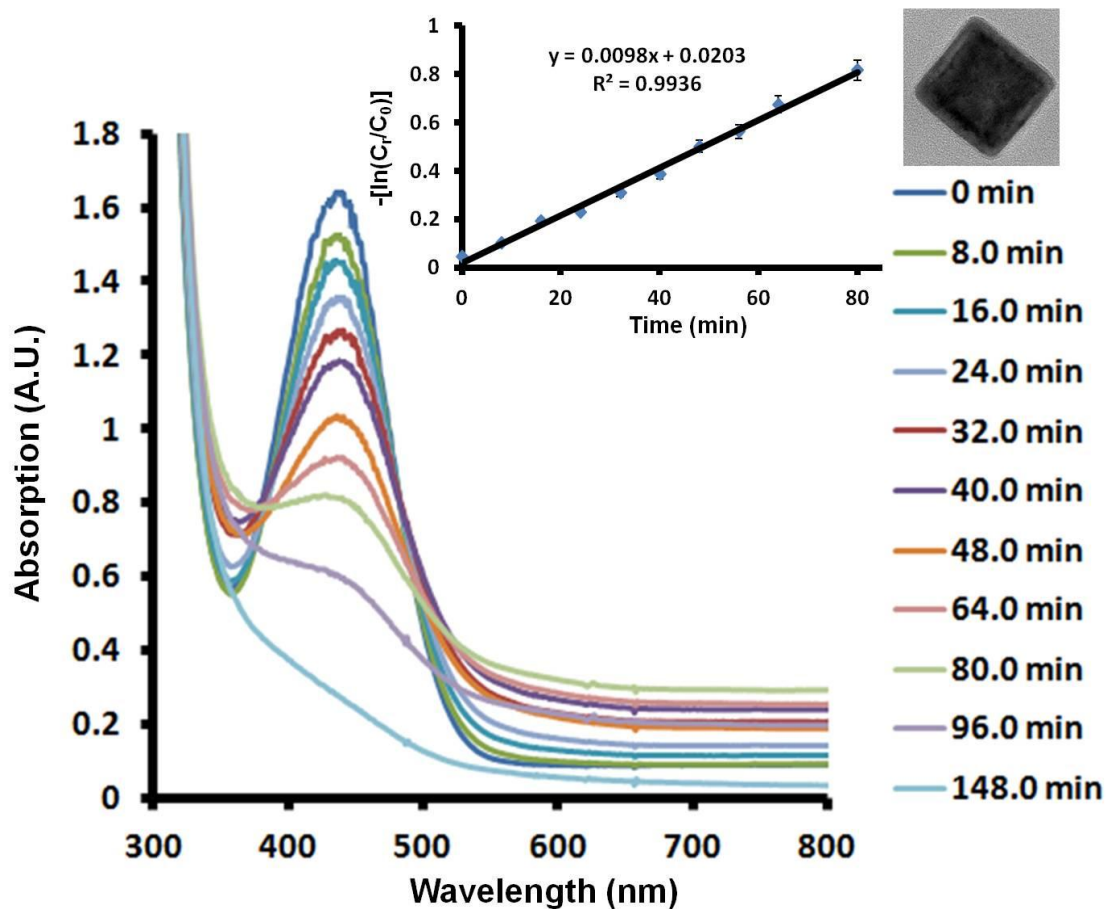


Figure S15. UV-vis spectra of xylenol orange oxidized by 30% H_2O_2 at different reaction time interval using $\text{Mn}_{0.05}\text{Fe}_{0.95}\text{O}$ prepared in pure argon as catalyst. The inset shows the logarithmic plot of concentration of xylenol orange as a function of degradation time.

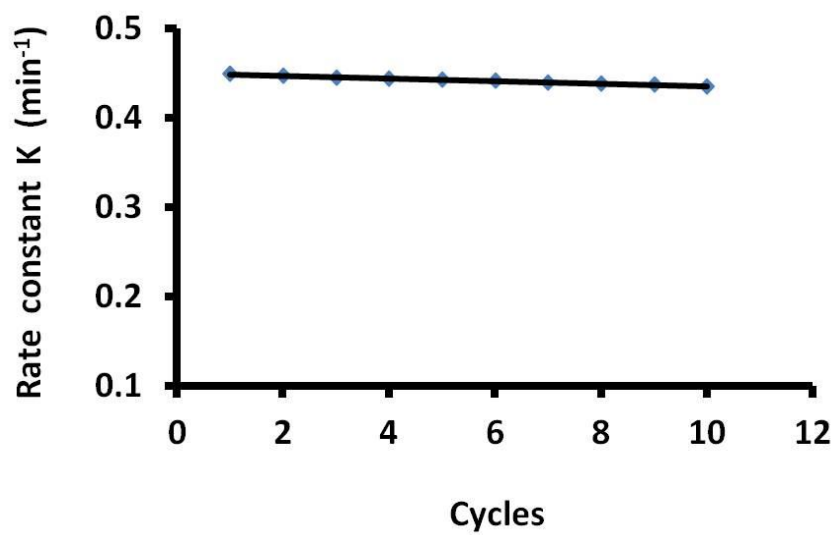


Figure S16. The plot shows the rate constant as a function of the number of recycle when using $\text{Mn}_{0.29}\text{Fe}_{0.71}\text{O}$ as catalyst.