

## Supporting Information

# Fabrication of Three-Dimensional Flower-Like Heterogeneous Fe<sub>3</sub>O<sub>4</sub>/Fe Particles with Tunable Chemical Composition and Microwave Absorption Performance

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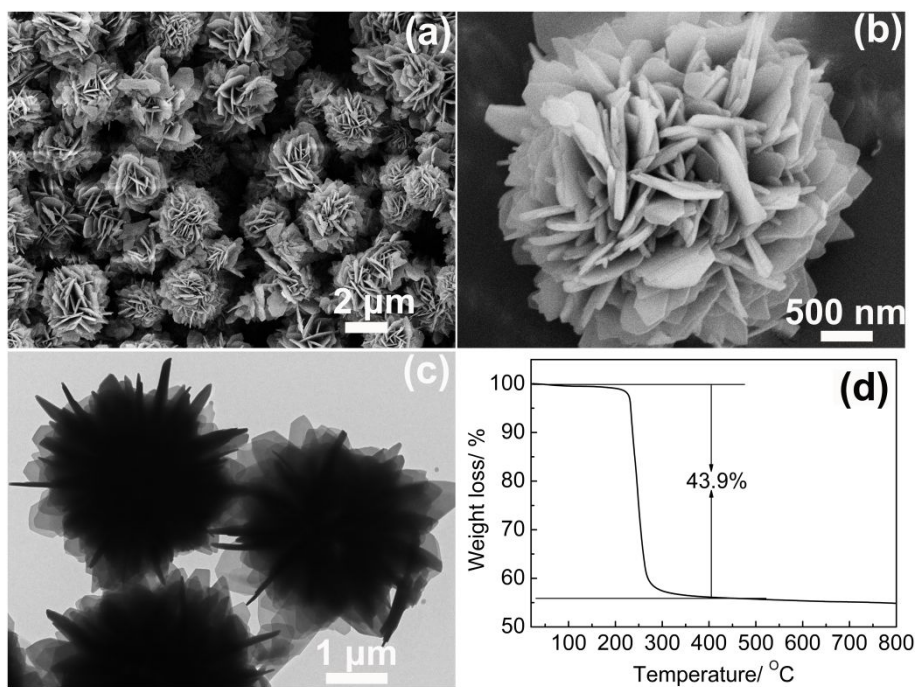
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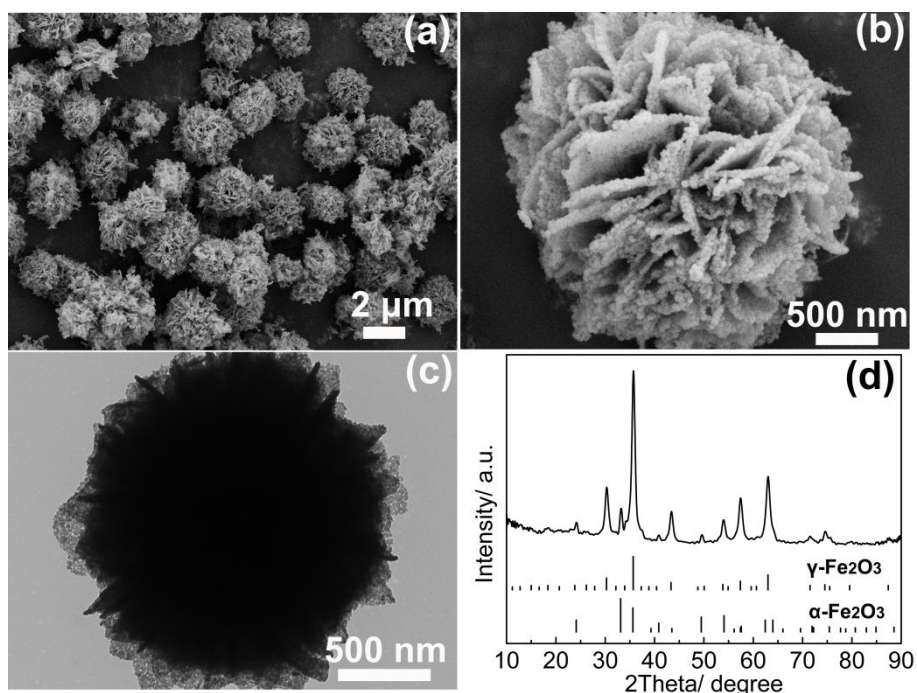
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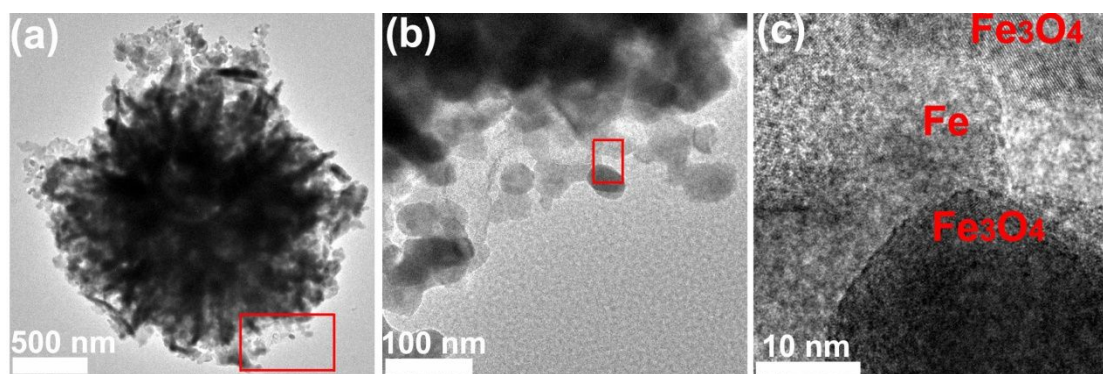
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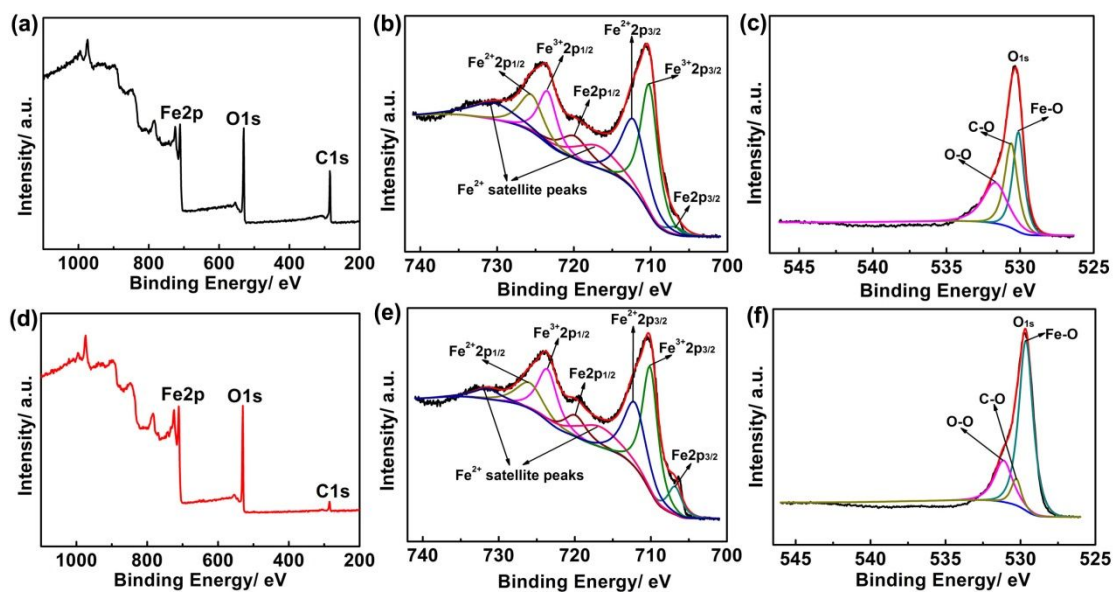
**Figure S1.** SEM (a, b), TEM (c) images, and TGA curve (d) of iron alkoxides precursor.



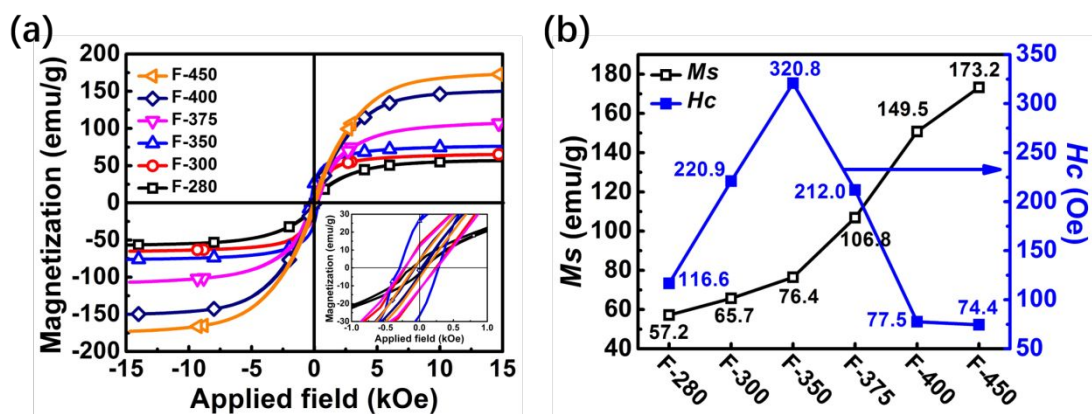
**Figure S2.** SEM (a, b), TEM (c) images, and XRD pattern (d) of iron oxides obtained by pyrolysis of precursor in air at 400 °C.



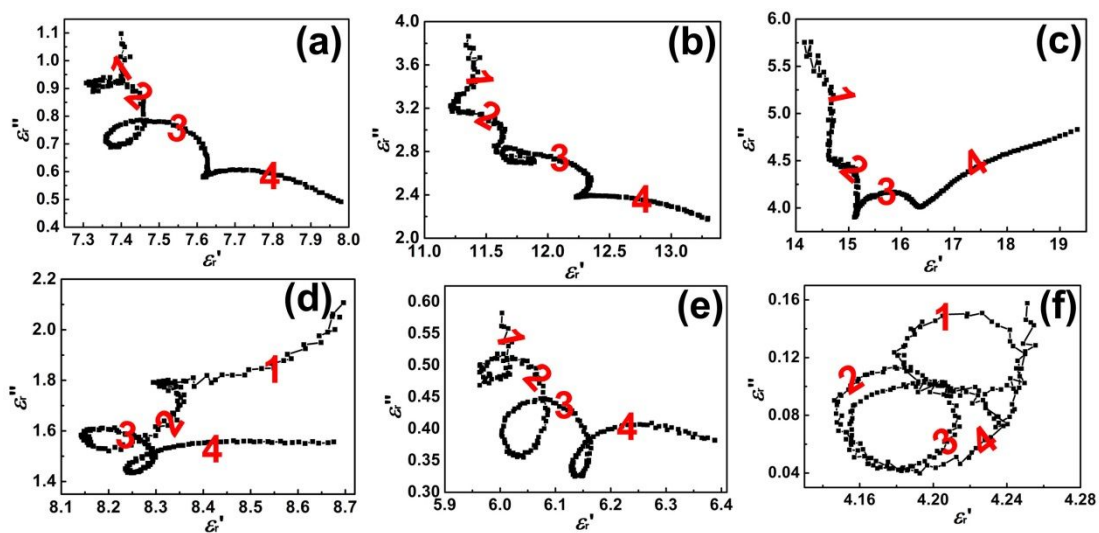
**Figure S3.** TEM image of F-350 (a), local magnification (b) of rectangle part in (a), HRTEM image (c) of rectangle part in (b).



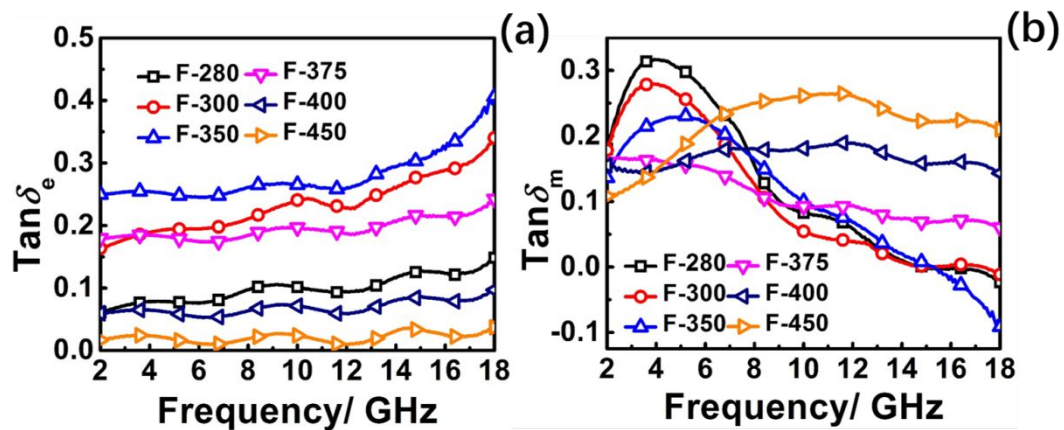
**Figure S4.** XPS survey (a), Fe 2p (b), and O 1s spectra (c) of F-350, and XPS survey (d), Fe 2p (e), and O 1s spectra (f) of F-450.



**Figure S5.** Magnetic hysteresis loops of 3D flower-like  $\text{Fe}_3\text{O}_4/\text{Fe}$  at room temperature (a), and  $M_s$  and  $H_c$  values (b) of all samples. Inset in (a) is a magnification of magnetic hysteresis loops.

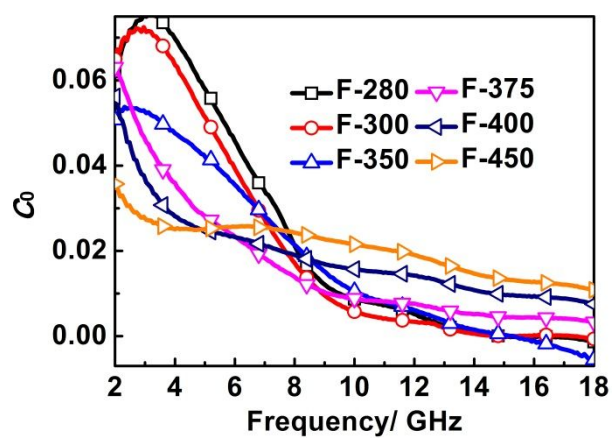


**Figure S6.** Cole-Cole plots of F-280 (a), F-300 (b), F-350 (c), F-375 (d), F-400 (e), and F-450 (f).

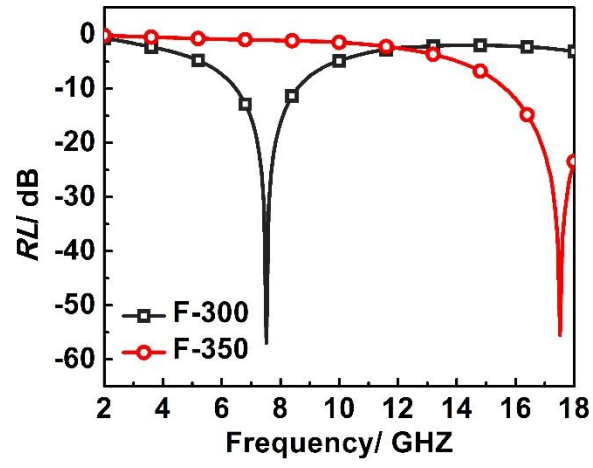


**Figure S7.** Frequency-dependent  $\tan \delta_e$  (a) and  $\tan \delta_m$  (b) of flower-like  $\text{Fe}_3\text{O}_4/\text{Fe}$ .

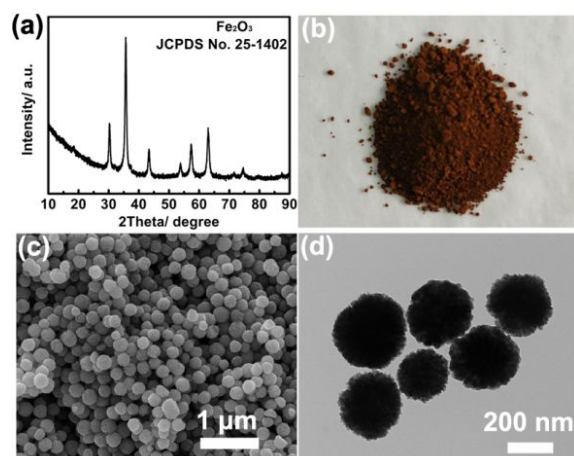




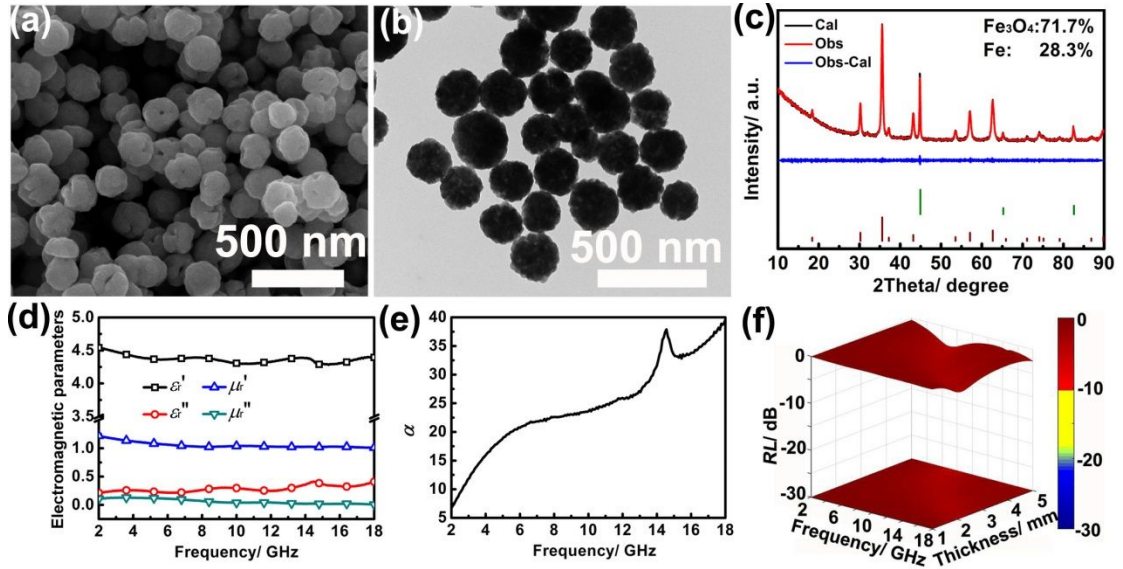
**Figure S8.**  $C_0$  values of  $\text{Fe}_3\text{O}_4/\text{Fe}$  composites in the frequency range of 2.0-18.0 GHz.



**Figure S9.** Reflection loss curves of F-300 (d=2.97 mm) and F-350 (d=1.19 mm).



**Figure S10.** XRD (a), optical image (b), SEM image (c), and TEM image (d) of Fe<sub>2</sub>O<sub>3</sub> spheres.



**Figure S11.** SEM image (a), TEM image (b), XRD pattern (c),  $\epsilon_r$  and  $\mu_r$  (d), attenuation constant (e), and reflection loss map of C-350 (f).

**Table S1.** ICP results of Fe<sub>3</sub>O<sub>4</sub>/Fe composites.

sample	Final concentration (mg/L)	ICP tested value (mg/L)	Mass ratio (%)	
			Fe	Fe <sub>3</sub> O <sub>4</sub>
F-280	5.00	3.636	0	100
F-300	5.00	3.907	20.1	79.9
F-350	5.00	4.018	28.4	71.6
F-375	5.00	4.215	43.0	57.0
F-400	5.00	4.899	93.8	6.2
F-450	5.00	4.983	100	0