

Solution Synthesis of Pure 2H CuFeO₂ at Low Temperatures

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Table S1. Reaction details for synthesizing CuFeO₂ crystals at different reaction time and temperature by using NaOH as base.

No.	CuI mmol	FeCl ₃ ·6H ₂ O mmol	NaOH mmol	Temp. °C	Time h	Phase composition
1	0.75	0.75	5.5	150	2	CuFeO ₂ (3R ^a , 2H ^b)
2	0.75	0.75	5.5	150	8	CuFeO ₂ (3R ^a , 2H ^b)
3	0.75	0.75	5.5	150	24	CuFeO ₂ (3R ^a , 2H ^b)
4	0.75	0.75	5.5	150	48	CuFeO ₂ (3R ^a , 2H ^b)
5	0.75	0.75	5.5	80	24	CuFeO ₂ (3R ^a , 2H ^b), α-Fe ₂ O ₃ , Fe(OH, I) _x , Cu ₂ O
6	0.75	0.75	5.5	90	24	CuFeO ₂ (3R ^a , 2H ^b)
7	0.75	0.75	5.5	120	24	CuFeO ₂ (3R ^a , 2H ^b)
8	0.75	0.75	5.5	190	24	CuFeO ₂ (3R ^a , 2H ^b)

^a majority phase, ^b minor phase

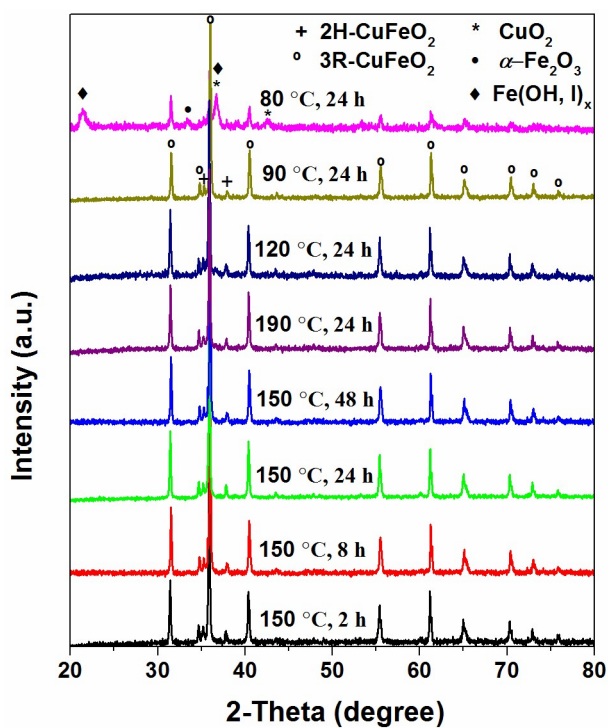


Figure S1. PXRD spectra of CuFeO_2 synthesized by using CuI (0.75 mmol), $\text{FeCl}_3 \cdot 6\text{H}_2\text{O}$ (0.75 mmol), H_2O (3.5 mL), NaOH (5.5 mmol) with different reaction time and temperature.

Table S2. Reaction details for synthesizing CuFeO_2 crystals with different amount of NaOH at 190 °C for 2 h.

No.	CuI mmol	$\text{FeCl}_3 \cdot 6\text{H}_2\text{O}$ mmol	NaOH mmol	Temp. °C	Time h	Phase composition
1	0.75	0.75	3.00	190	2	$\alpha\text{-Fe}_2\text{O}_3$, Cu_2O
2	0.75	0.75	3.35	190	2	CuFeO_2 (3R ^a , 2H ^b)
3	0.75	0.75	4.75	190	2	CuFeO_2 (3R ^a , 2H ^b)
4	0.75	0.75	5.50	190	2	CuFeO_2 (3R ^a , 2H ^b)
5	0.75	0.75	13.5	190	2	CuFeO_2 (3R ^a , 2H ^b)

^a majority phase, ^b minor phase

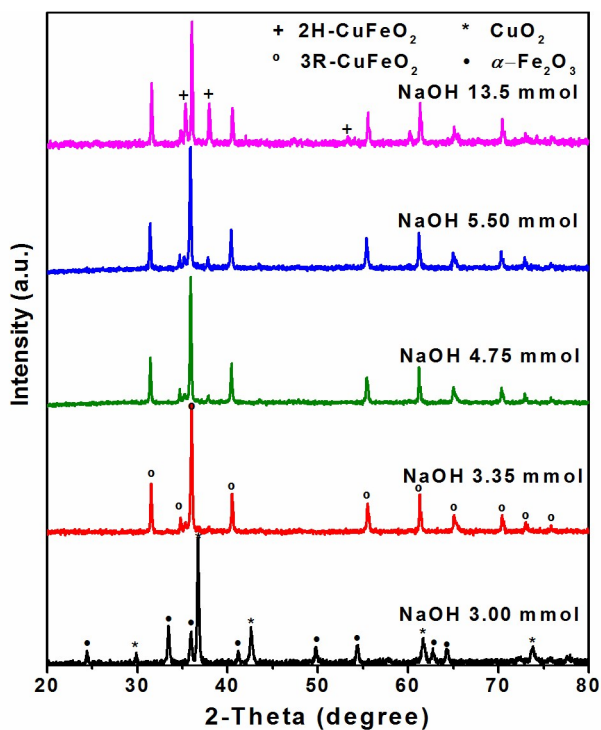


Figure S2. PXRD spectra of CuFeO_2 synthesized by using CuI (0.75 mmol), $\text{FeCl}_3 \cdot 6\text{H}_2\text{O}$ (0.75 mmol), H_2O (3.5 mL) at 190 °C for 2 h with different amount of NaOH.

Table S3. Reaction details for synthesizing CuFeO_2 crystals with different additives.

No.	CuI mmol	$\text{FeCl}_3 \cdot 6\text{H}_2\text{O}$ mmol	NaOH mmol	Temp. °C	Time h	Phase composition	additives
1	0.75	0.75	4.75	120	24	CuFeO_2 (3R ^a , 2H ^b)	PEG 6000 (0.75 mmol) ^c
2	0.75	0.75	4.75	120	24	CuFeO_2 (3R ^a , 2H ^b)	PEG 6000 (3.75 mmol) ^c
3	0.75	0.75	6.50 ^d	120	24	CuFeO_2 (3R ^a , 2H ^b)	Propanoic acid (1.75 mmol)
4	0.75	0.75	6.50 ^d	120	24	CuFeO_2 (3R ^a , 2H ^b)	5-phenylpentanoic acid (1.75 mmol)

^a majority phase, ^b minor phase, ^c the molar amount was calculated based on repeat unit of

PEG 6000, ^d the effective NaOH was 4.75 mmol after reacting with carboxylic acid.

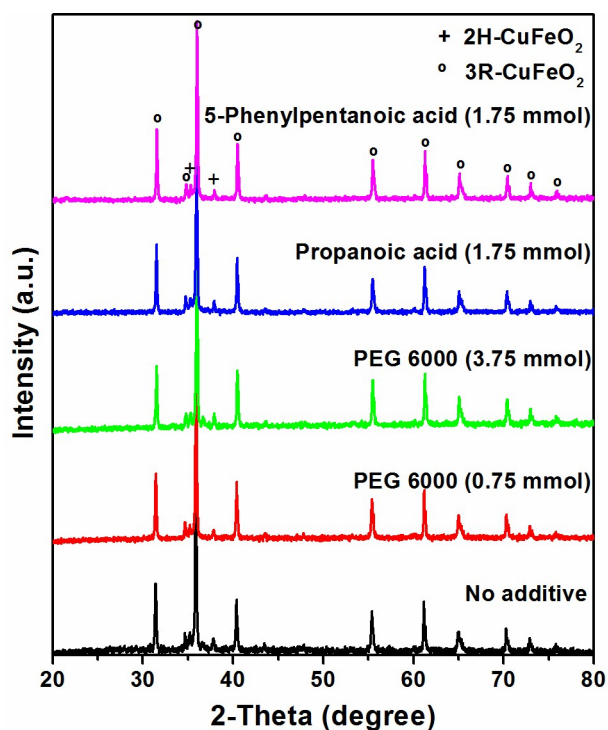


Figure S3. PXRD spectra of CuFeO_2 synthesized by using CuI (0.75 mmol), $\text{FeCl}_3 \cdot 6\text{H}_2\text{O}$ (0.75 mmol), H_2O (3.5 mL), NaOH (effective amount 4.75 mmol), at 120 °C for 24 h with different additives.

Table S4. Reaction details for synthesizing CuFeO_2 crystals with different amount of NaOH at 120 °C for 24 h.

No.	CuI mmol	$\text{FeCl}_3 \cdot 6\text{H}_2\text{O}$ mmol	NaOH mmol	Temp. °C	Time h	Phase composition
1	0.75	0.75	6.5	120	24	CuFeO_2 (3R ^a , 2H ^b)
2	0.75	0.75	13.5	120	24	CuFeO_2 (3R ^a , 2H ^b)
3	0.75	0.75	24.0	120	24	CuFeO_2 (3R ^b , 2H ^a)
4	0.75	0.75	38.0	120	24	CuFeO_2 (3R ^b , 2H ^a)
5	0.75	0.75	46.8	120	24	CuFeO_2 (3R ^b , 2H ^a)
6	0.75	0.75	55.5	120	24	CuFeO_2 (2H)

^a majority phase, ^b minor phase

Table S5. Reaction details for synthesizing CuFeO_2 crystals with high concentration of NaOH at 120 °C for different reaction time.

No.	CuI mmol	FeCl ₃ ·6H ₂ O mmol	NaOH mmol	Temp. °C	Time h	Phase composition
1	0.75	0.75	55.5	120	1.5	CuFeO ₂ (2H), Cu ₂ O
2	0.75	0.75	55.5	120	2	CuFeO ₂ (2H)
3	0.75	0.75	55.5	120	6	CuFeO ₂ (2H)
4	0.75	0.75	55.5	120	12	CuFeO ₂ (2H)
5	0.75	0.75	55.5	120	24	CuFeO ₂ (2H)
6	0.75	0.75	55.5	120	48	CuFeO ₂ (2H)

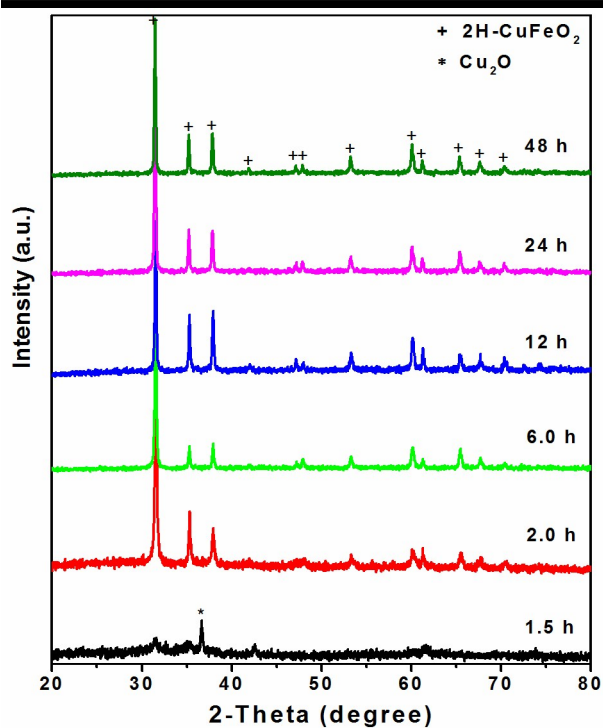


Figure S4. PXRD spectra of CuFeO₂ synthesized by using CuI (0.75 mmol), FeCl₃·6H₂O (0.75 mmol), H₂O (3.5 mL), NaOH (55.5 mmol) with different reaction time at 120 °C.

Table S6. Reaction details for synthesizing CuFeO₂ crystals at different concentration of reactants and reaction temperature.

No.	CuI mmol	FeCl ₃ ·6H ₂ O mmol	NaOH mmol	Temp. °C	Time h	Phase composition	crystallite size nm
1	0.75	0.75	55.5	100	17	CuFeO ₂ (2H)	42
2	0.75	0.75	55.5	160	2	CuFeO ₂ (2H)	58
3	0.75	0.75	55.5	200	2	CuFeO ₂ (2H)	73
4	0.75	0.75	55.5	120	3	CuFeO ₂ (2H)	42
5	0.35	0.35	55.5	120	3	CuFeO ₂ (2H)	35
6	0.25	0.25	55.5	120	3	CuFeO ₂ (2H)	36

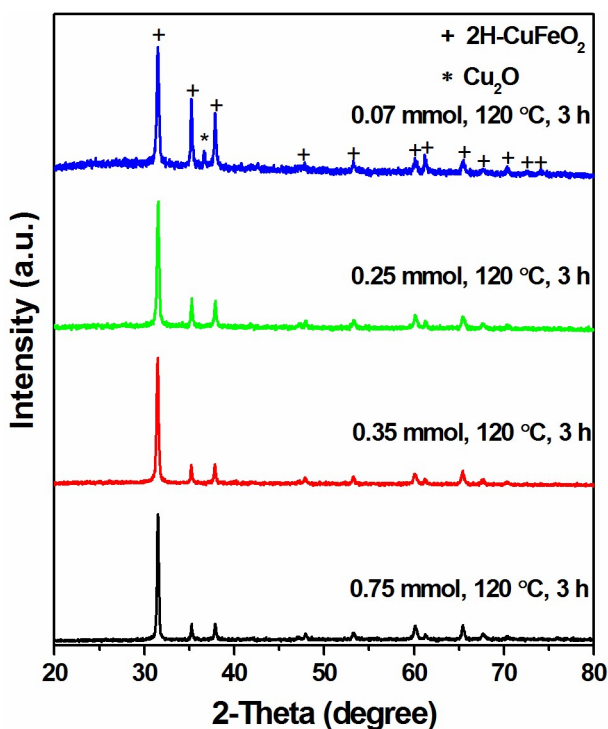


Figure S5. PXRD spectra of CuFeO₂ synthesized by using H₂O (3.5 mL), NaOH (55.5 mmol) at 120 h for 3 h with different amount of CuI and FeCl₃·6H₂O.

Table S7. Reaction details for synthesizing CuFeO₂ crystals at different concentration of reactants and reaction time with high concentration of KOH at 100 °C.

No.	CuI mmol	FeCl ₃ ·6H ₂ O mmol	KOH mmol	Temp. °C	Time h	Phase composition
1	0.35	0.35	55.5	100	24	CuFeO ₂ (2H ^a , 3R ^b)
2	0.525	0.525	55.5	100	24	CuFeO ₂ (2H ^a , 3R ^b)
3	0.700	0.700	55.5	100	24	CuFeO ₂ (2H)
4	1.05	1.05	55.5	100	24	CuFeO ₂ (2H)
5	0.35	0.35	55.5	100	6	CuFeO ₂ (2H), Cu ₂ O
6	0.35	0.35	55.5	100	12	CuFeO ₂ (2H ^a , 3R ^b)
7	0.35	0.35	55.5	100	18	CuFeO ₂ (2H ^a , 3R ^b)
8	0.35	0.35	55.5	100	36	CuFeO ₂ (2H ^a , 3R ^b)
9	0.35	0.35	55.5	100	48	CuFeO ₂ (2H ^a , 3R ^b)

^a majority phase, ^b minor phase

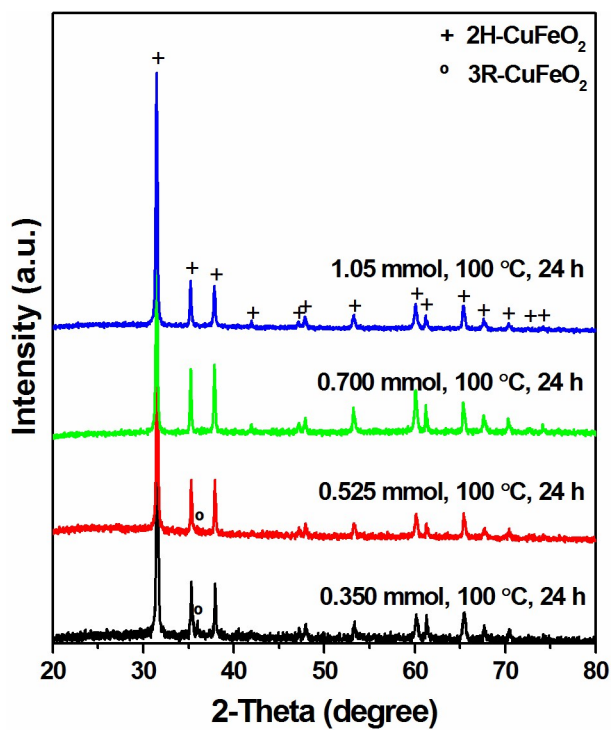


Figure S6. PXRD spectra of CuFeO_2 synthesized by using H_2O (3.5 mL), KOH (55.5 mmol) at $100\text{ }^\circ\text{C}$ for 24 h with different amount of CuI and $\text{FeCl}_3 \cdot 6\text{H}_2\text{O}$.

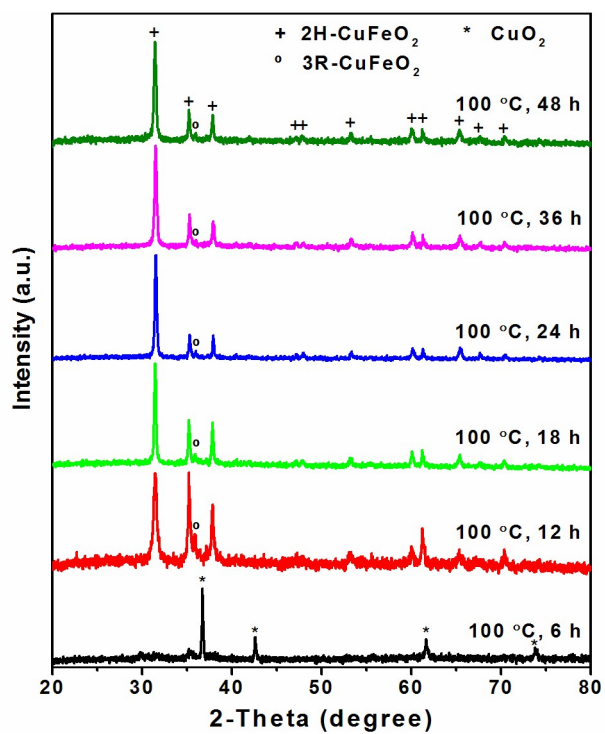


Figure S7. PXRD spectra of CuFeO_2 synthesized by using CuI (0.35 mmol), $\text{FeCl}_3 \cdot 6\text{H}_2\text{O}$ (0.35 mmol), H_2O (3.5 mL), KOH (55.5 mmol) with different reaction time at 100 °C.

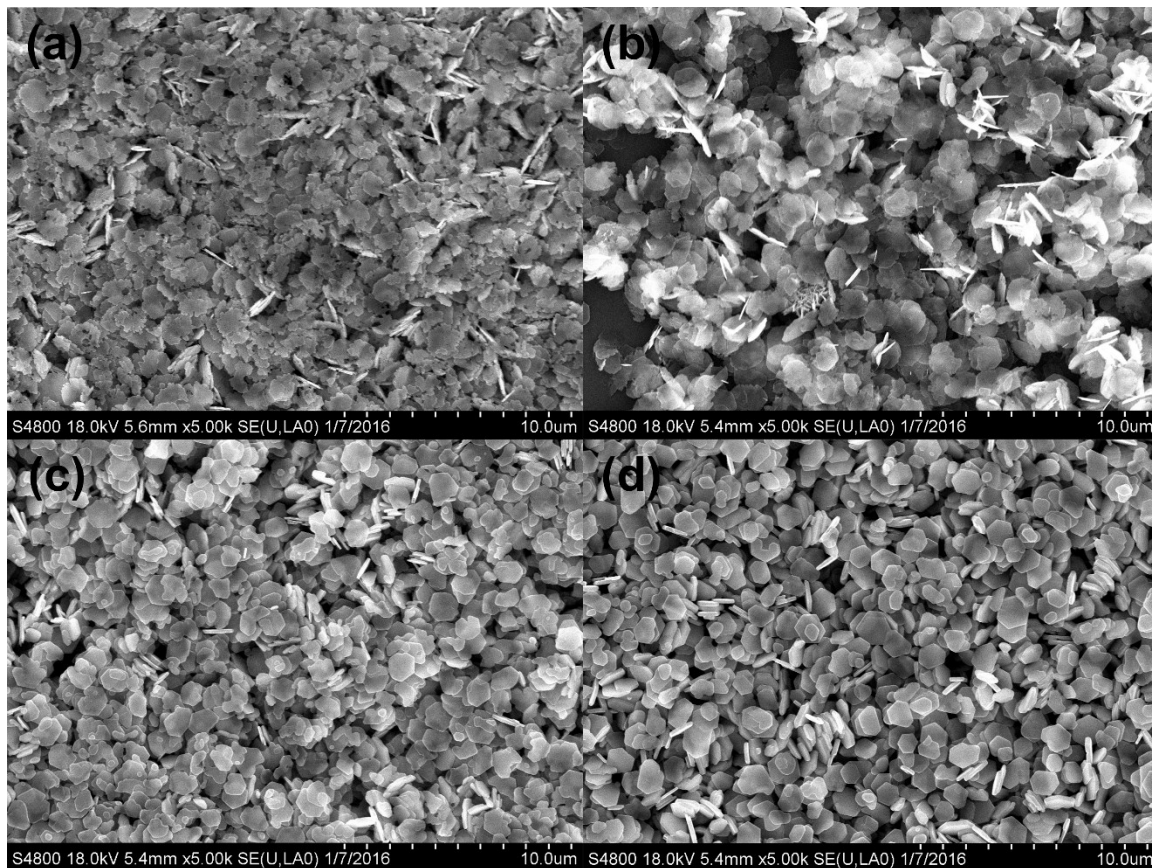


Figure S8. SEM images of CuFeO_2 synthesized by using CuI (0.75 mmol), $\text{FeCl}_3 \cdot 6\text{H}_2\text{O}$ (0.75 mmol), H_2O (3.5 mL), NaOH (55.5 mmol) at (a) 100 °C, (b) 120 °C, (c) 160 °C and (d) 200 °C, respectively.

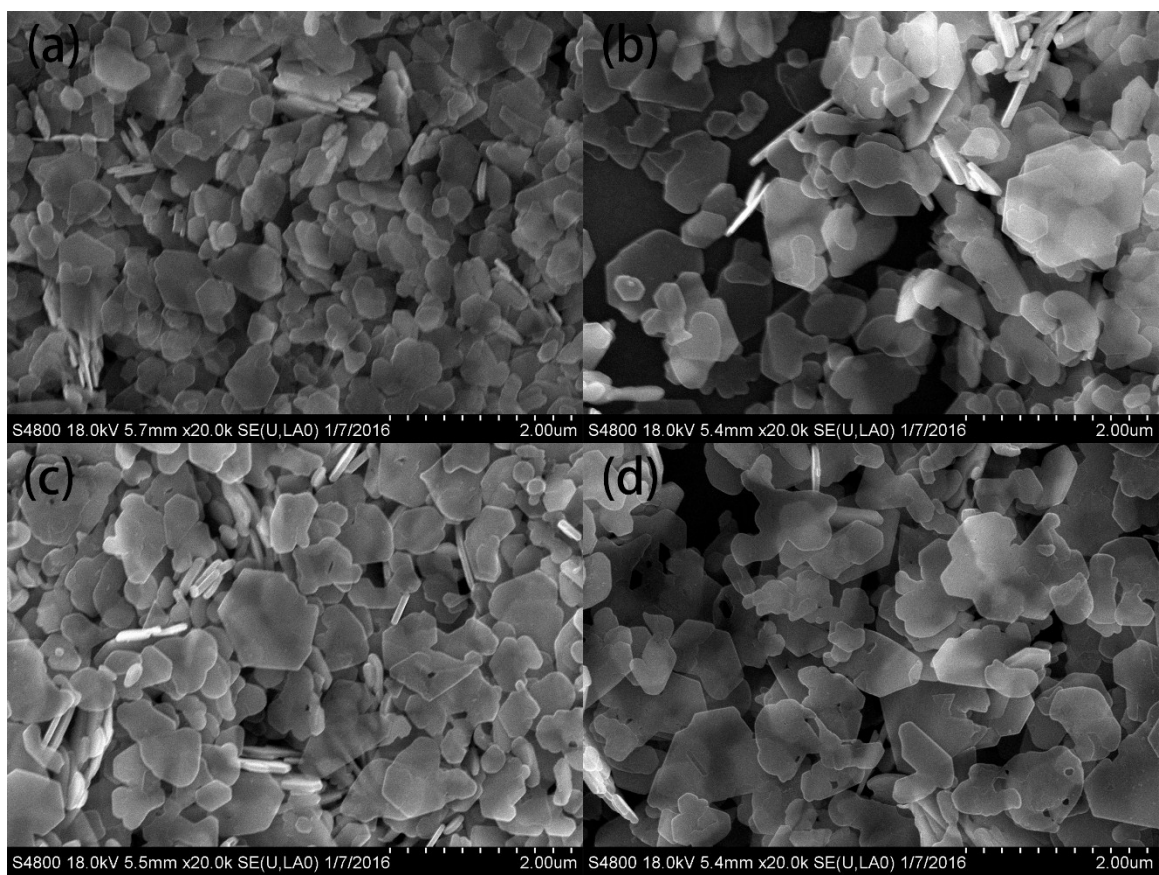


Figure S9. SEM images (20K times magnification) of CuFeO_2 synthesized by using H_2O (3.5 mL), KOH (55.5 mmol) at 100 h for 24 h with different amount of CuI and $\text{FeCl}_3 \cdot 6\text{H}_2\text{O}$, (a) 0.35 mmol, (b) 0.525 mmol, (c) 0.700 mmol, and (d) 1.05 mmol, respectively.

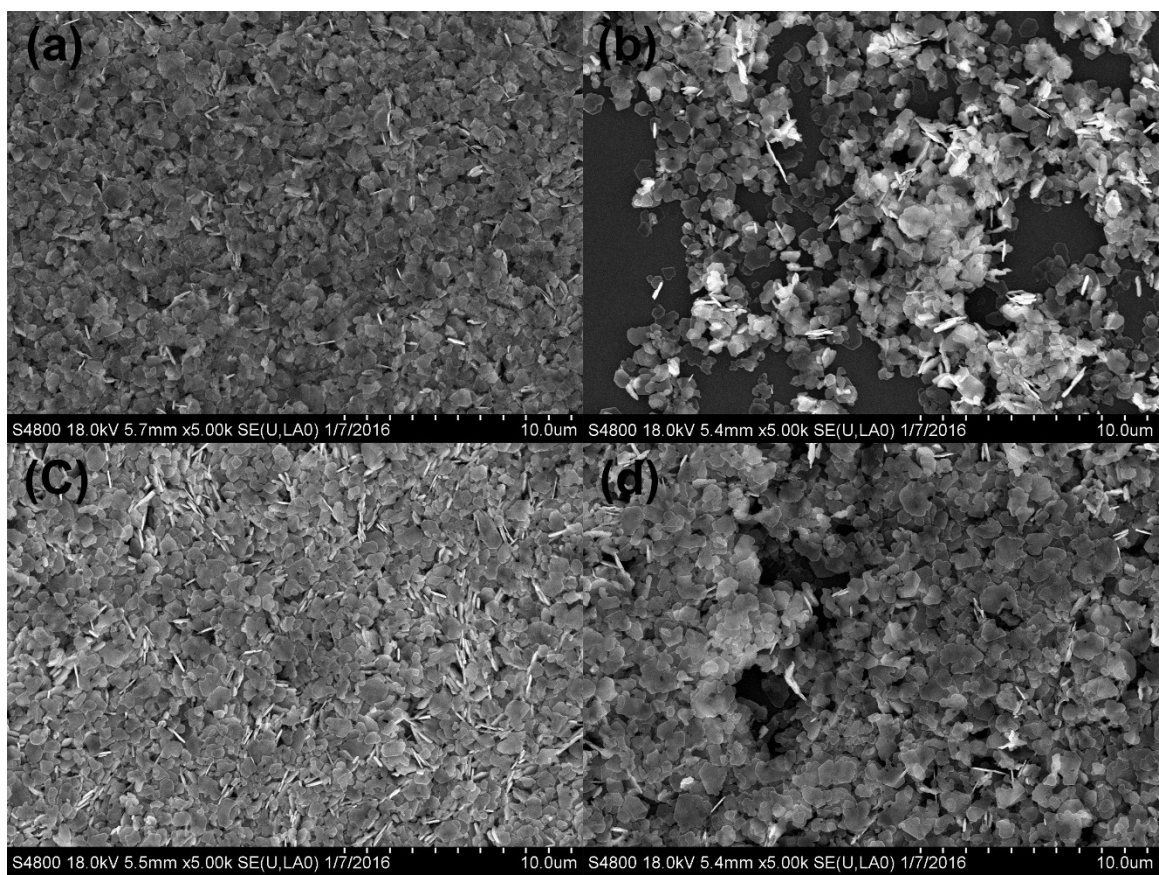


Figure S10. SEM images (5K times magnification) of CuFeO_2 synthesized by using H_2O (3.5 mL), KOH (55.5 mmol) at 100 h for 24 h with different amount of CuI and $\text{FeCl}_3 \cdot 6\text{H}_2\text{O}$, (a) 0.35 mmol, (b) 0.525 mmol, (c) 0.700 mmol, and (d) 1.05 mmol, respectively.

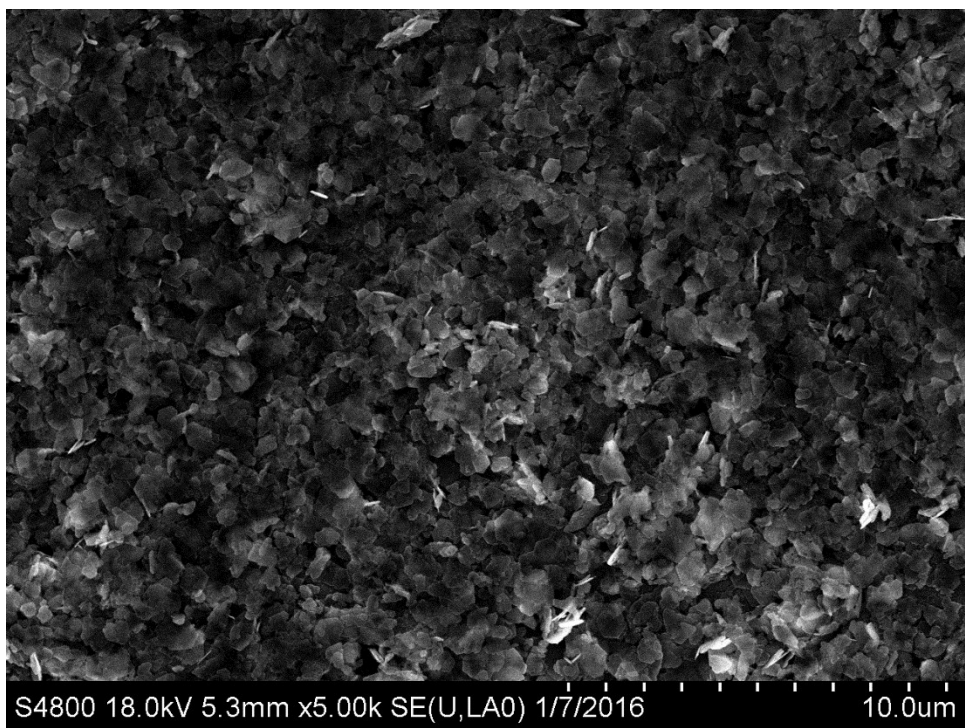


Figure S11. SEM image of 2H-CuFeO₂ film on glass/ITO/ZnO substrate.