SUPPORTING INFORMATION

Response Surface Analysis of $CuInSe_2$ Nanoparticle Synthesis: Unravelling the Interplay of Temperature, Time, and Ligand Composition for Size Control

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Table S1. Variables and levels used in the Box-Behnken design for $CuInSe_2$ nanoparticle synthesis.

Variable	Name	Units	Low (-1)	Center (0)	High (+1)
A	Temperature	°C	200	220	240
B	Time	Min	5	17.5	30
C	Ligand Composition[a] (OAm/Oa)	Coded	1	2	3

[a] To maintain a constant total ligand volume of 4 mL in Flask A, the composition was varied using discrete volumetric ratios of Oleylamine (OAm) and Oleic Acid (OA). These were assigned coded values for the RSA model as follows: **1** = 1 mL OAm / 3 mL OA; **2** = 2 mL OAm/2 mL OA; **3** = 3 mL OAm/1 mL OA.

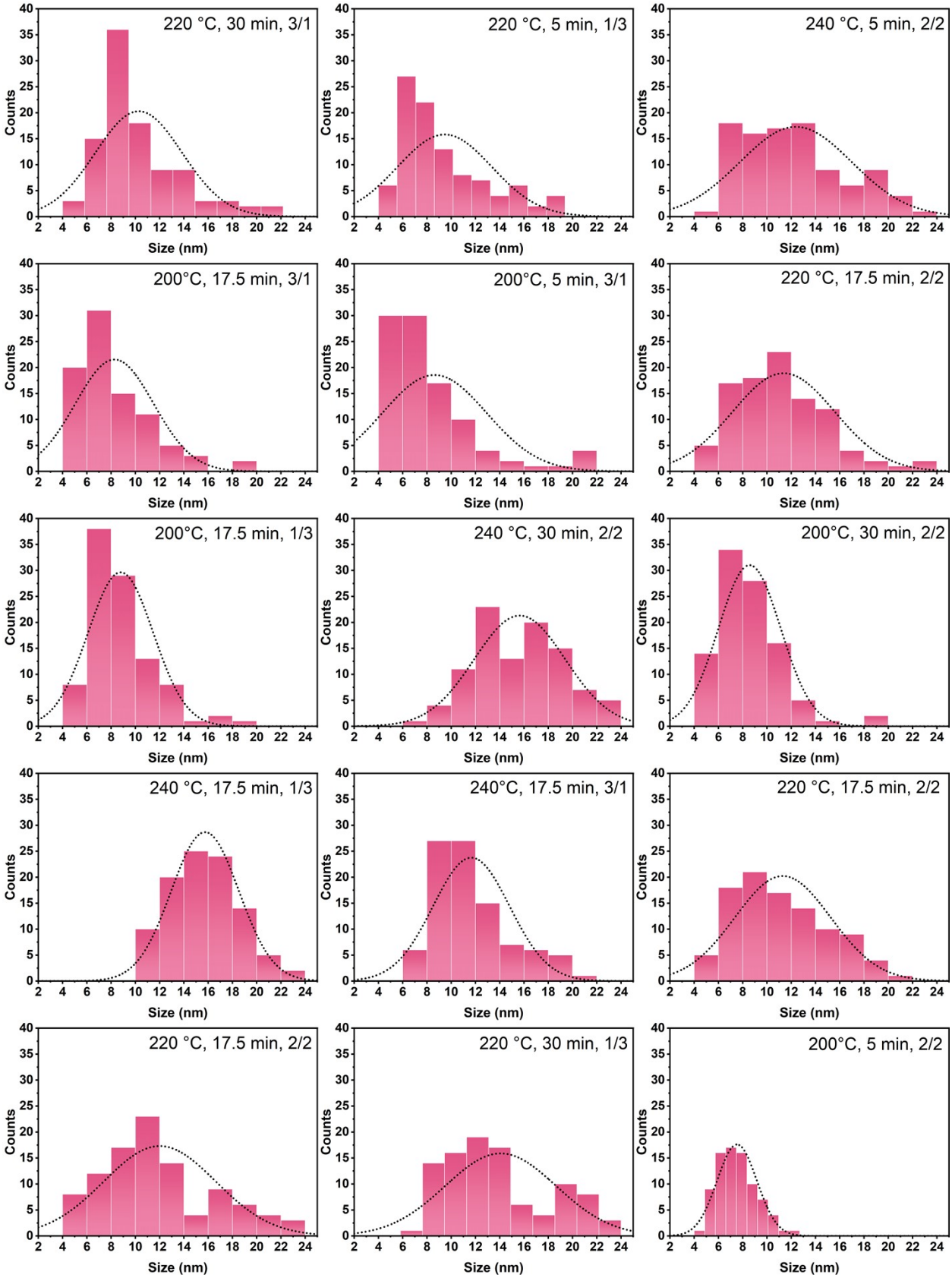


Figure S1. Particle size distribution histograms of $CuInSe_2$ nanoparticles synthesized under different conditions (temperature, time, and oleylamine/oleic acid volumes (mL)), fitted with normal distribution curves.

Table S2. Particle size statistics under different synthesis conditions (temperature, reaction time, and oleylamine/oleic acid volumes) for the $CuInSe_2$ nanoparticle samples.

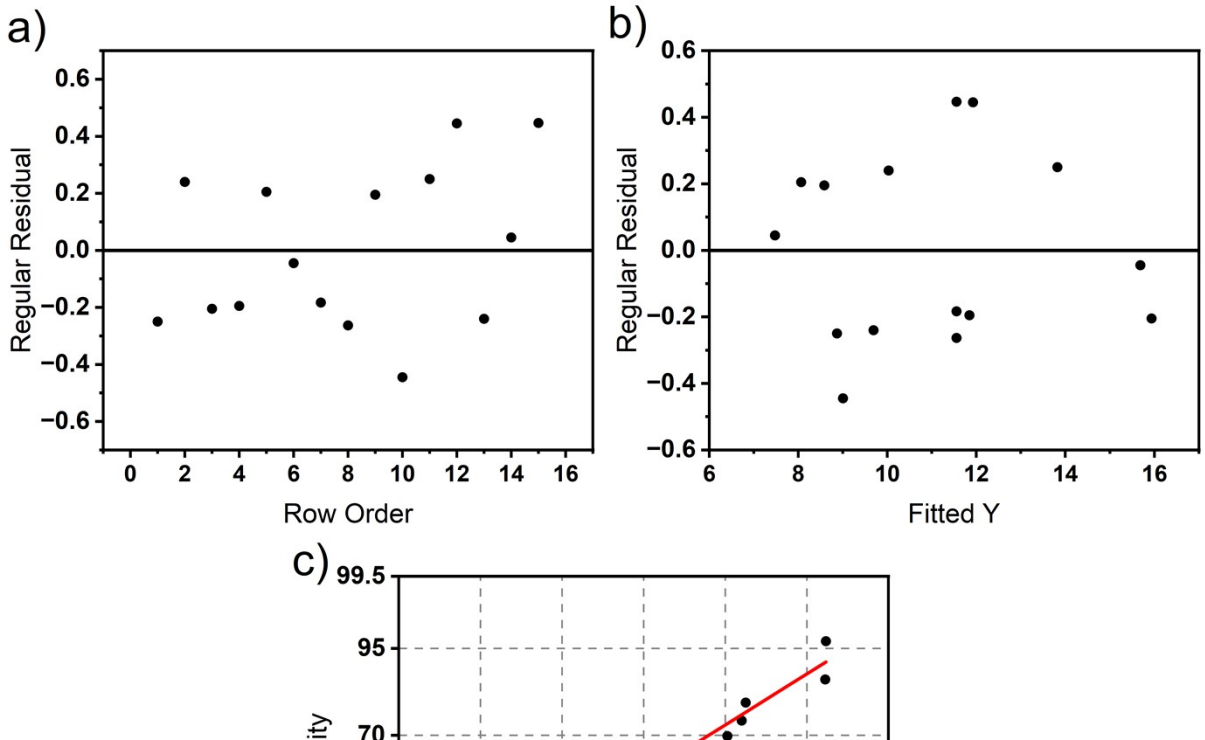
	N total	Mean	Standard Deviation	Minimum	Median	Maximum
220°C 30 min 3/1	100	10.27	3.58	5.27	9.23	21.81
220°C 5 min 1/3	100	9.46	3.89	4.41	8.23	24.12
240°C 5 min 2/2	100	12.37	4.62	5.80	11.65	28.64
200°C 5 min 2/2	83	7.52	1.64	3.81	7.30	12.06
220°C 30 min 1/3	100	14.08	4.57	6.85	13.00	27.05
200°C 17.5 min 3/1	87	8.27	3.22	4.47	6.91	19.92
220°C 5 min 3/1	100	8.62	4.30	4.45	7.23	29.10
220°C 17.5 min 2/2	100	11.37	4.22	4.80	10.68	25.92
200°C 17.5 min 1/3	100	8.78	2.69	4.64	8.25	18.05
240°C 30 min 2/2	100	15.65	3.75	6.49	15.48	24.10
200°C 30 min 2/2	100	8.56	2.57	4.74	8.09	19.04
220°C 17.5 min 2/2	100	12.00	4.61	4.80	11.51	23.26
240°C 17.5 min 1/3	100	15.74	2.78	10.67	15.20	22.39
240°C 17.5 min 3/1	94	11.66	3.16	7.19	10.96	21.10
220°C 17.5 min 2/2	100	11.30	3.95	5.03	10.55	24.93

Table S3. Percentage Contribution of Experimental Factors based on ANOVA Results

Variable	Sum of squares	Percentage of contribution*
Temperature	61.938	65.29%
Time	13.992	14.75%
Ligand Composition	10.626	11.20%
Temperature*Temperature	6.16E-04	0%
Time*Time	0.990	1.04%
Ligand Composition*Ligand Composition	0.692	0.73%
Temperature*Time	1.243	1.31%
Temperature*Ligand Composition	3.186	3.36%
Time*Ligand Composition	2.205	2.32%
Total	95.869	100%

*The percentage of contribution was calculated based on the sum of squares of the individual factors and their interactions only, excluding the residual error, to highlight the relative influence of the controlled parameters within the model.

Figure S2. Residual analysis of the response surface model for particle size: a) residuals versus row order, b) residuals versus fitted values, and c) normal probability plot of the residuals.



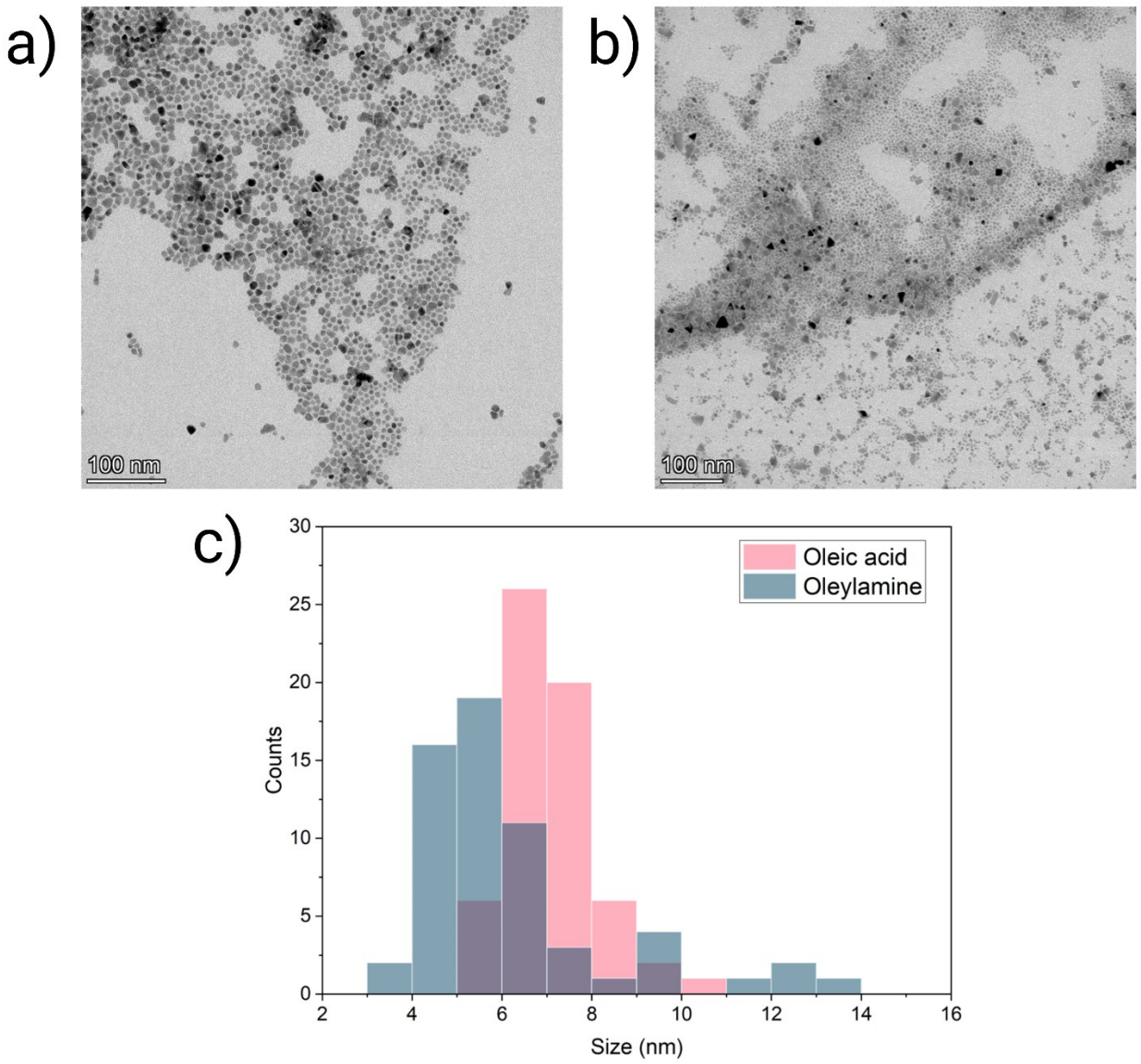


Figure S3. a) $CuInSe_2$ synthesized using only oleic acid at 220 °C for 17.5 min; b) $CuInSe_2$ synthesized using only oleylamine; and c) size histograms comparing the sizes of nanoparticles obtained from oleic-acid-only and oleylamine-only syntheses.

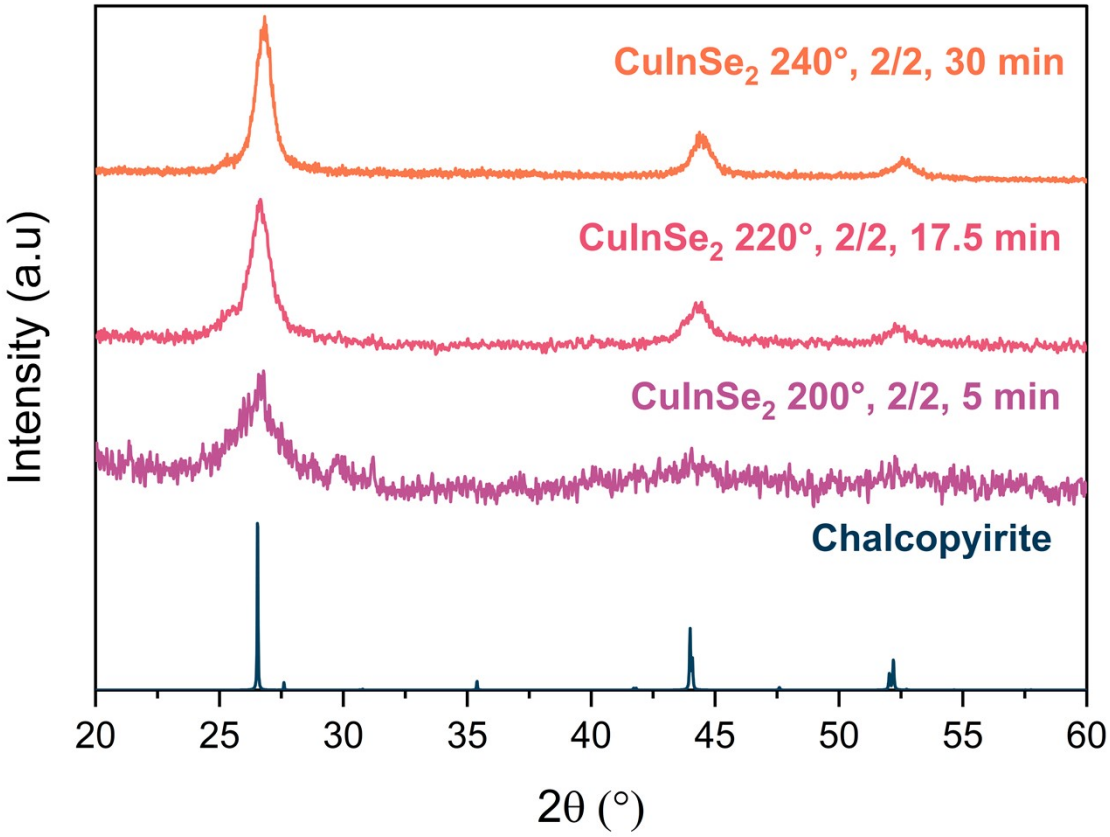


Figure S4. Powder X-ray diffractograms of $CuInSe_2$ nanoparticle samples synthesized at low, medium, and high temperature–time conditions and prepared using the same oleylamine/oleic acid ligand composition, showing the characteristic chalcopyrite phase of $CuInSe_2$.

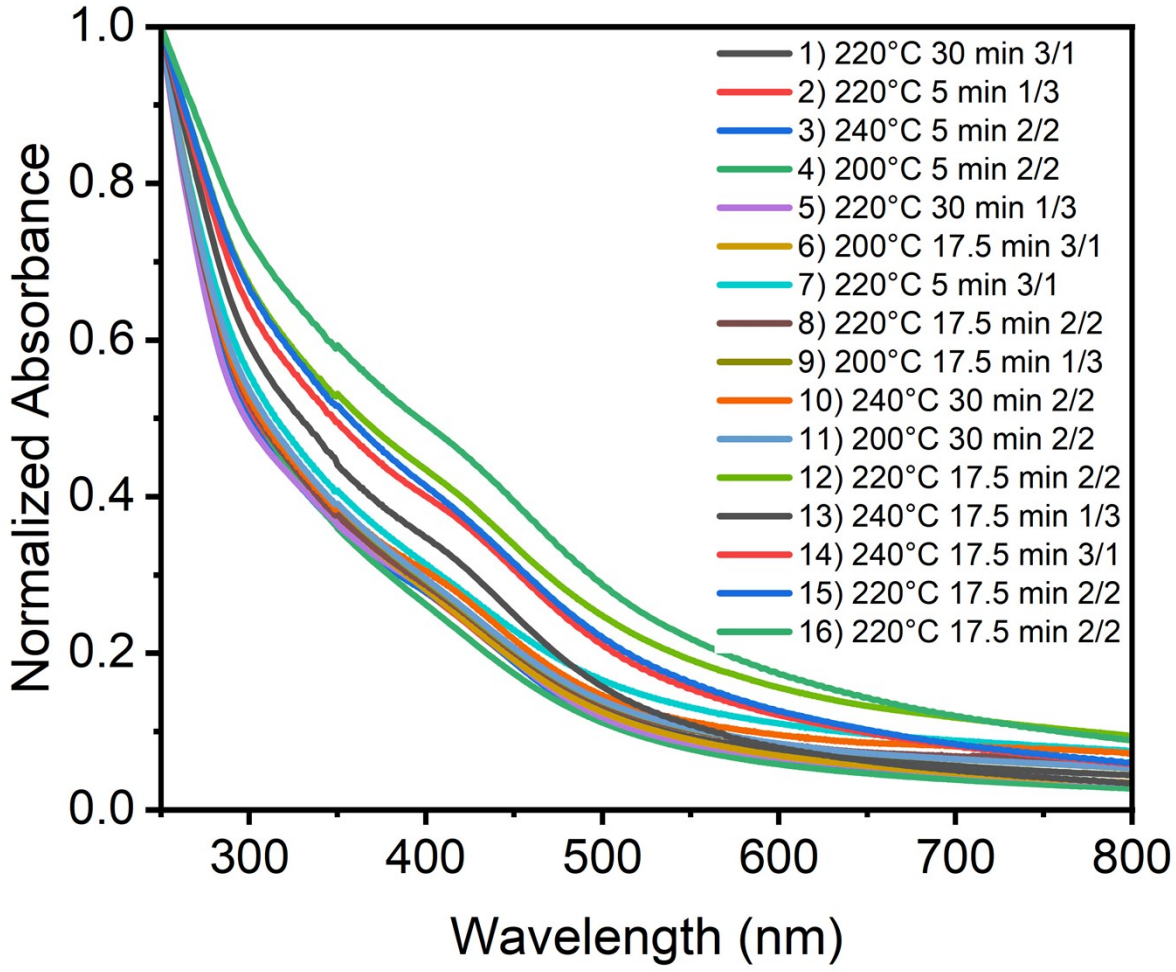


Figure S5. UV-Vis spectrogram of the synthesized $CuInSe_2$ NPs according to the generated condition in the surface response analysis, all the samples possess a featureless spectrum commonly reported for CuInSe NPs.

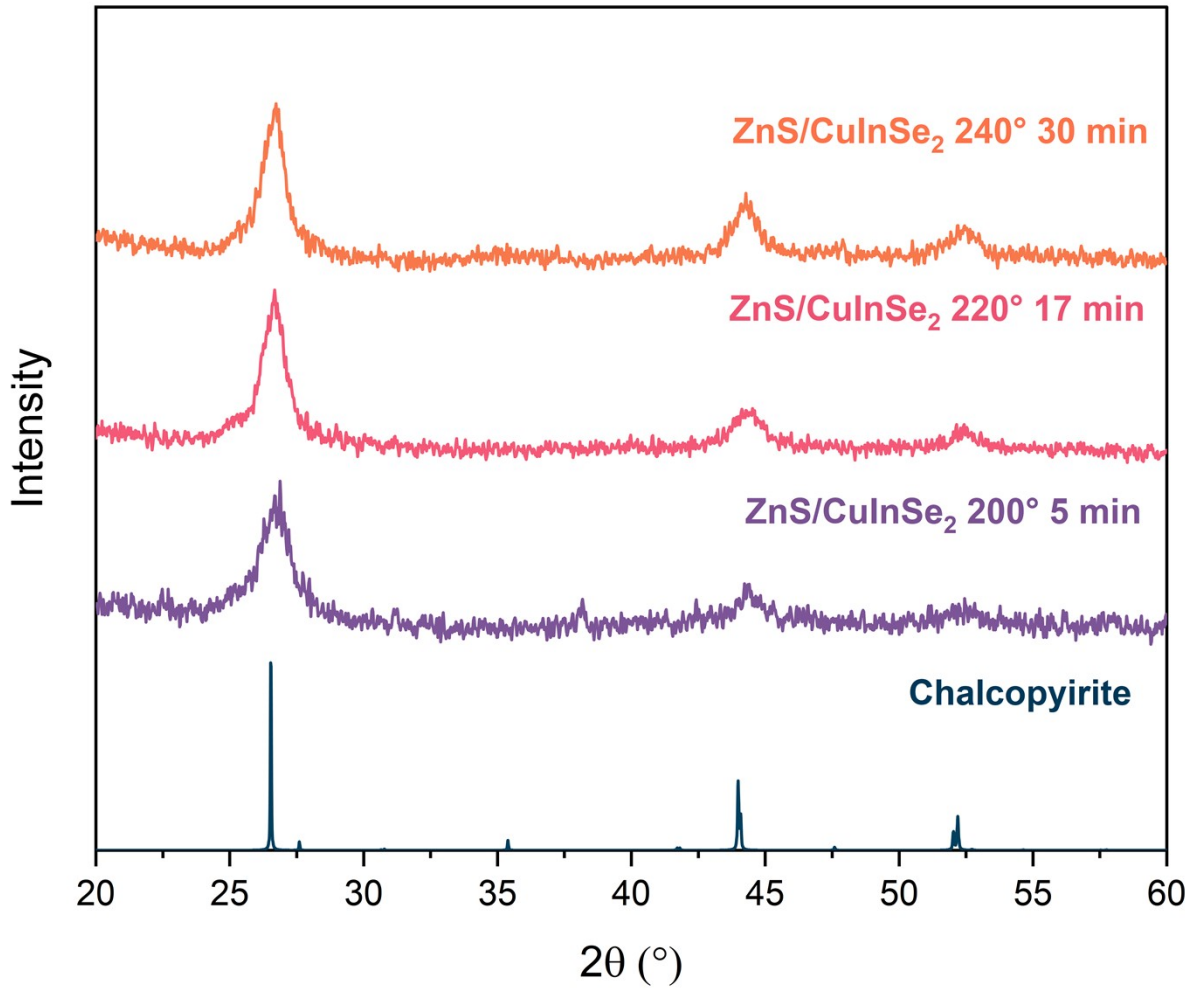


Figure S6. Powder X-ray diffractograms of ZnS-passivated $CuInSe_2$ nanoparticle samples synthesized at low, medium, and high temperature–time conditions and prepared using the same oleylamine/oleic acid ligand composition, showing the characteristic chalcopyrite phase of $CuInSe_2$ and the absence of other crystalline phases.

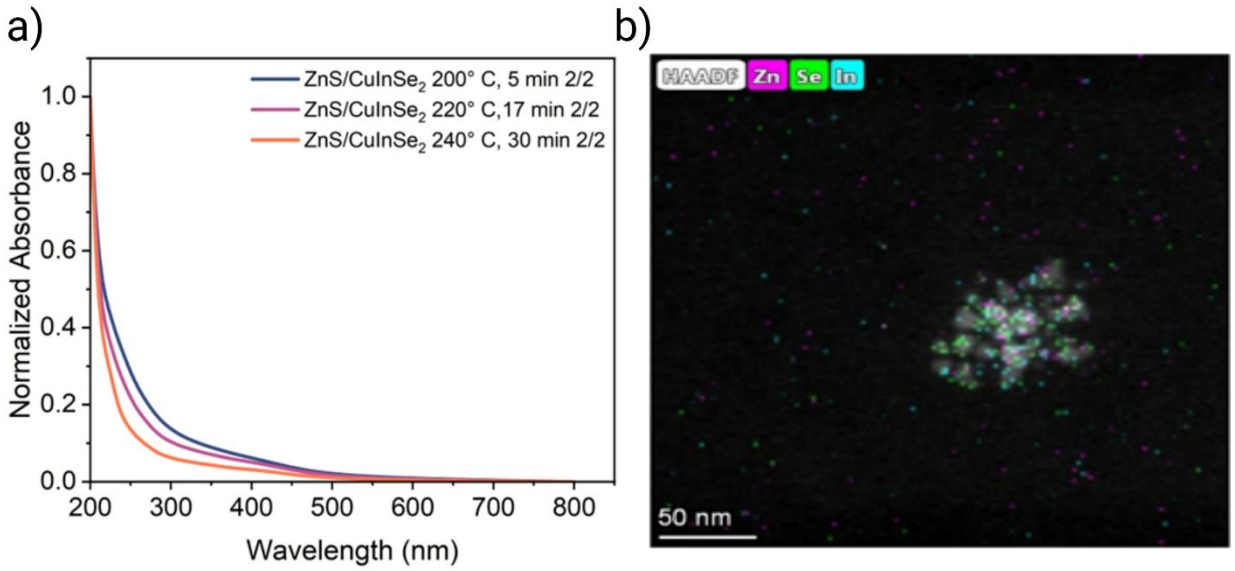


Figure S7. a) UV-Vis spectra of ZnS-passivated $CuInSe_2$ nanoparticles synthesized samples synthesized at low, medium, and high temperature–time conditions and prepared using the same oleylamine/oleic acid ligand composition. All samples show a featureless absorption profile typically reported for $CuInSe_2$ nanoparticles. b) HAADF-STEM image and corresponding EDX elemental mapping of a representative ZnS/$CuInSe_2$ nanoparticle. The distribution of indium, selenium, and zinc confirms the presence of the constituent elements.