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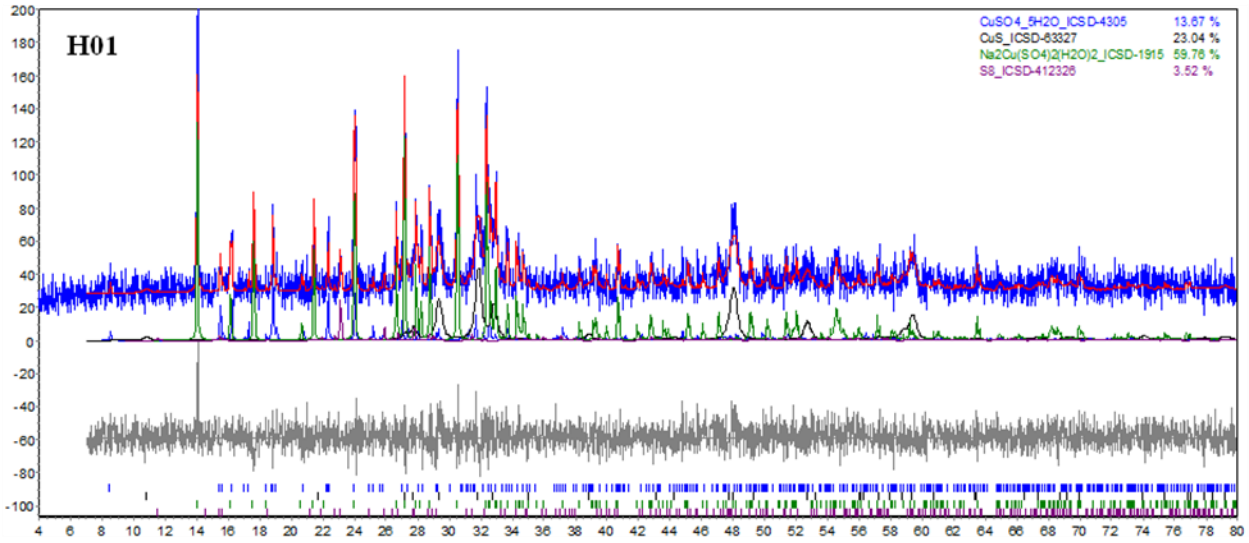
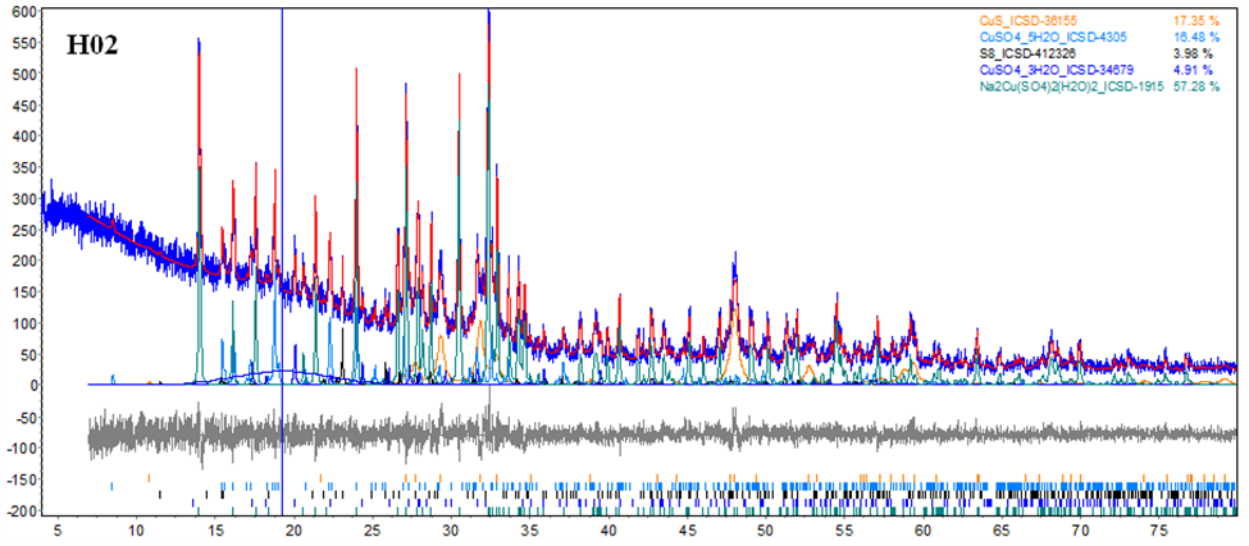
APPENDICES

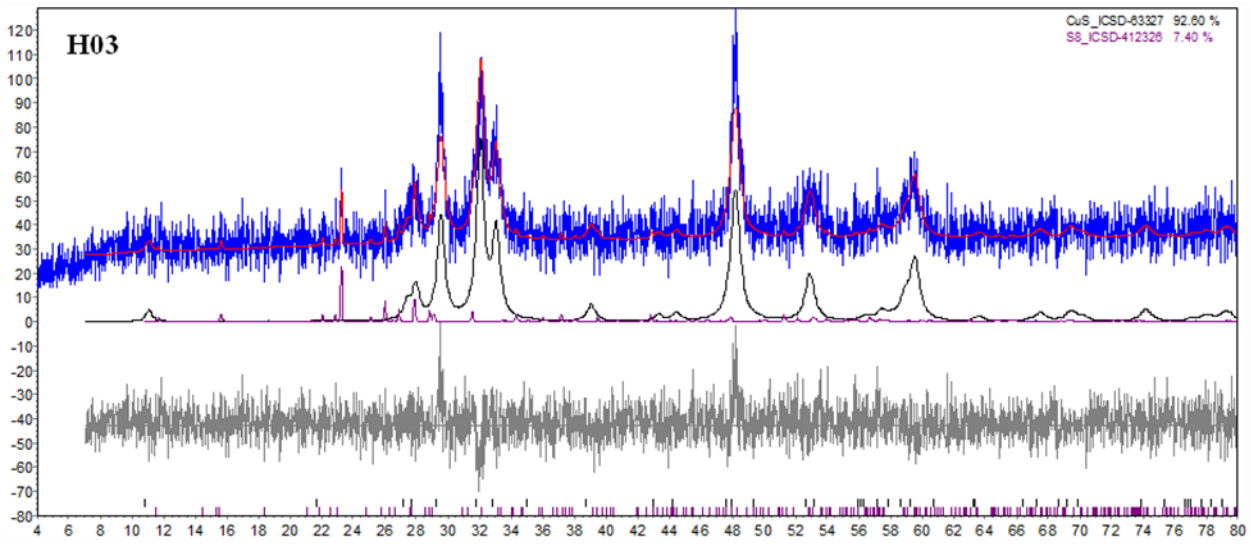
Appendix A



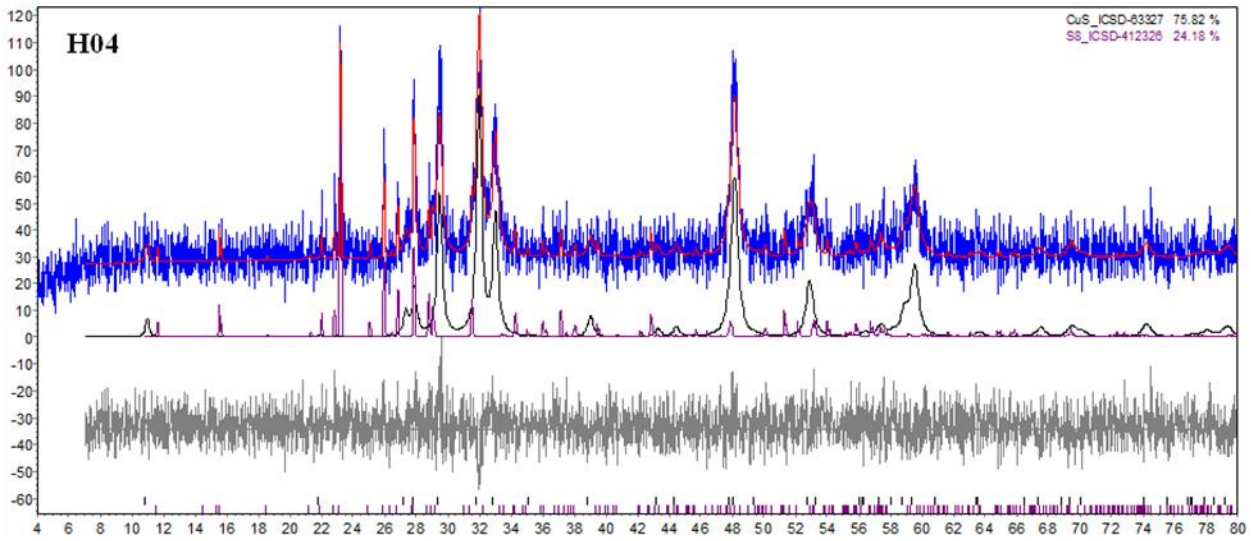
Rietveld refinement analysis for product formed at 25 °C (H01).



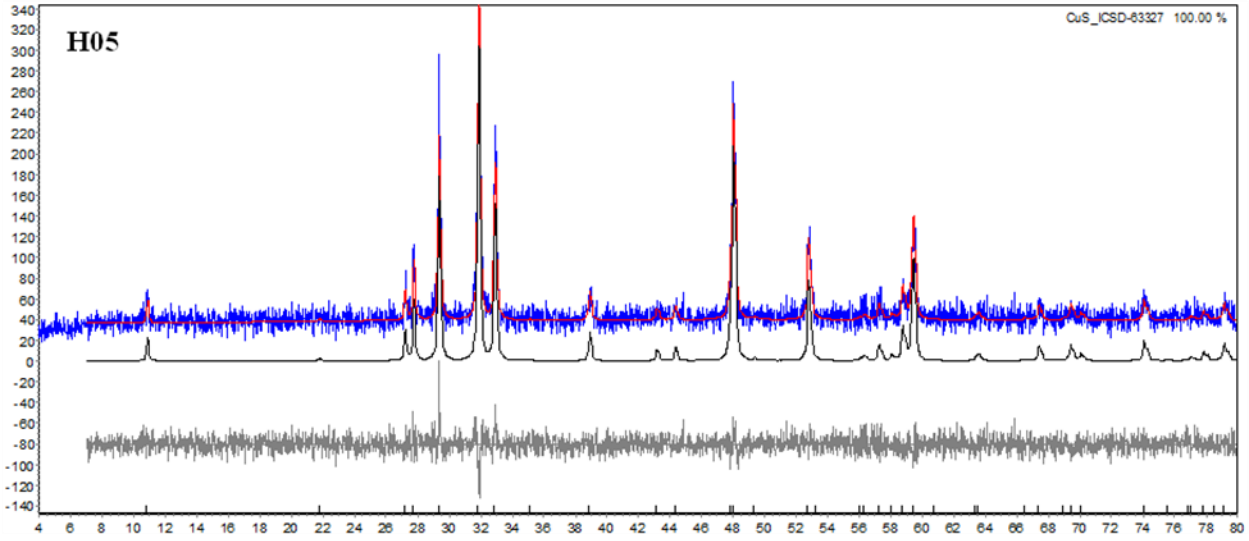
Rietveld refinement analysis for product formed at 35 °C (H02).



Rietveld refinement analysis for product formed at 65 °C (H03).

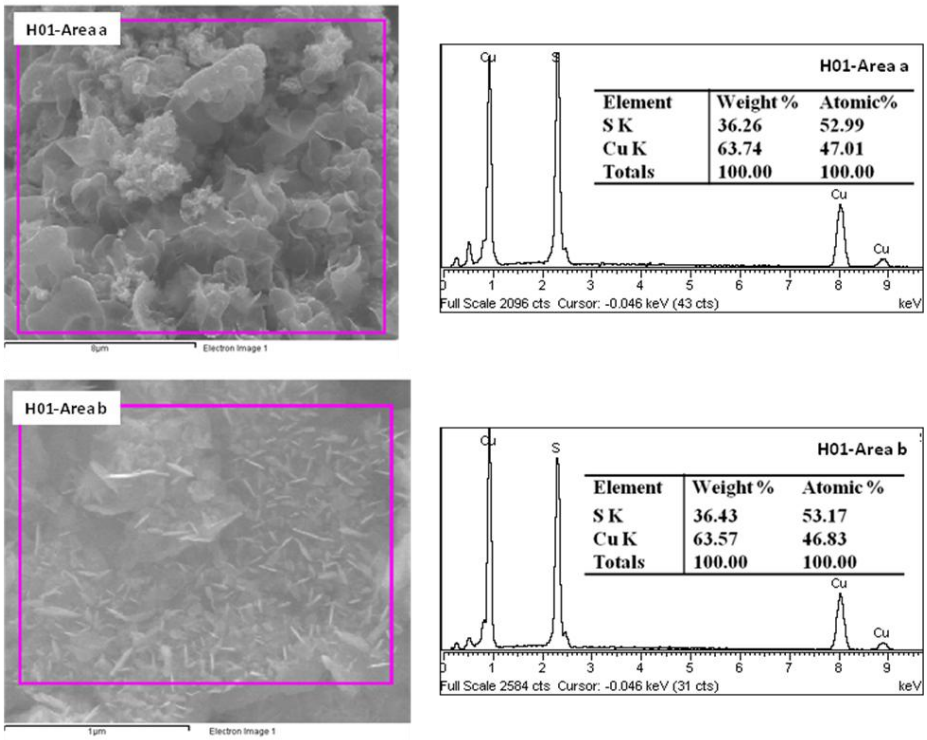


Rietveld refinement analysis for product formed at 95 °C (H04).

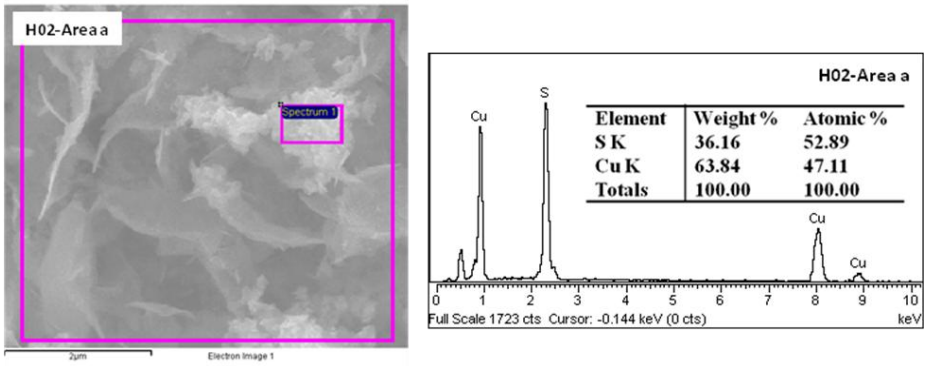


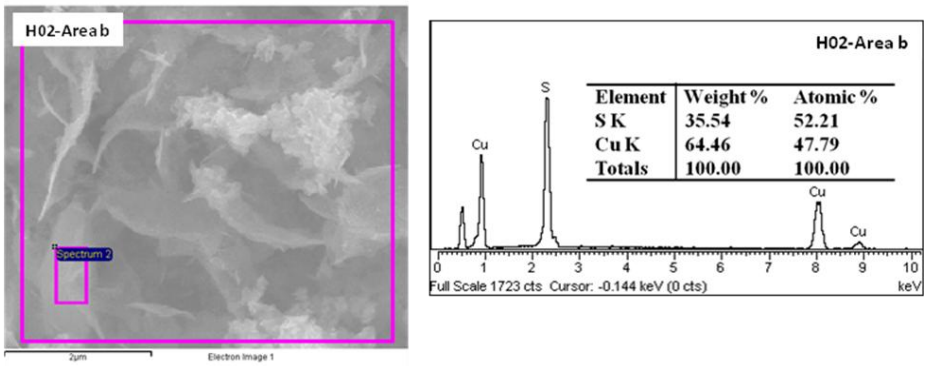
Rietveld refinement analysis for product formed at 155 ℃ (H05).

Appendix B

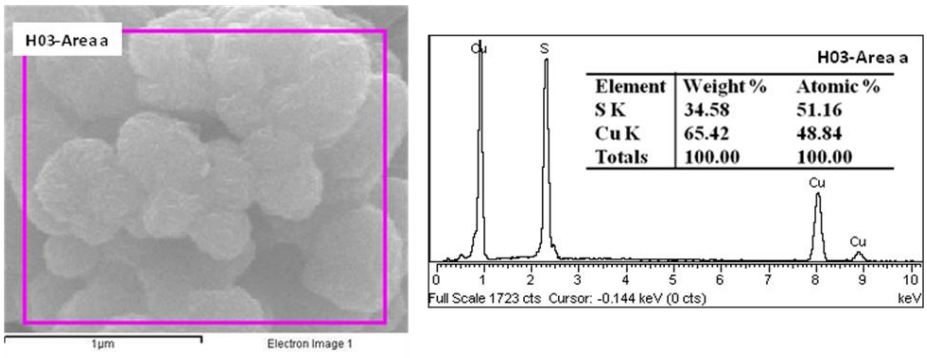


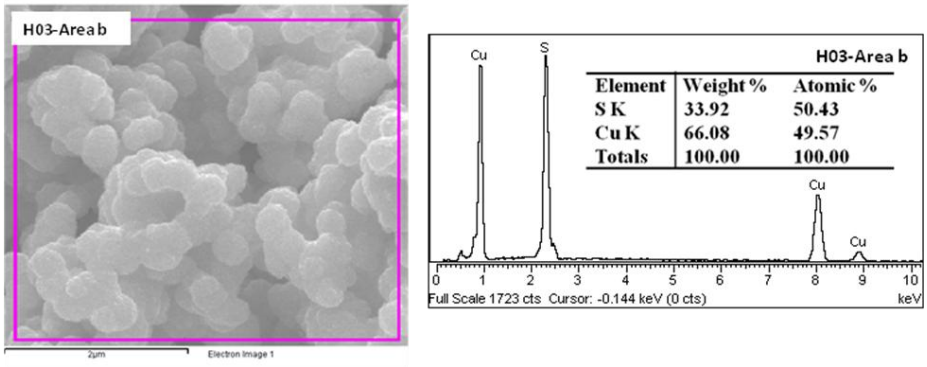
EDX analysis of H01 sample prepared at 25 ℃, Cu: S mole ratio = 1: 2 for 12 hours.



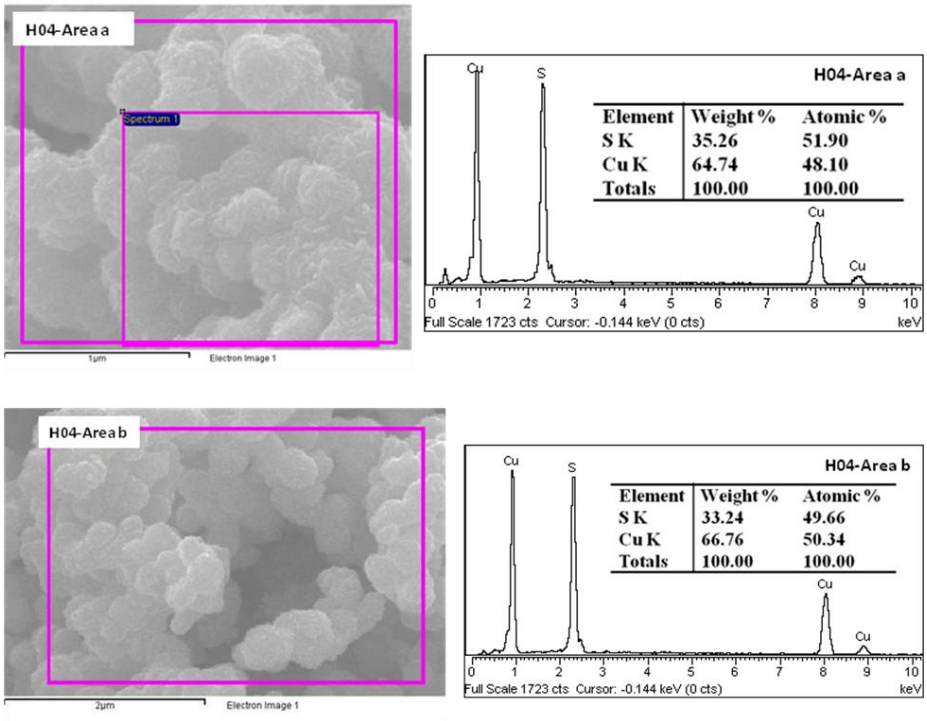


EDX analysis of H02 sample prepared at 35 °C, Cu: S mole ratio = 1: 2 for 12 hours.

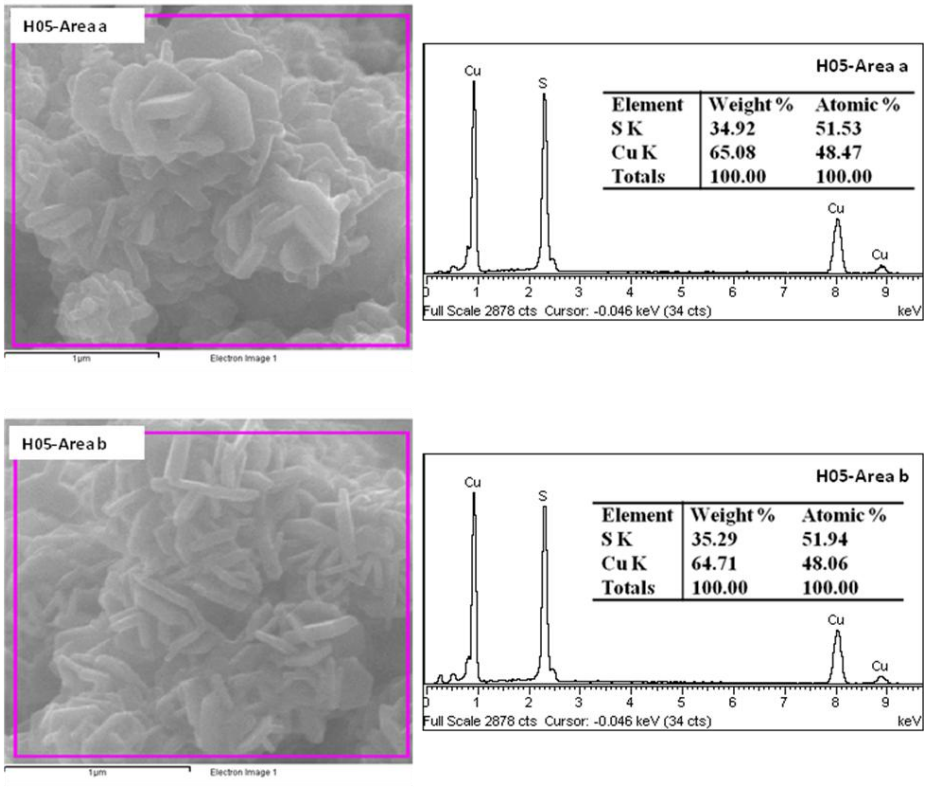




EDX analysis of H03 sample prepared at 65 °C, Cu: S mole ratio = 1: 2 for 12 hours.



EDX analysis of H04 sample prepared at 95 ℃, Cu: S mole ratio = 1: 2 for 12 hours.



EDX analysis of H04 sample prepared at 155 ℃, Cu: S mole ratio = 1: 2 for 12 hours.

Example of calculation for XRF analysis (Sample H01):

Wt % of Cu element = 52.8/100 × 63.546
= **8.309×10^{-3} moles**

Wt % of S element = 47.2/100 × 32.064
= **1.472×10^{-2} moles**

Atomic ratio (Cu/S) = 8.309×10^{-3} moles/1.472×10^{-2} moles
= **0.56**

Cu: S = $8.309 \times 10^{-3}/8.309 \times 10^{-3}$: $1.472 \times 10^{-2}/8.309 \times 10^{-3}$
= **1: 1.77**

Example of calculation for EDX analysis (Sample H01):

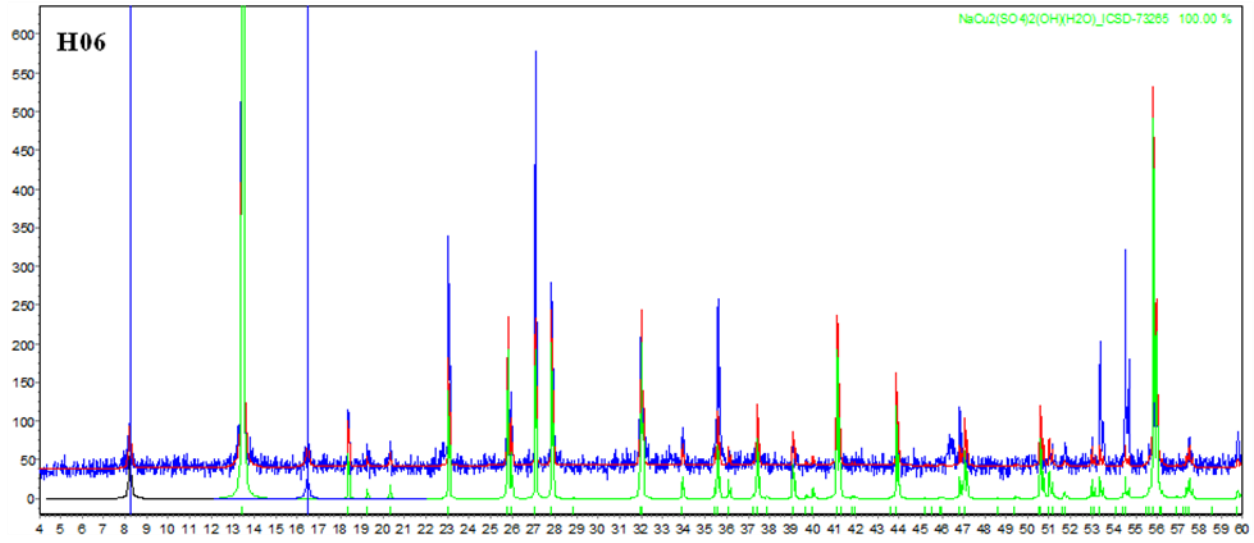
Conc. (at % average) of Cu element = (47.01 + 46.83)/ 2
= **46.92 %**

Conc. (at % average) of S element = (52.99 + 53.17)/ 2
= **53.08 %**

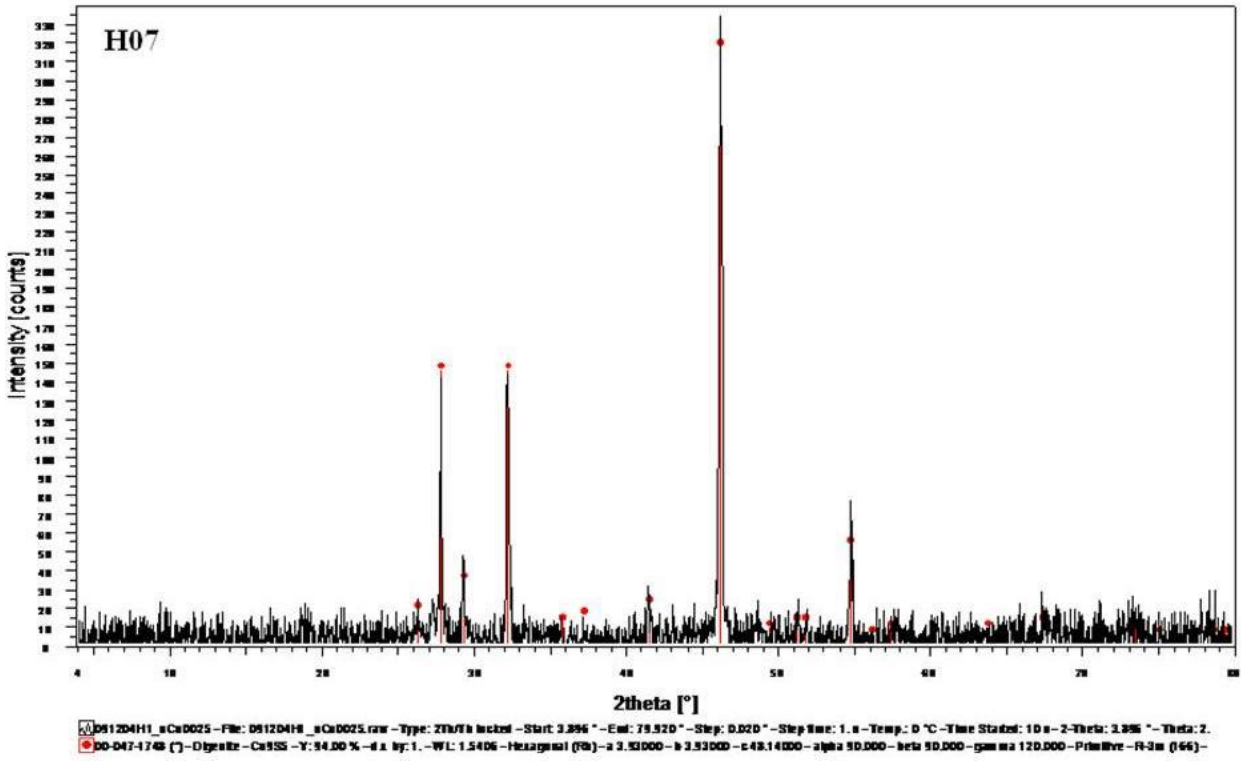
Calculation Atomic ratio (Cu/S) = 46.92/ 53.08
= **0.88**

Cu: S = 46.92/46.92: 53.08/53.08
= **1: 1.13**

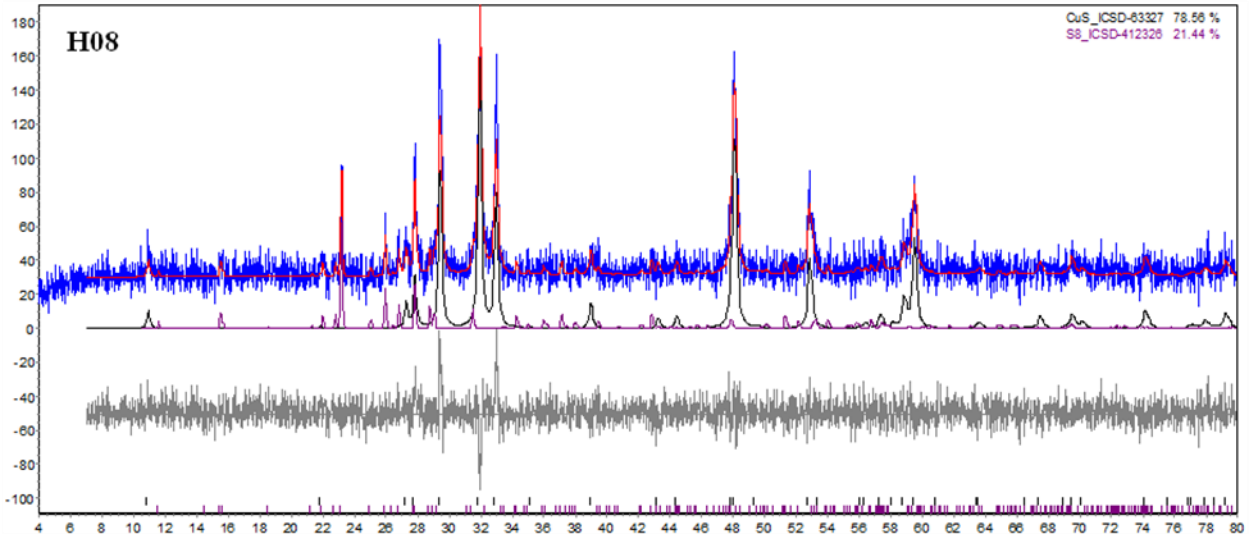
Appendix C



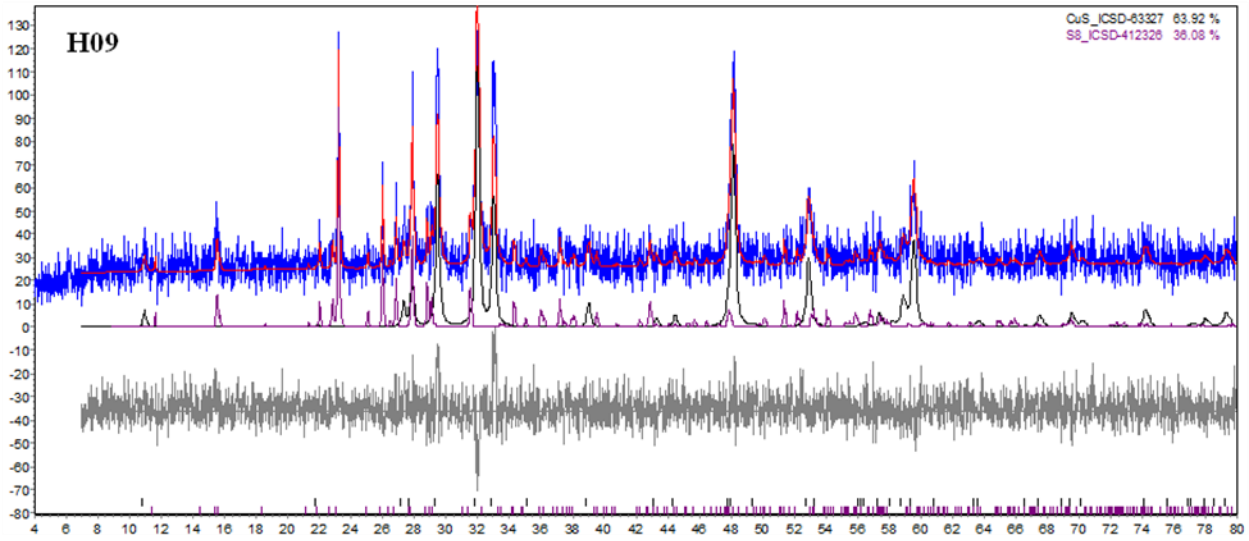
Rietveld refinement analysis for product formed at Cu: S mole ratio of 1: 1 (H06).



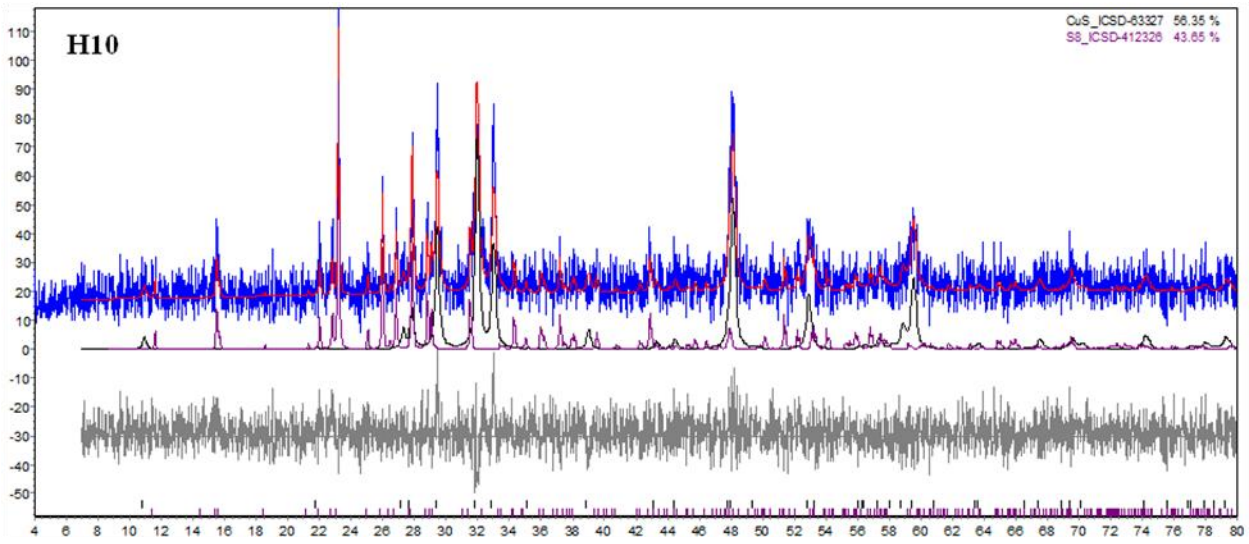
Peak matching for product formed at Cu: S mole ratio of 1: 1.5 (H07).



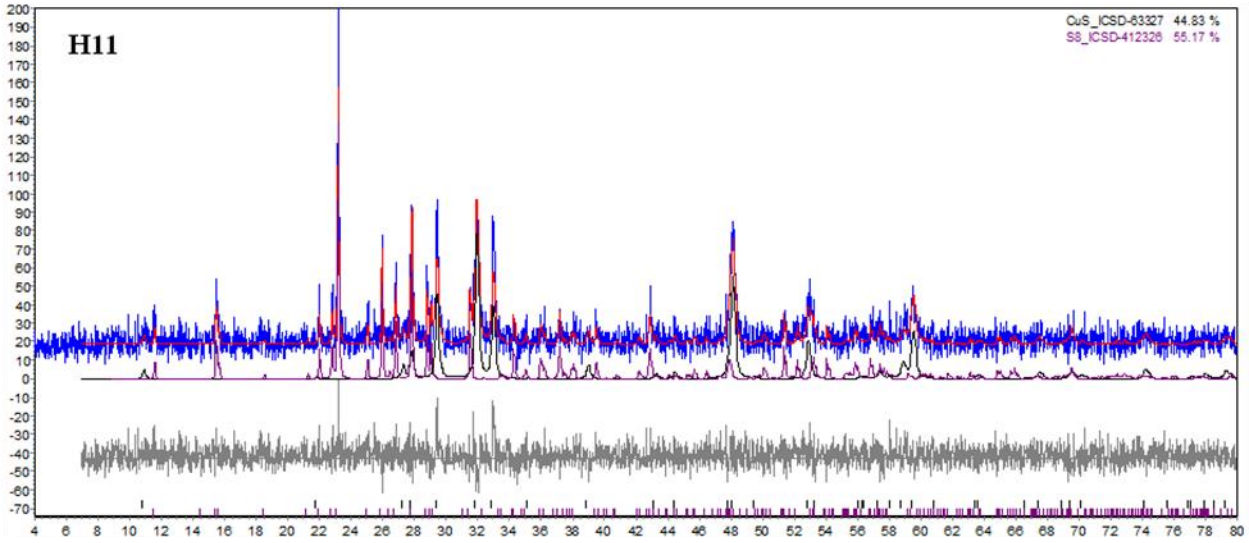
Rietveld refinement analysis for product formed at Cu: S mole ratio of 1: 2.5 (H08).



Rietveld refinement analysis for product formed at Cu: S mole ratio of 1: 3 (H09).

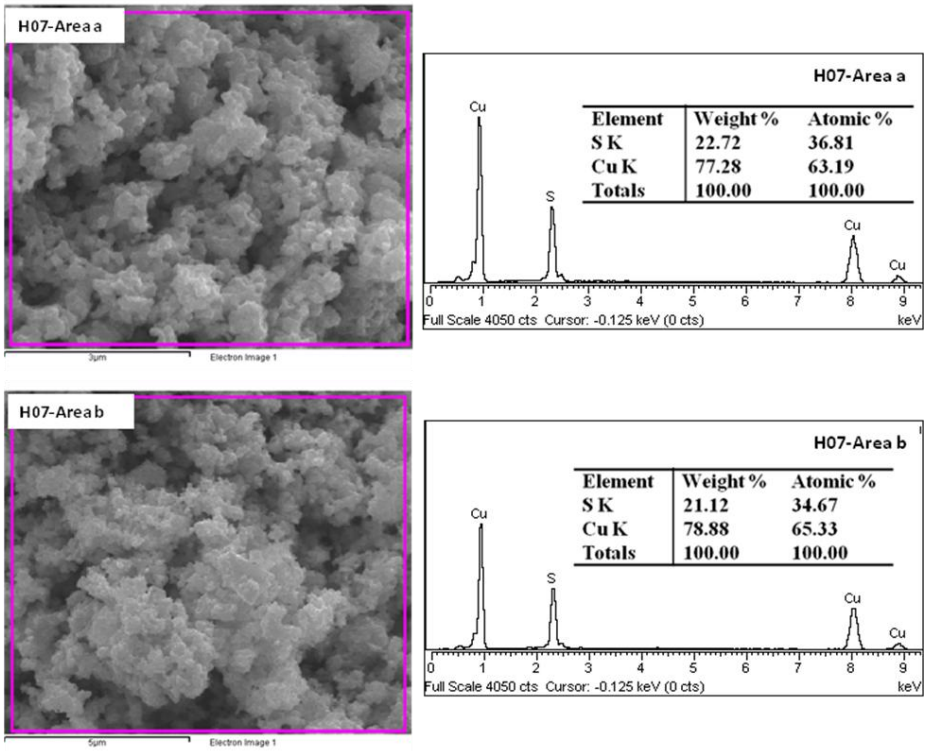


Rietveld refinement analysis for product formed at Cu: S mole ratio of 1: 3.5 (H10).

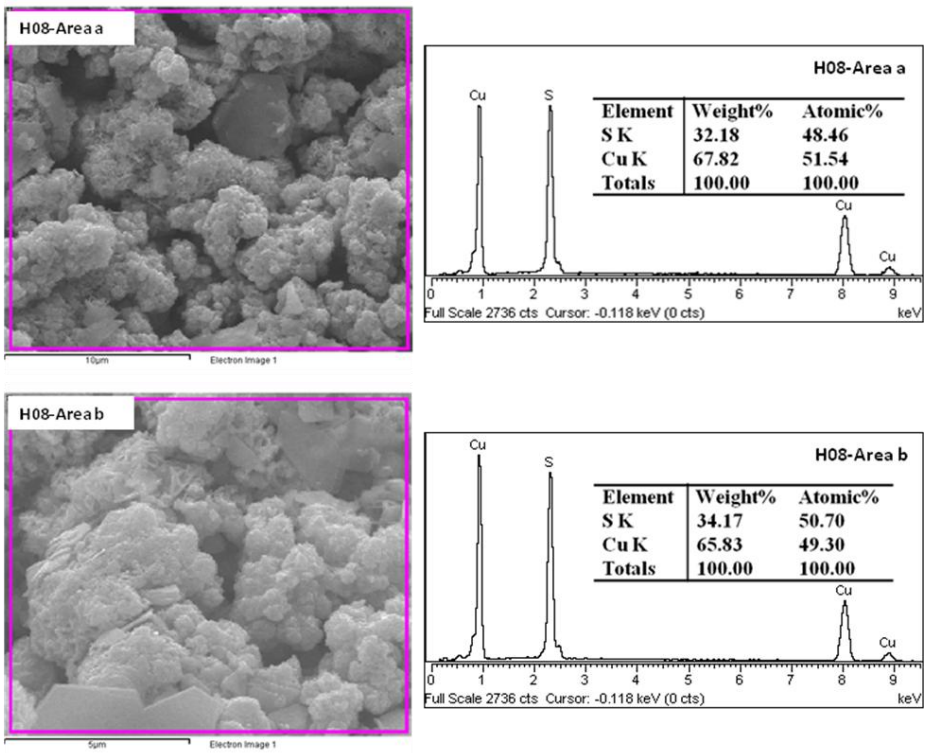


Rietveld refinement analysis for product formed at Cu: S mole ratio of 1: 5 (H11).

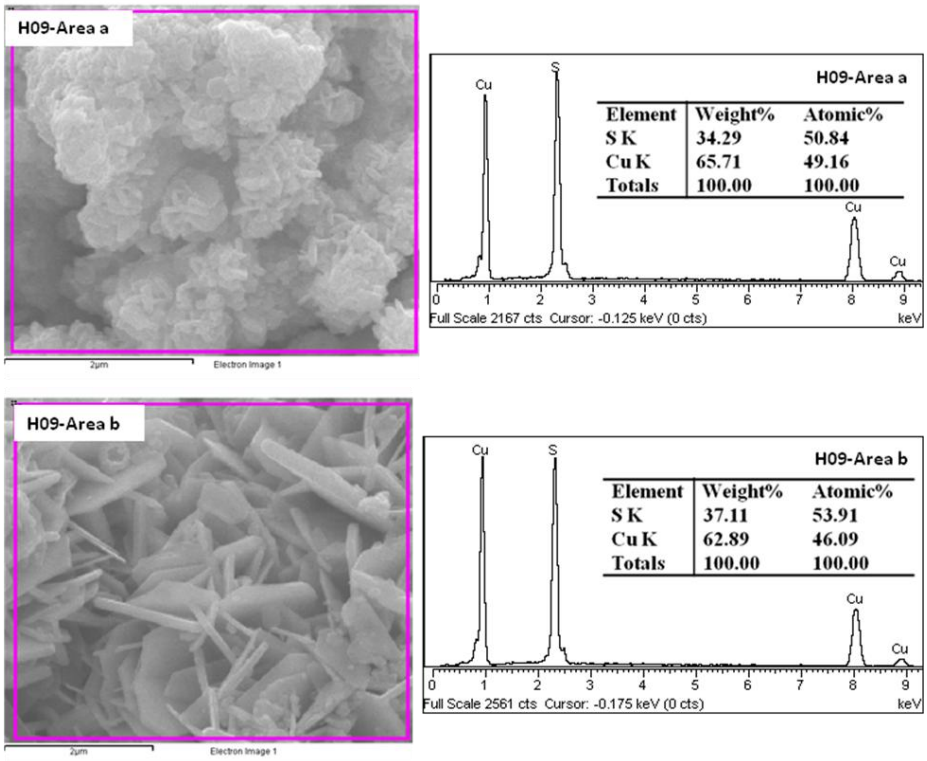
Appendix D



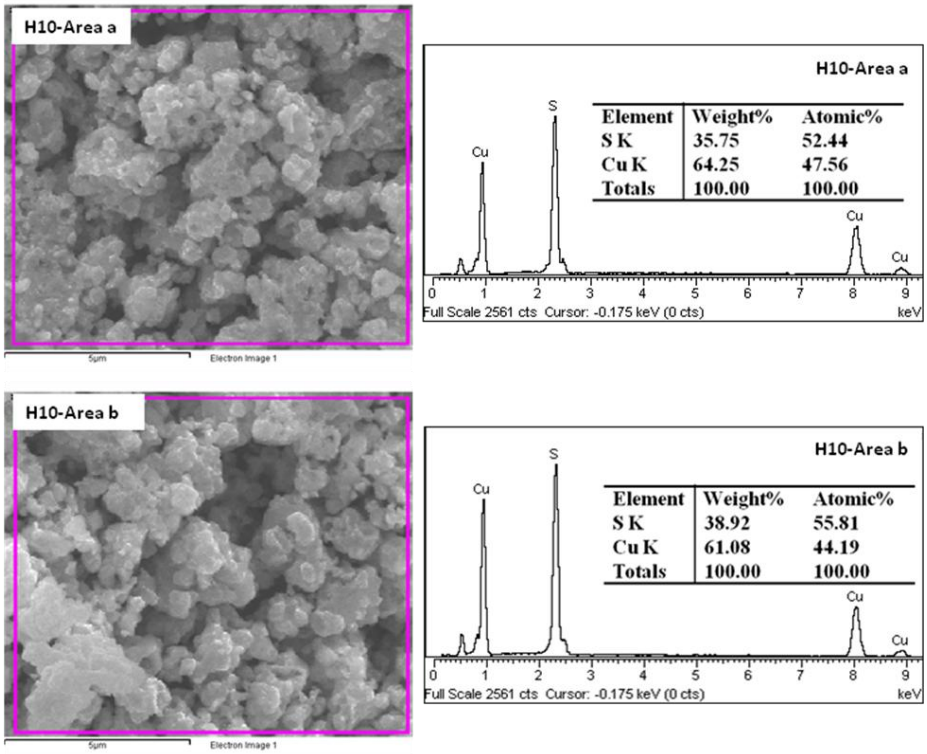
EDX analysis of H07 sample prepared at 155 °C, Cu: S mole ratio = 1: 1.5 for 12 hours.



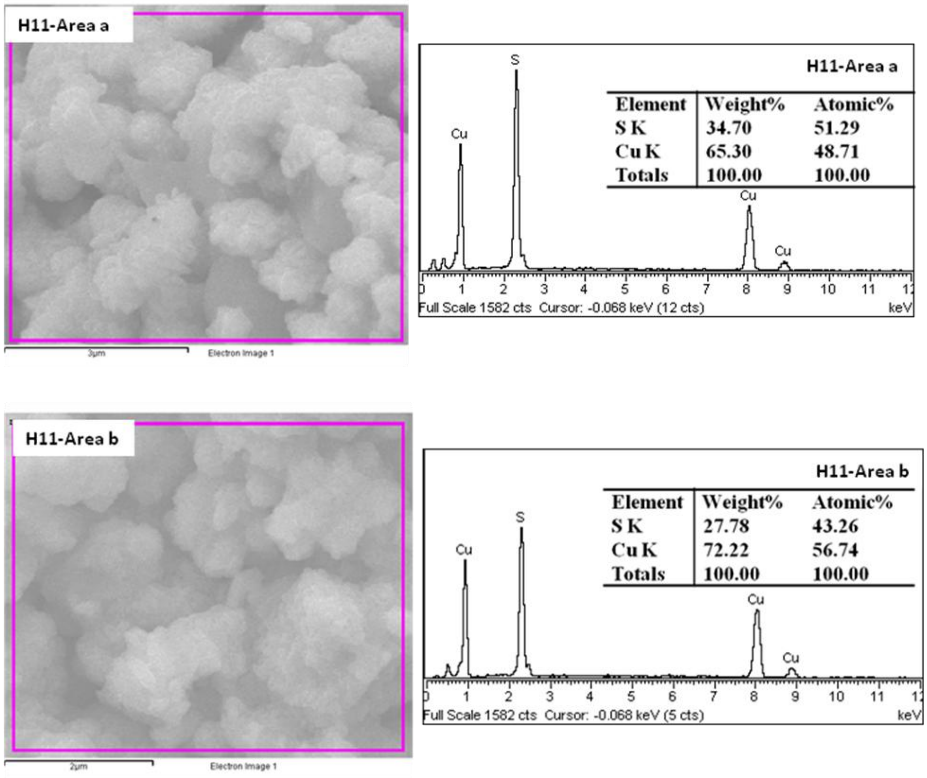
EDX analysis of H08 sample prepared at 155 °C, Cu: S mole ratio = 1: 2.5 for 12 hours.



EDX analysis of H09 sample prepared at 155 ℃, Cu: S mole ratio = 1: 3 for 12 hours.

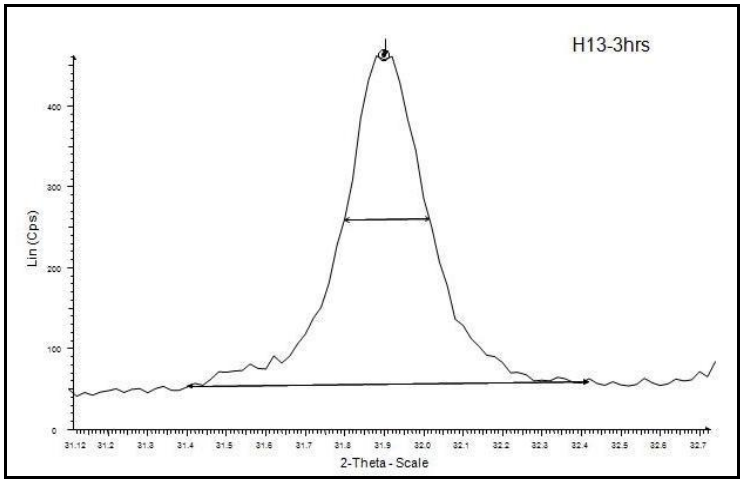


EDX analysis of H10 sample prepared at 155 °C, Cu: S mole ratio = 1: 3.5 for 12 hours.

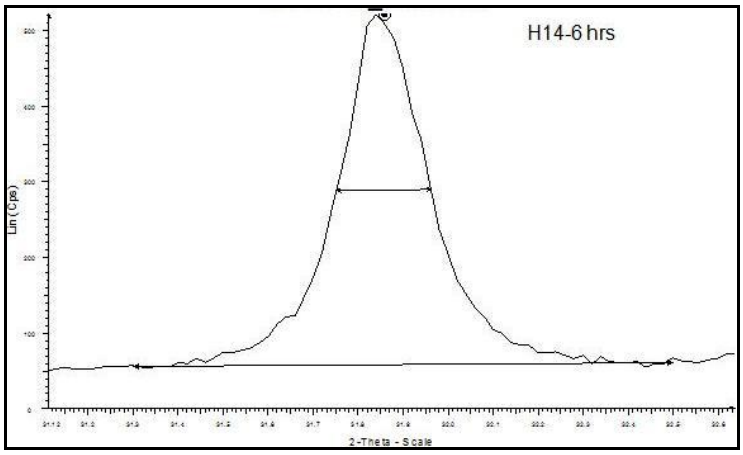


EDX analysis of H11 sample prepared at 155 ℃, Cu: S mole ratio = 1: 5 for 12 hours.

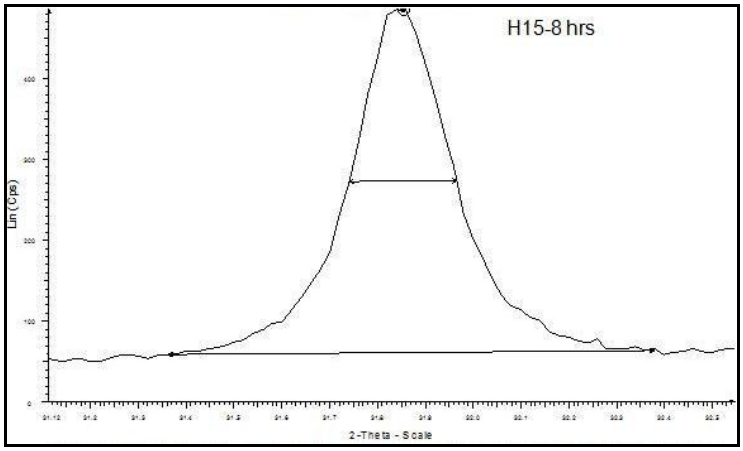
Appendix E



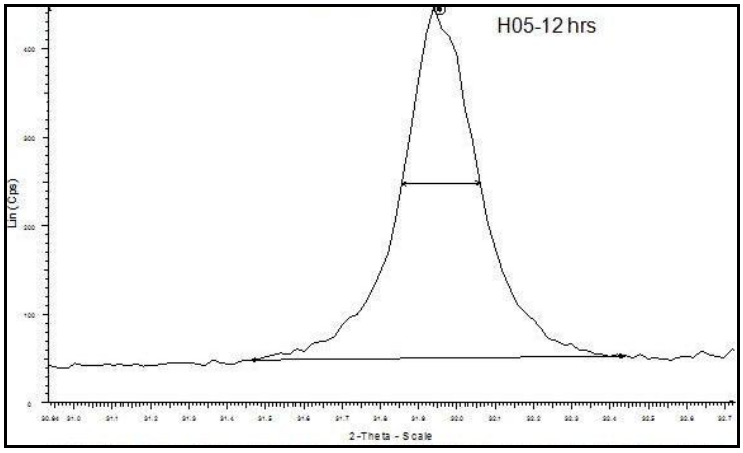
Peak fitting of characteristic peak of product synthesized at 3 hours (Sample H13).



Peak fitting of characteristic peak of product synthesized at 6 hours (Sample H14).



Peak fitting of characteristic peak of product synthesized at 8 hours (Sample H15).



Peak fitting of characteristic peak of product synthesized at 12 hours (Sample H05).

Example of calculation for peak profile analysis (Sample H13):

FWHM (°) = measured from each of the peak
FWHM (rad) = 0.274 × (3.142/180) rad
= **0.00478**
Angle (θ) = Angle (2θ) /2
= **15.9675**
Crystallite size = $(K\lambda)/(\beta \times \cos\theta)$
= (0.94 × 0.15406)/(0.00478 × *cos* 15.9675)
= **31.49 nm**

Error Analysis:

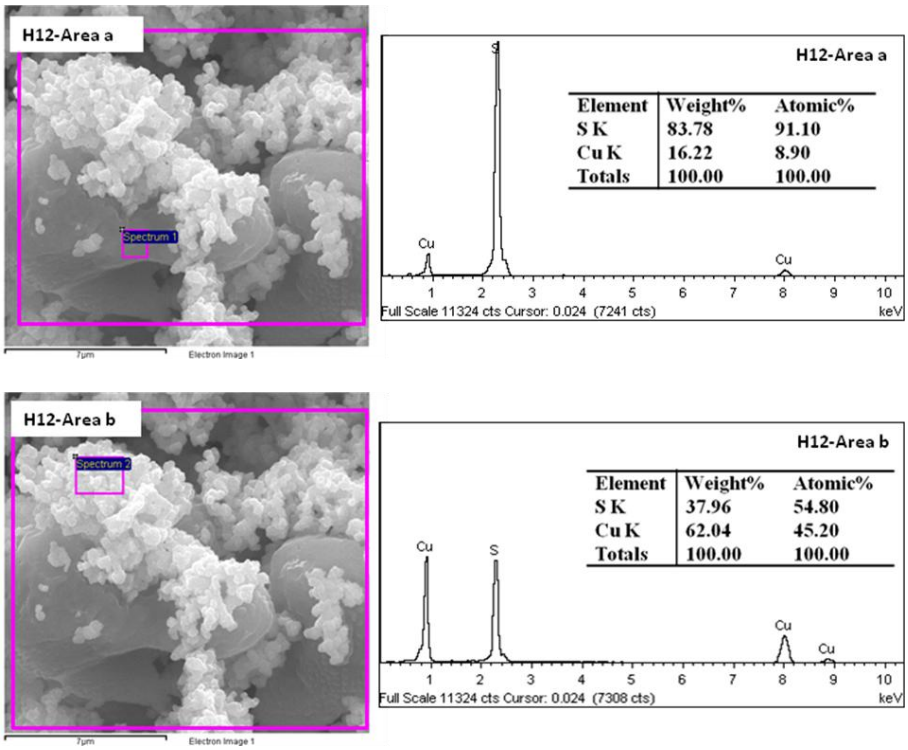
$$dL = \frac{K\lambda}{\beta} \times (\tan\vartheta/\cos\vartheta) \times d\vartheta$$

$$dL = \frac{(0.94 * 0.15406)}{0.00478} \times (\tan 15.9675/\cos 15.9675) \times (15.9675 \times 1\ \%)$$

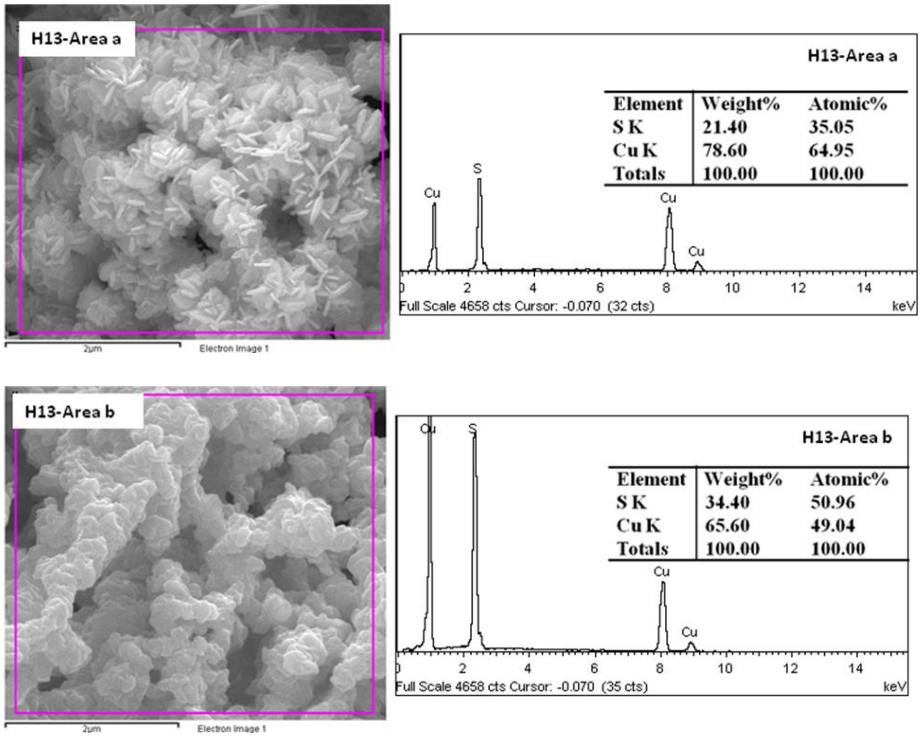
= **1.44 nm**

Therefore, crystallite size for sample **H13** is **(31.49 ± 1.44) nm**.

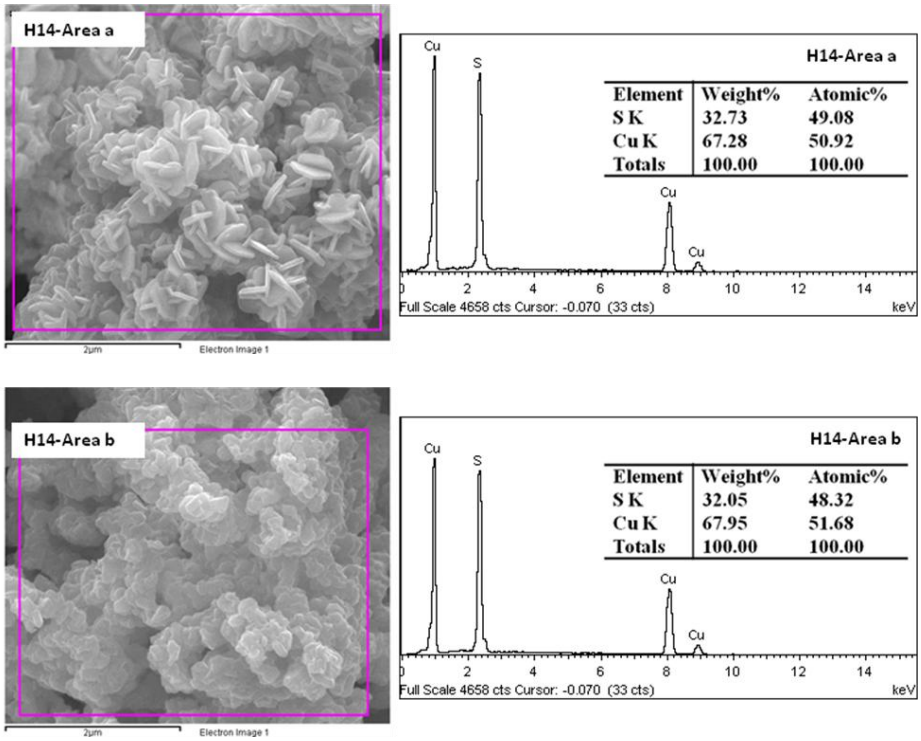
Appendix F



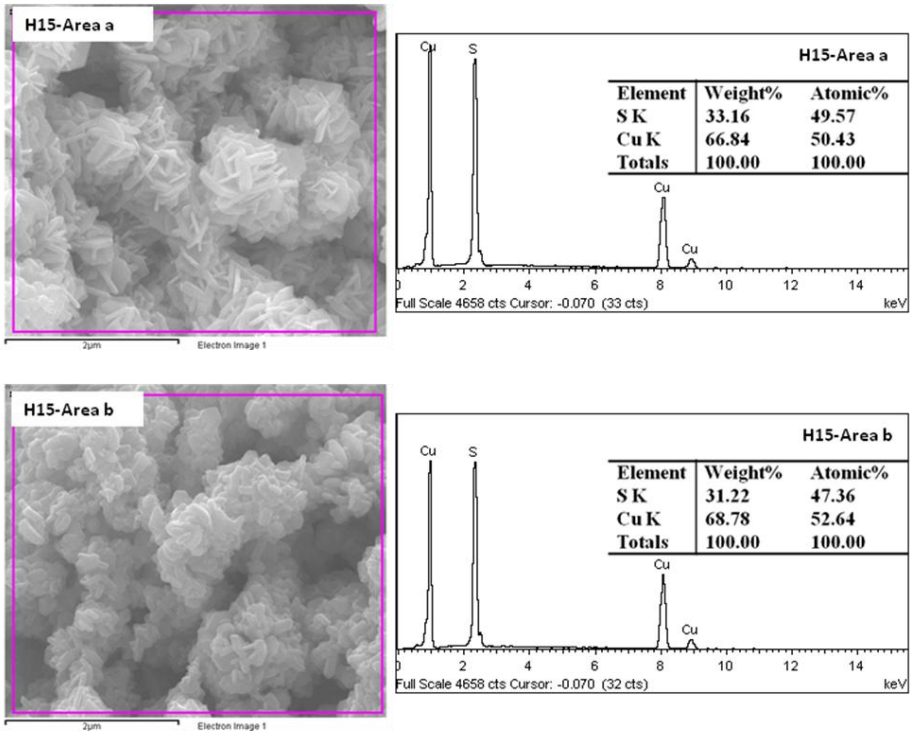
EDX analysis of H12 sample prepared at 155 ℃, Cu: S mole ratio = 1: 2 for 1 hour.



EDX analysis of H13 sample prepared at 155 ℃, Cu: S mole ratio = 1: 2 for 3 hours.

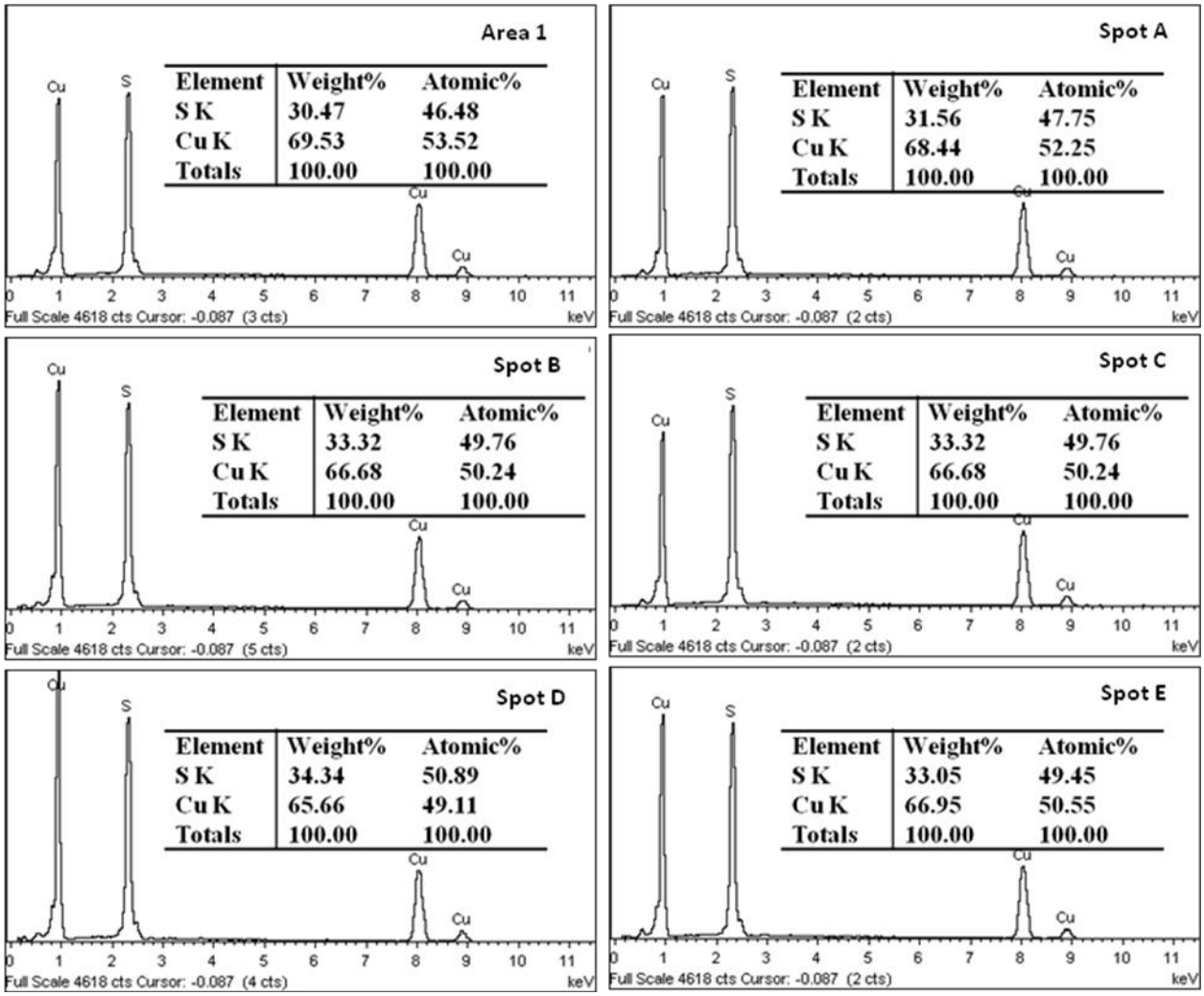


EDX analysis of H13 sample prepared at 155 °C, Cu: S mole ratio = 1: 2 for 6 hours.



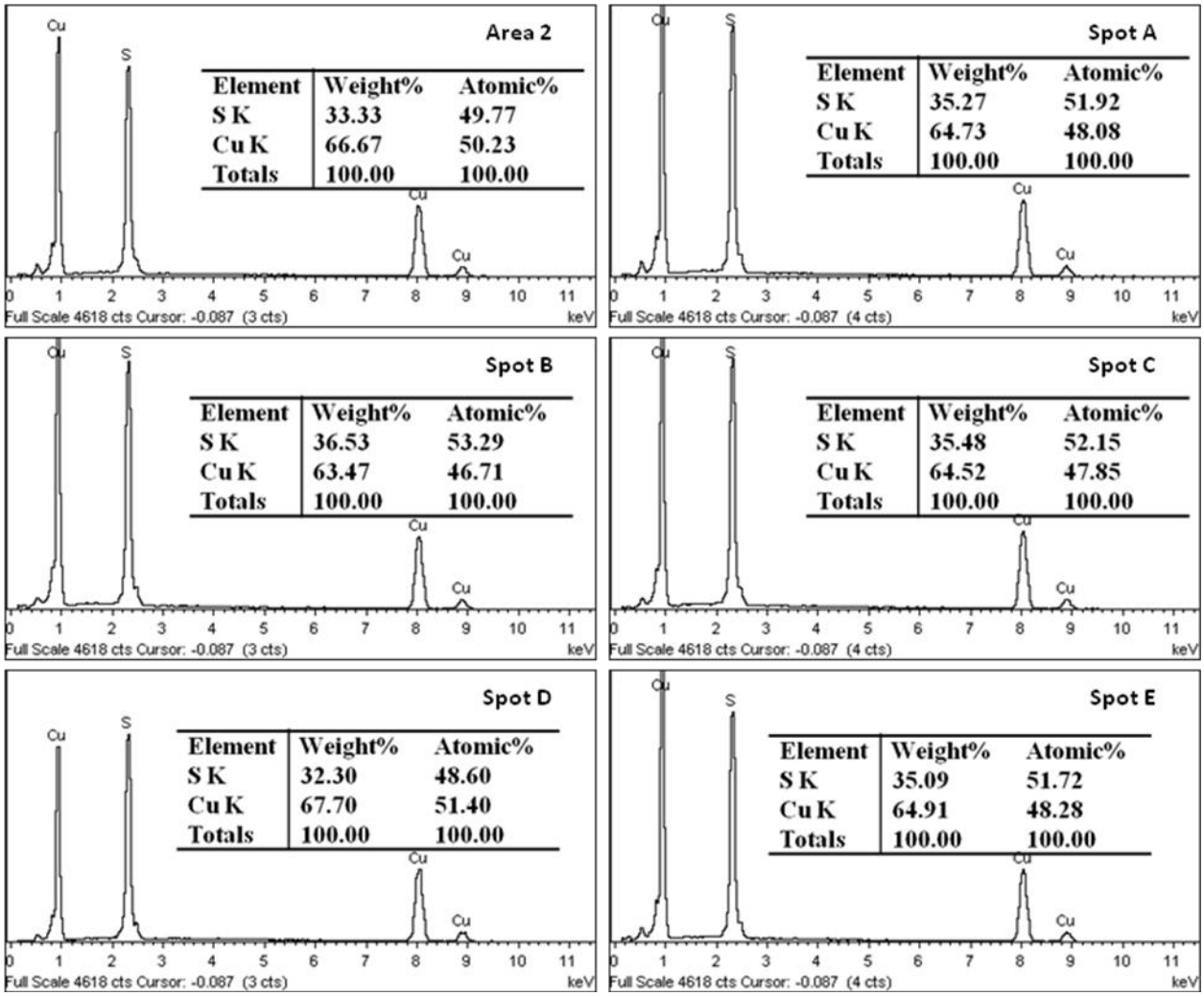
EDX analysis of H13 sample prepared at 155 °C, Cu: S mole ratio = 1: 2 for 8 hours.

Appendix G



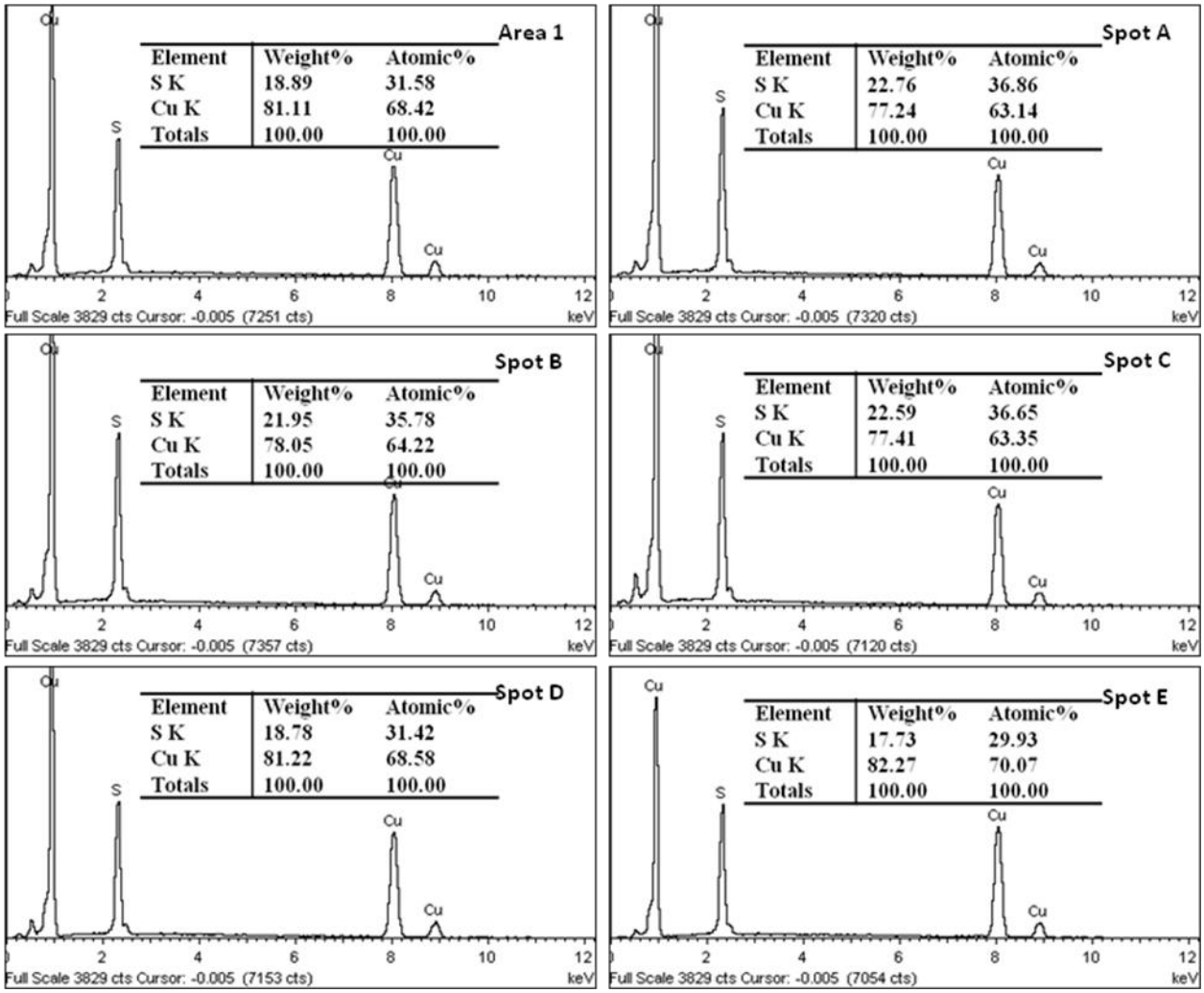
EDX spectra of phase pure covellite (area 1) prepared at 155 °C, Cu: S mole ratio = 1: 2 for 12 hours.

Appendix H



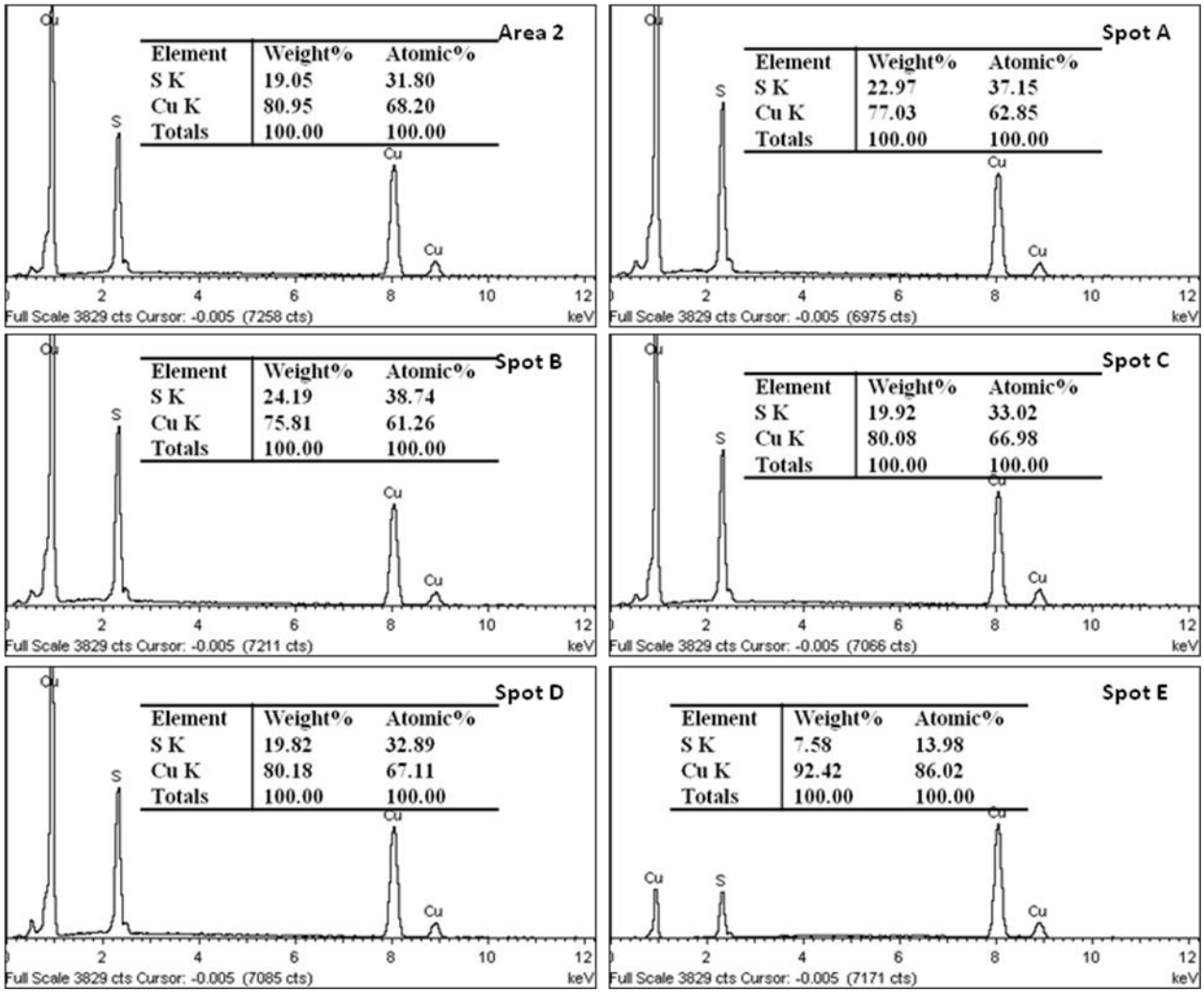
EDX spectra of phase pure covellite (area 2) prepared at 155 °C, Cu: S mole ratio = 1: 2 for 12 hours.

Appendix I



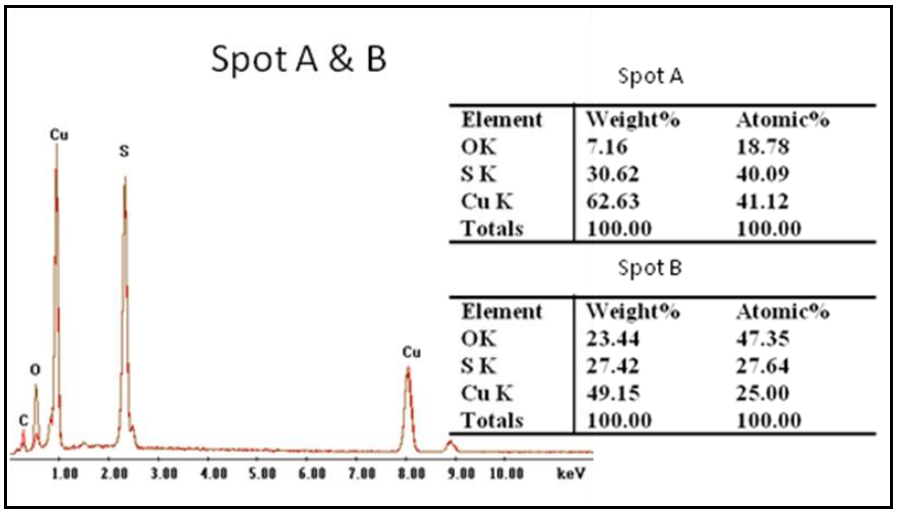
EDX spectra of phase pure digenite (area 1) prepared at 155 °C, Cu: S mole ratio = 1: 1.5 for 12 hours.

Appendix J



EDX spectra of phase pure digenite (area 2) prepared at 155 °C, Cu: S mole ratio = 1: 1.5 for 12 hours.

Appendix K

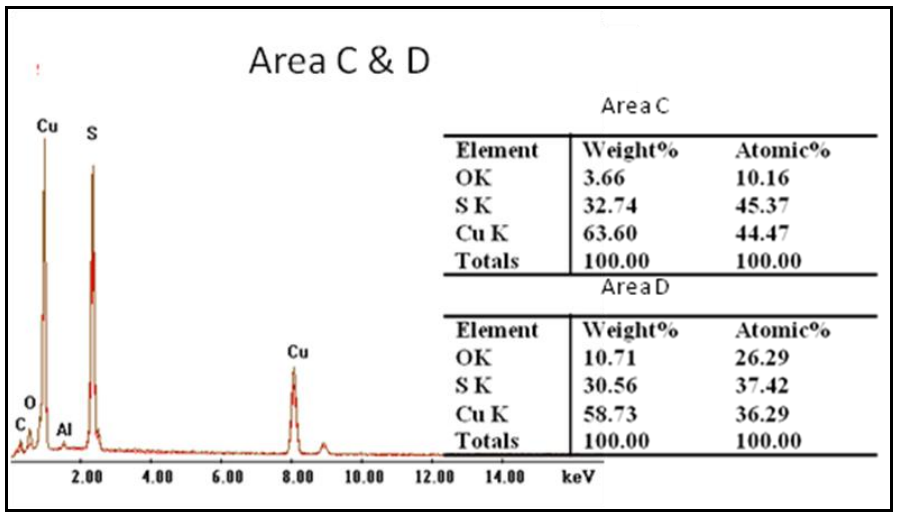


Spot A

Element	Weight%	Atomic%
O K	7.16	18.78
S K	30.62	40.09
Cu K	62.63	41.12
Totals	100.00	100.00

Spot B

Element	Weight%	Atomic%
O K	23.44	47.35
S K	27.42	27.64
Cu K	49.15	25.00
Totals	100.00	100.00

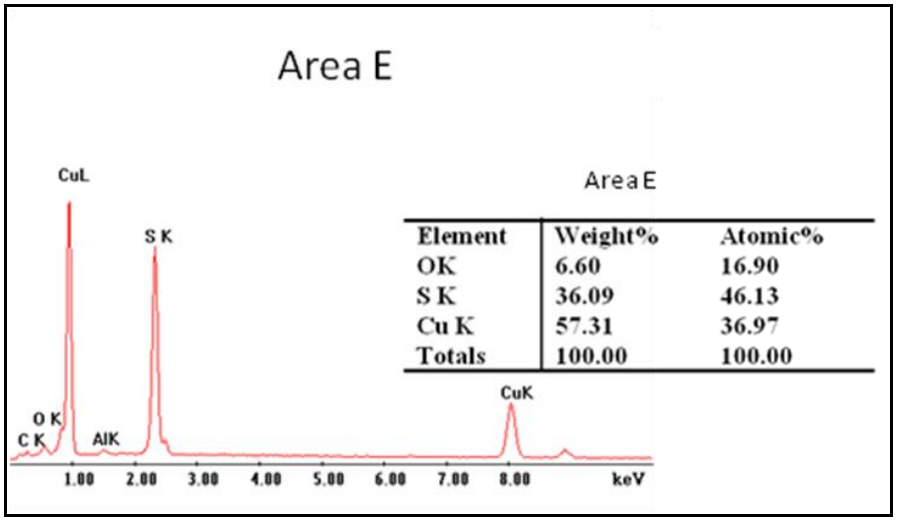


Area C

Element	Weight%	Atomic%
O K	3.66	10.16
S K	32.74	45.37
Cu K	63.60	44.47
Totals	100.00	100.00

Area D

Element	Weight%	Atomic%
O K	10.71	26.29
S K	30.56	37.42
Cu K	58.73	36.29
Totals	100.00	100.00



Area E

Element	Weight%	Atomic%
O K	6.60	16.90
S K	36.09	46.13
Cu K	57.31	36.97
Totals	100.00	100.00

EDX spectra of phase transformed covellite.