

# Evaluation of Processing Parameters in the Solvothermal Synthesis of Cu-Rich Tetrahedrites for Potential Application as Thermoelectric Materials

Itzel J. Soní-Castro<sup>1</sup>, Ana B. López-Oyama<sup>2,3\*</sup>, Eugenio Rodríguez-González<sup>1\*</sup>, Deyanira Del-Ángel-López<sup>1</sup>, Miguel A. Aguilar-Frutis<sup>4</sup> and Juan J. Reyes-Valdez<sup>1</sup>

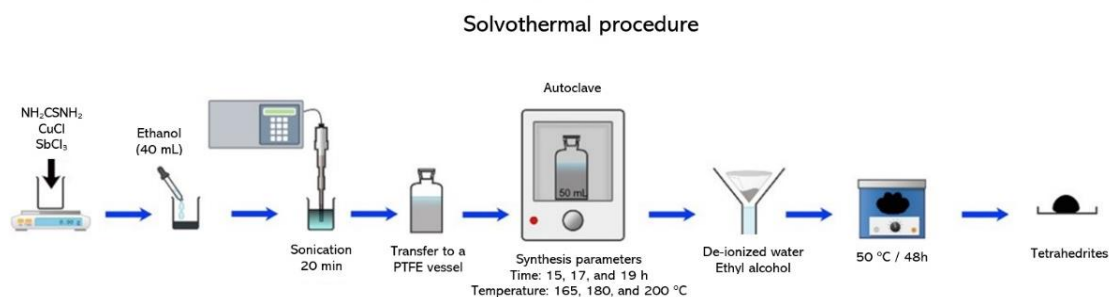
<sup>1</sup>Instituto Politécnico Nacional, Centro de Investigación en Ciencia Aplicada y Tecnología Avanzada Unidad Altamira, Km 14.5 Carr. Puerto Industrial.89600. Altamira, Tamaulipas, México

<sup>2</sup>Departamento de Investigación en Física. Universidad de Sonora, Rosales y Transversal 83000 Hermosillo, Sonora, México

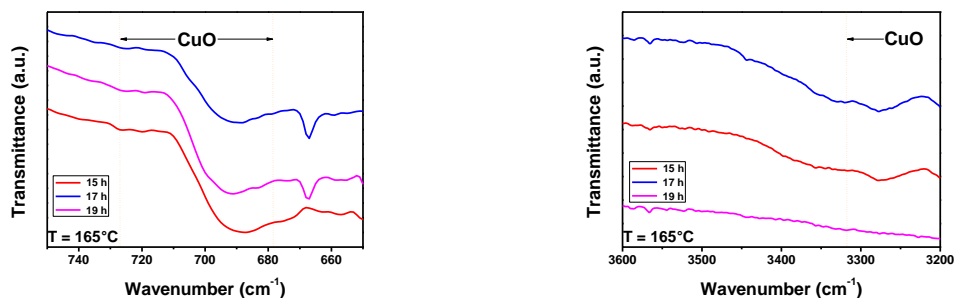
<sup>3</sup>Conahcyt- Universidad de Sonora, Rosales y Transversal 83000 Hermosillo, Sonora, México

<sup>4</sup>Instituto Politécnico Nacional, Calzada Legaria 694, Col Irrigación, Alcaldía Miguel Hidalgo. 11500. Ciudad de México, México

Correspondence: ablopezoy@conahcyt.mx (ABLO); eugenior62@gmail.com (ERG)



**Figure S1. Schematic representation of the process carried out for synthesizing Cu-rich tetrahedrites**



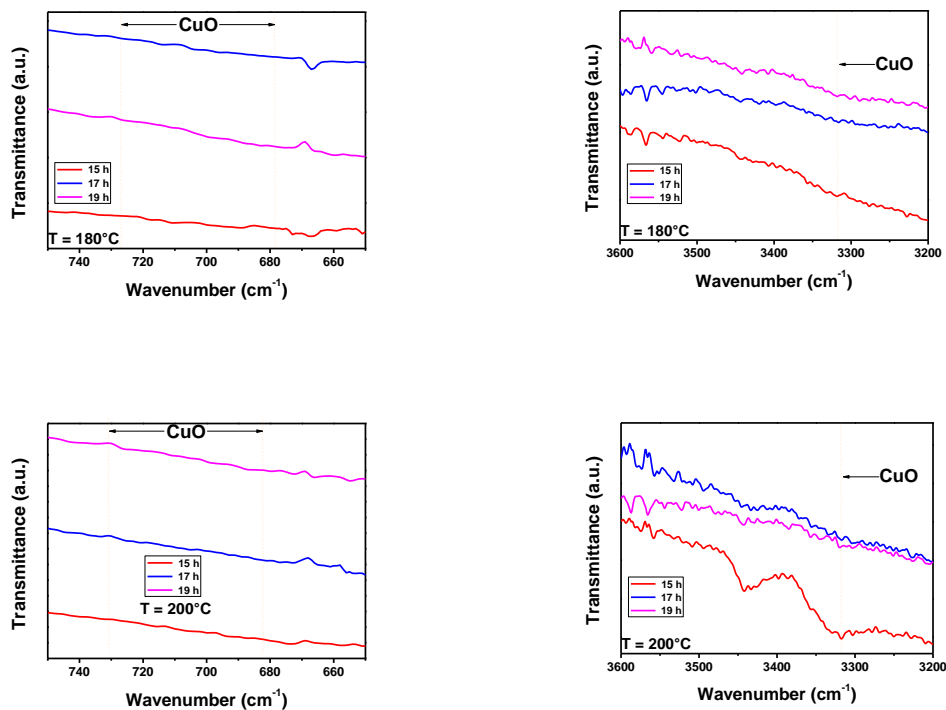


Figure S2. FTIR spectra of tetrahedrites ranged from 650-750  $\text{cm}^{-1}$

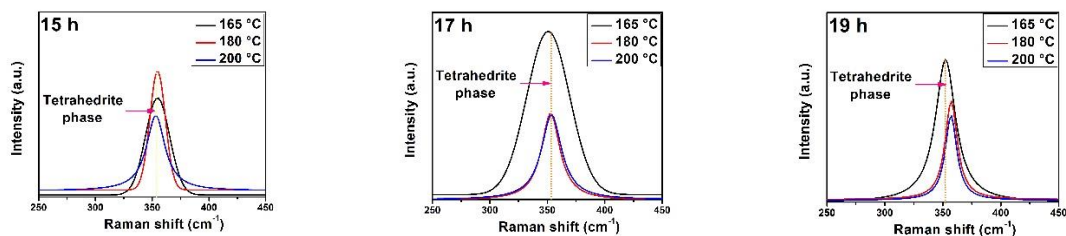
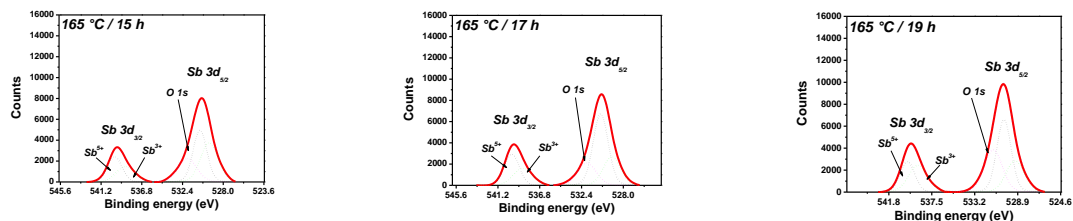


Figure S3. Raman spectra of the vibrational mode at 352  $\text{cm}^{-1}$  attributed to the tetrahedrite phase



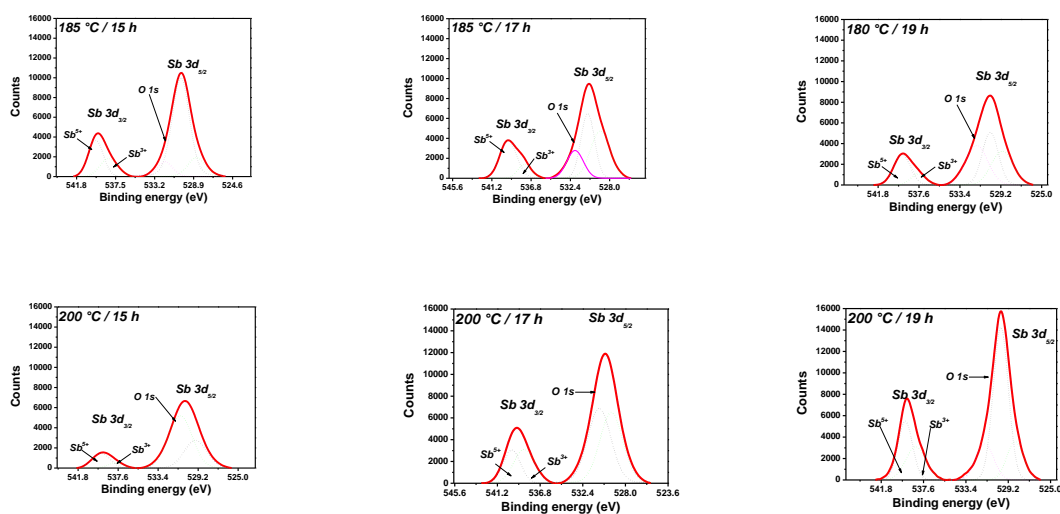


Figure S4. Sb 3d and O 1s core-level of the compounds prepared at 15, 17 and 19 h.

Table S1. Chi-square value extracted from the XPS data.

Solvothermal conditions		Chi-Square value				
Processing temperature	Reaction time (h)	Cu 2p $3/2$	Cu 2p $1/2$	Sb 3d $3/2$	Sb 3d $5/2$	S 2p
165 °C	15	0.839	1.018	0.994	1.680	1.979
	17	0.967	0.764	1.036	1.159	2.04
	19	2.05	1.313	1.423	1.43	1.236
180 °C	15	1.759	2.07	1.11	1.126	1.305
	17	0.973	0.764	1.036	1.466	2.076
	19	1.46	1.11	1.2608	1.426	0.617
200 °C	15	2.906	1.673	1.16	1.802	0.968
	17	2.00	1.15	1.433	1.07	1.0201
	19	2.00	1.925	1.4786	1.915	1.04

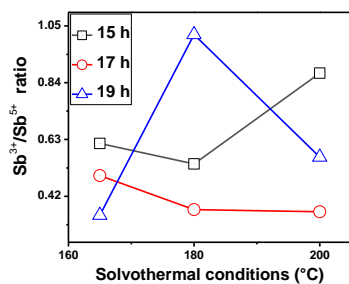


Figure S5.  $\text{Sb}^{3+}/\text{Sb}^{5+}$  ratio of the compounds prepared at different solvothermal conditions

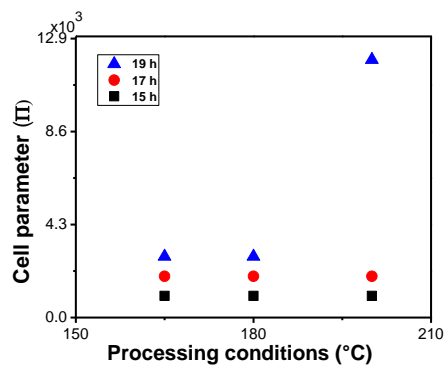


Figure S6. Cell parameters of the compounds prepared at different processing conditions such as time and temperature

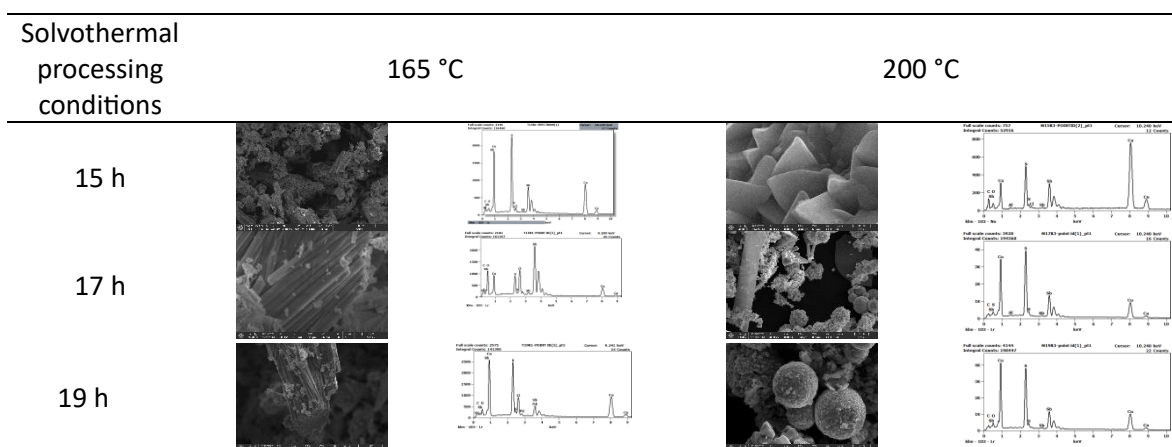


Figure S7. SEM micrographs and their XEDS of the compounds processed at 165 and 200 °C during 15, 17, and 19 h.

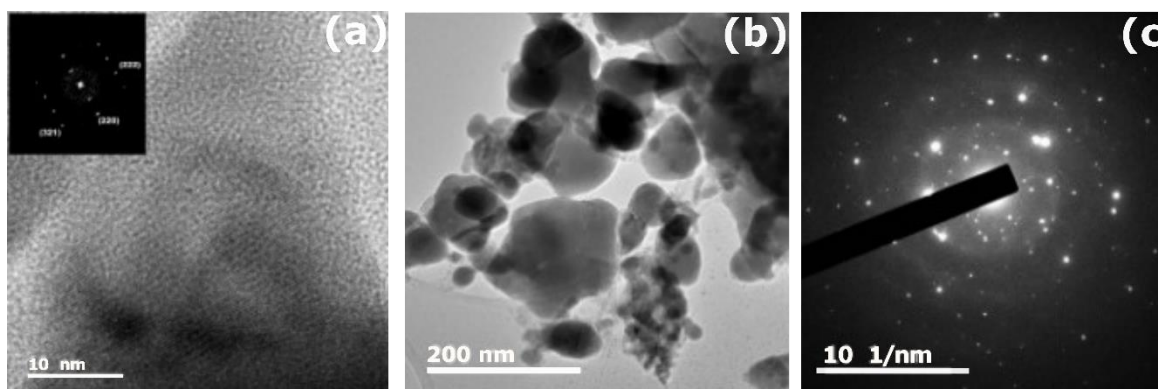


Figure S8. TEM micrographs and SAED pattern of tetrahedrites