

# Structural Study of Sulfur-Added Carbon Nanohorns

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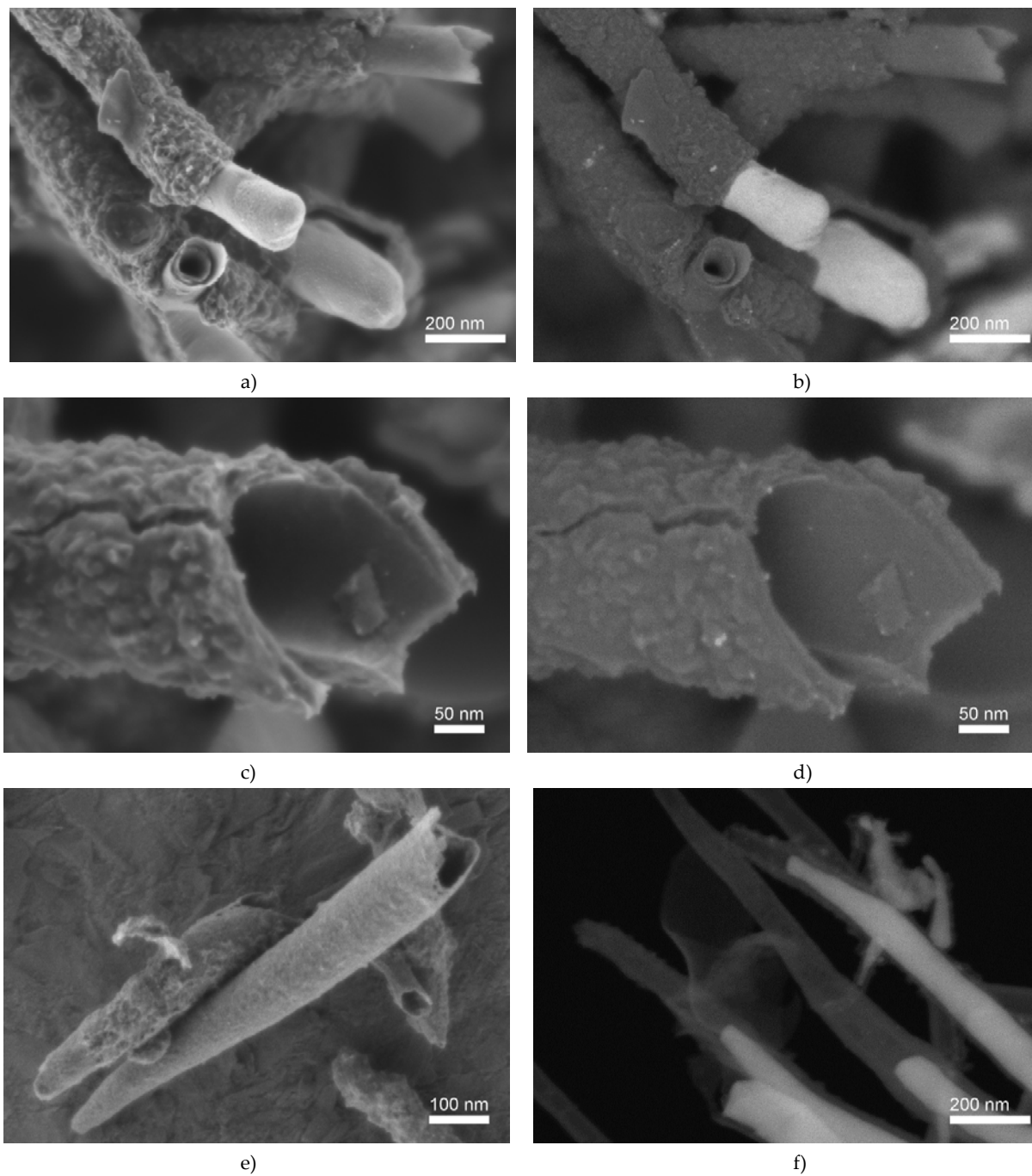
## S1. EDS Results

**Table S1.** Elemental Analysis (EDS) from the Bulk of the Carbon Nanohorns.

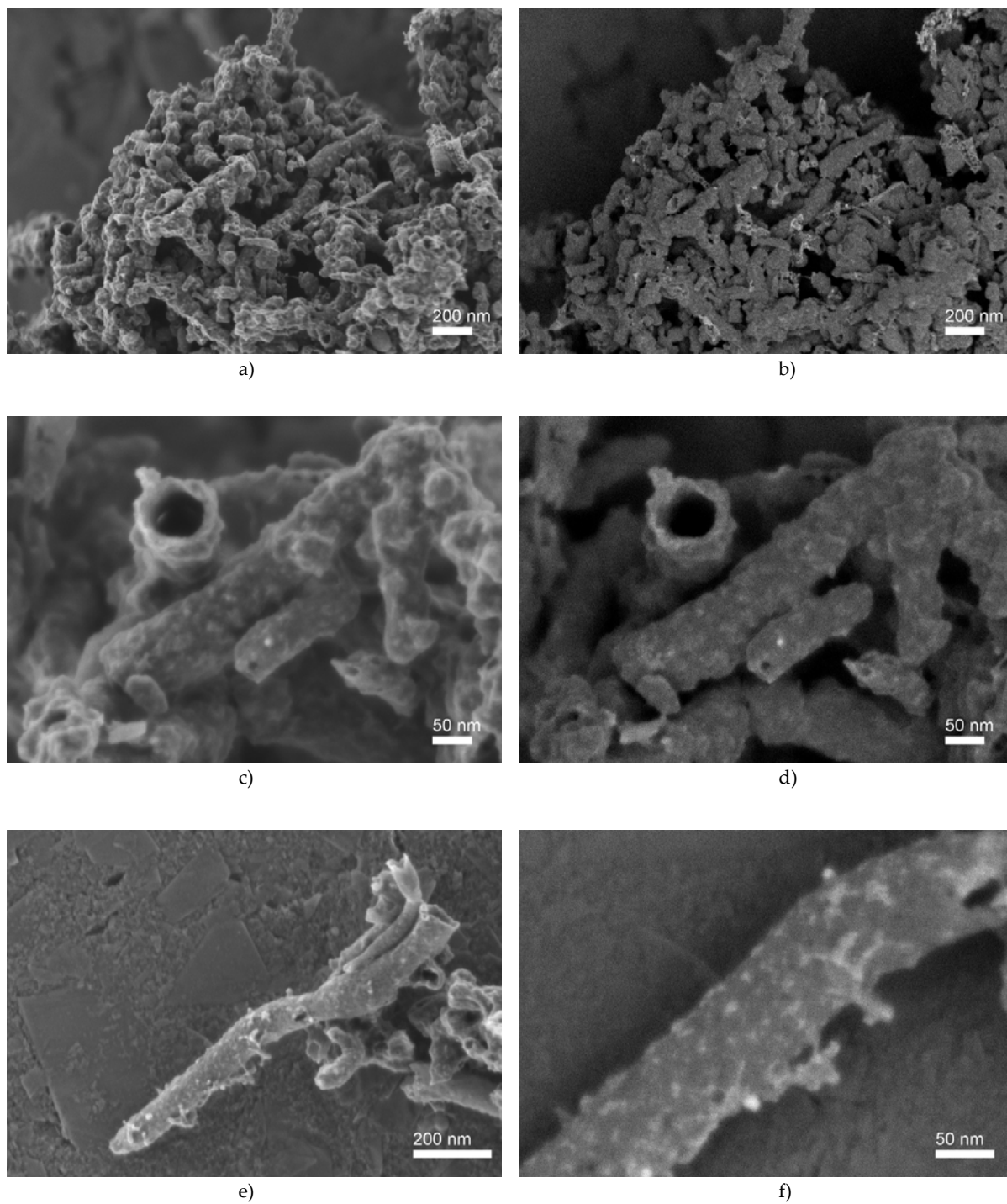
SAMPLE	wt. %				
	C	Fe	S	O	N
SCNA8	80.0	8.7	0.3	7.5	3.3
SCNB8	77.9	6.5	0.3	12.7	2.7
SCNA9	84.9	5.1	1.1	6.0	3.4
SCNB9	81.6	11.0	1.0	6.2	0

## S2. High-Resolution Scanning Electron Microscopy and Microanalysis

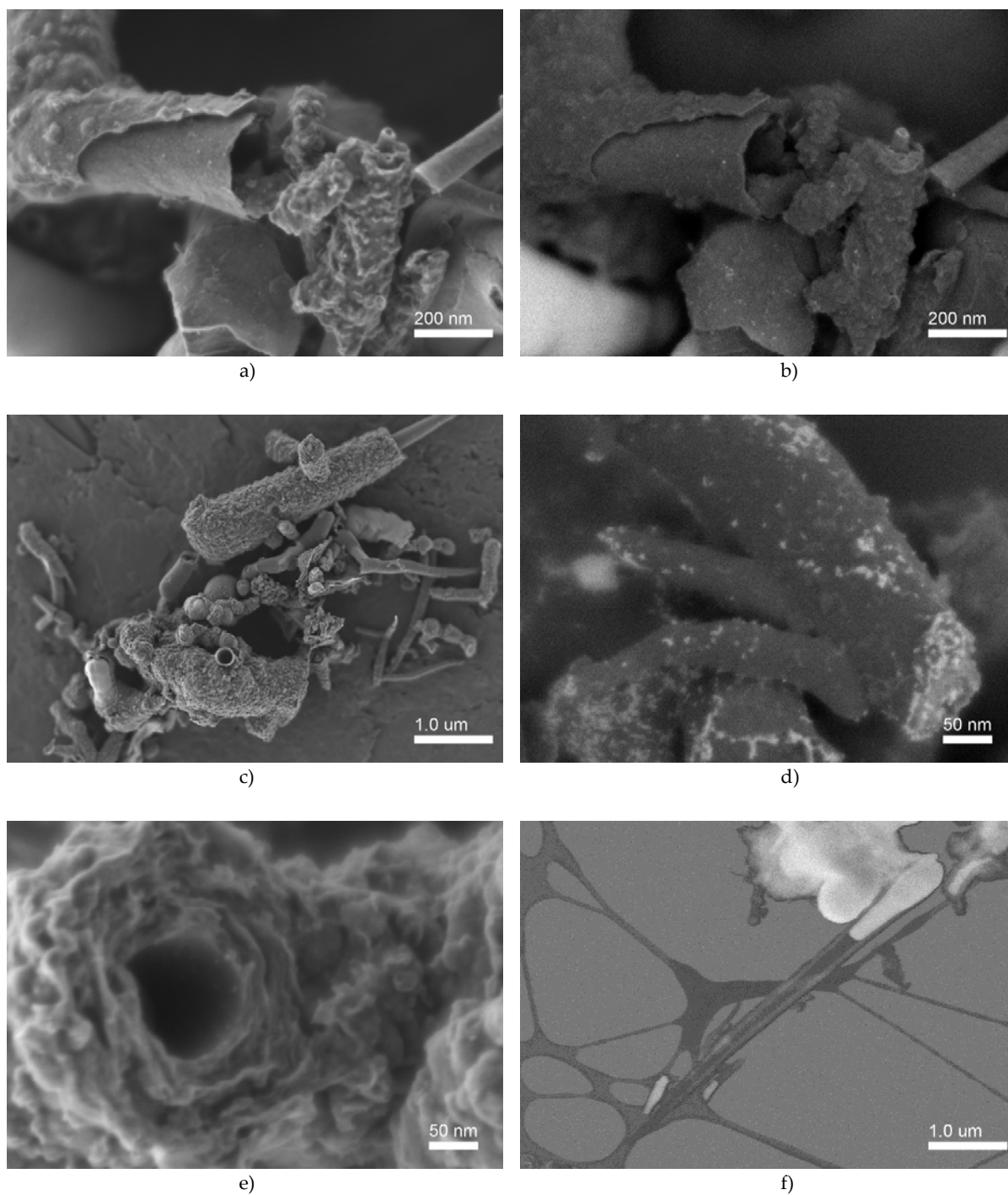
Figures S1 to S4 show the HRSEM micrographs of each sample using different detectors. The horn-like structures, the two layers, and iron nanoparticles (white spots in the backscattering images) can be appreciated in all samples.



**Figure S1.** High-resolution scanning electron microscopy micrographs of the SCNA8 sample using the upper secondary electrons detector (Figures a, c and e) and top energy-filtered backscattered electrons (Figures b and d) in-lens detectors at  $E_0 = 0.5$  kV (a-d) and  $E_0 = 0.2$  kV (e); and a dark-field transmitted electron detector (STEM-DF) at  $E_0 = 30$  kV (f).

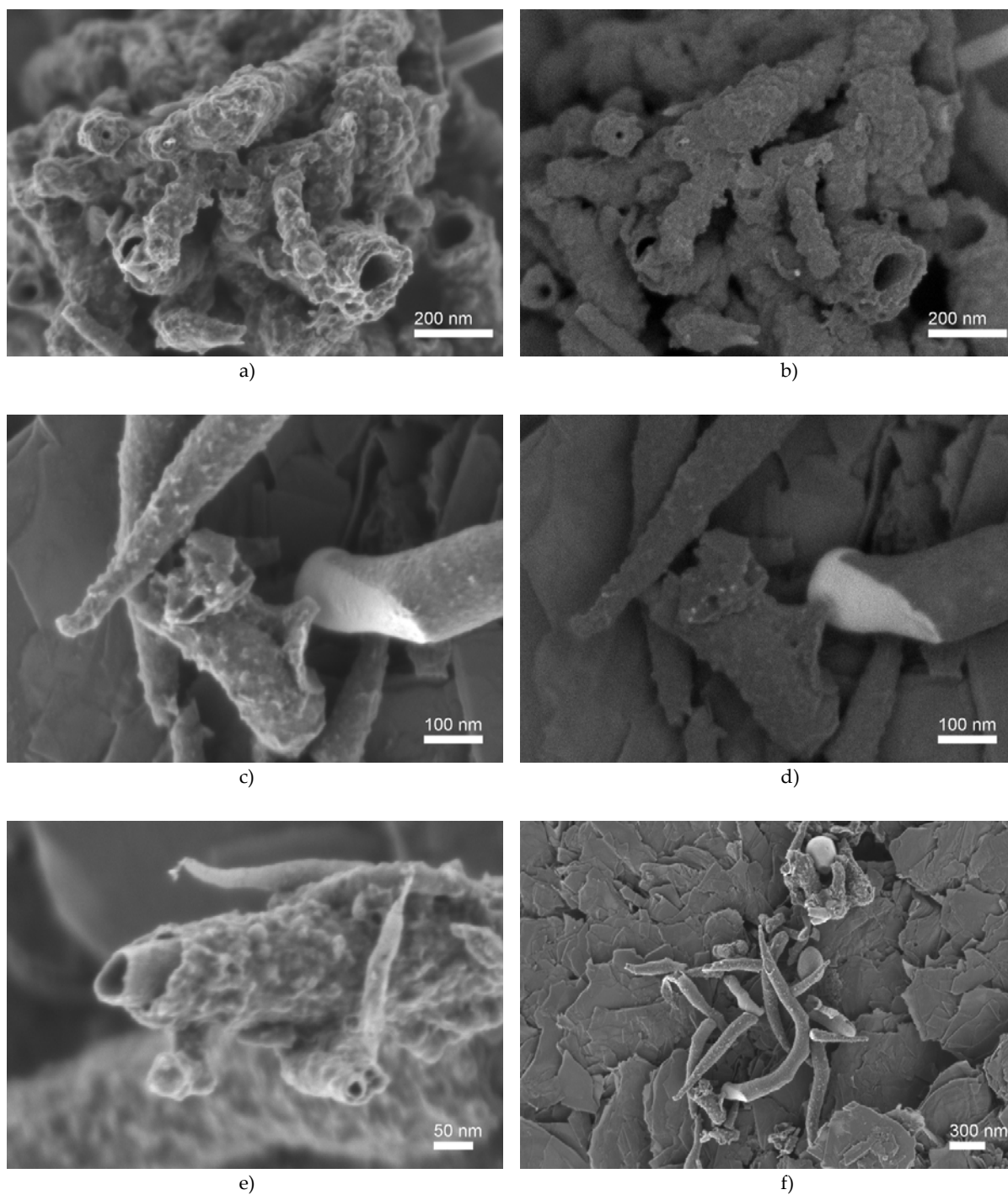


**Figure S2.** High-resolution scanning electron microscopy micrographs of the SCNB8 sample using the upper (secondary electrons, a, c, and e) and top (energy-filtered backscattered electrons, b, d, and f) in-lens detectors at  $E_0 = 0.5$  kV.



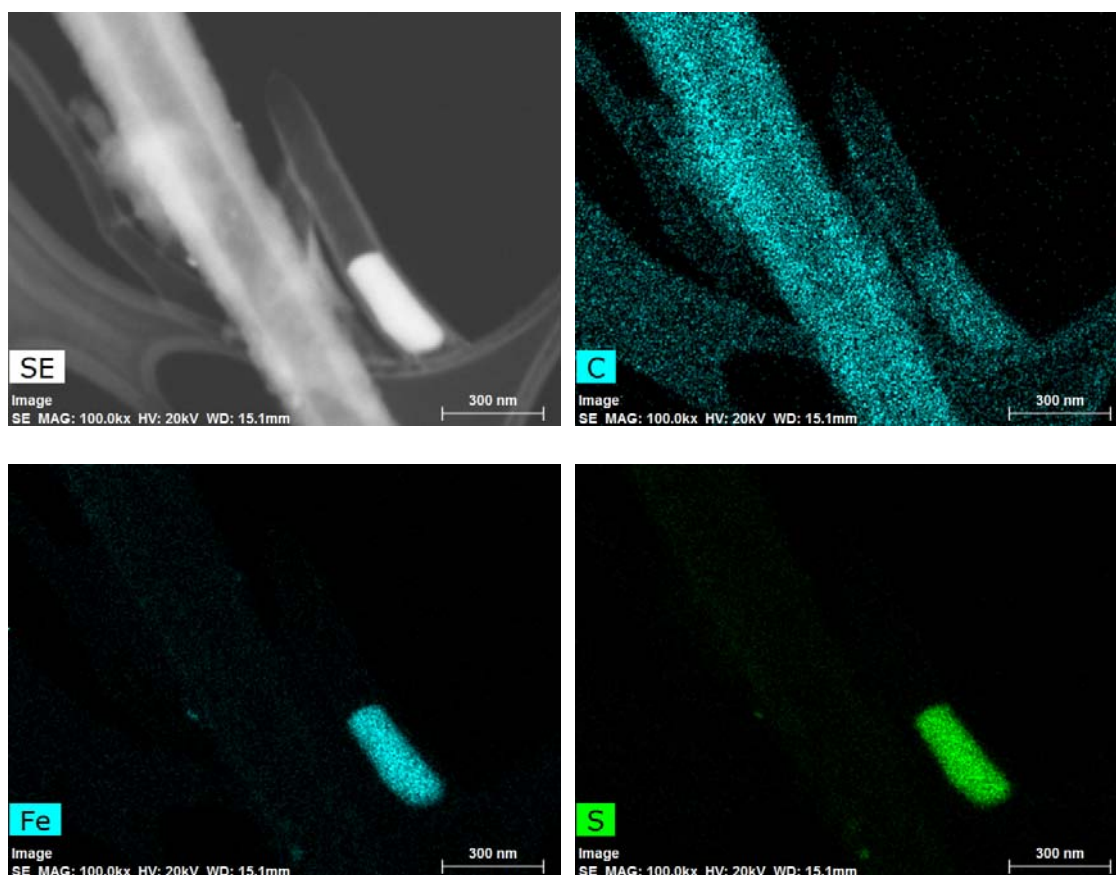
**Figure S3.** High-resolution scanning electron microscopy micrographs of the SCNA9 sample using the upper (secondary electrons, a, c, and e) and top (energy-filtered backscattered electrons, b, and d) in-lens detectors at  $E_0 = 0.5$  kV, and a dark-field transmitted electron detector (STEM-DF) at  $E_0 = 30$  kV (f).





**Figure S4.** High-resolution scanning electron microscopy micrographs of the SCNB9 sample. using the upper (secondary electrons, a, c, e, and f) and top (energy-filtered backscattered electrons, b and d) in-lens detectors at  $E_0 = 0.5$  kV.

In Figure S5, X-ray microanalysis mapping using FlatQuad EDS detector exhibit the presence of iron, carbon, and sulfur in the core of the nanohorns, suggesting the formation of iron sulfide, and iron carbide; consistent with XRD patterns.



**Figure S5.** Elemental X-Ray map of SCNA9 sample in STEM mode at  $E_0 = 20$  kV.