Supplementary material

Synthesis and Characterization of Nanoscale Composite Particles Formed by 2D Layers of Cu-Fe Sulfide and Mg-Based Hydroxide

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Fig. S1. Photos of autoclave in air heating device (thermostat) along with the components and scheme of the pressure vessel: 1 – Teflon lead, 2 – Ti nut, 3 – Teflon liner, 4 – plate, 5 – Ti base body.

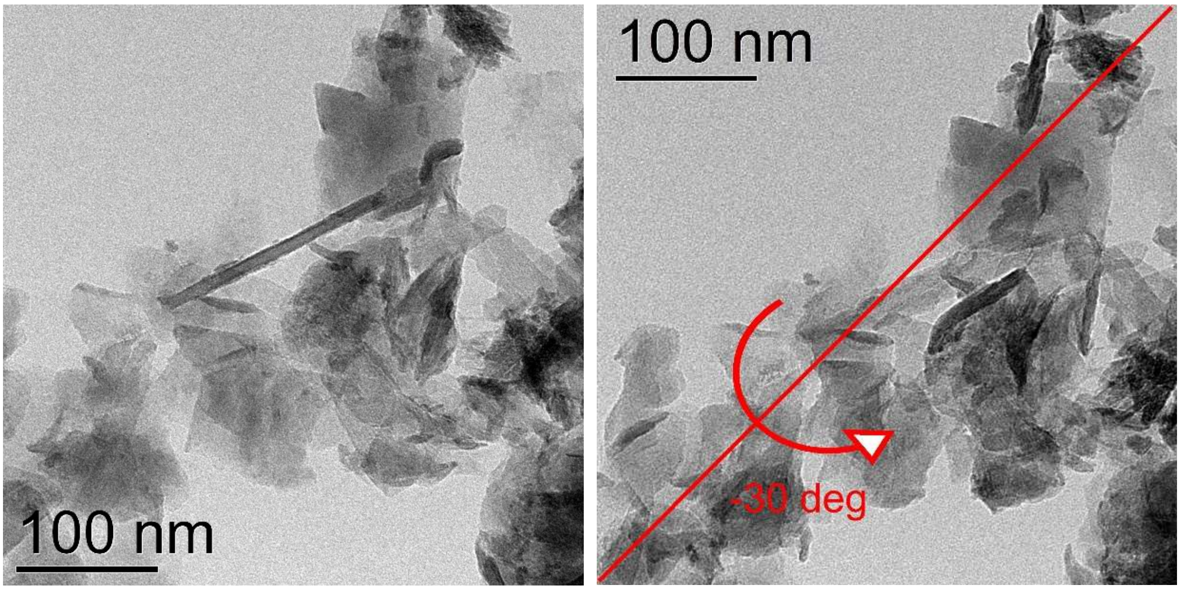

Fig. S2. TEM images from the valleriite (sample b) before and after rotation as shown by red arrow. The Figure demonstrates that entities seen as rods are actually the same particles as the plain ones.

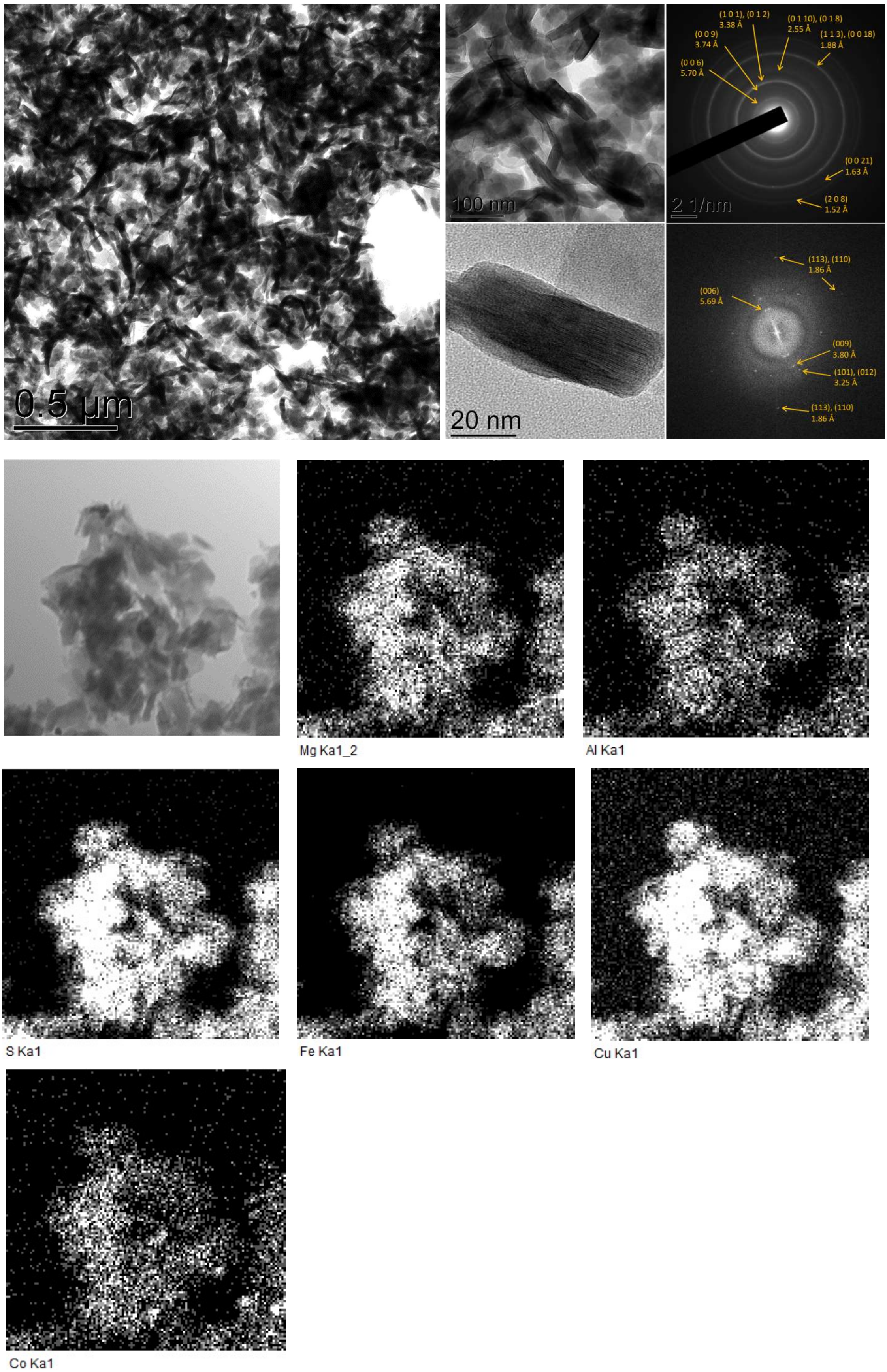

Fig. S3. TEM images, electron diffraction patterns and elemental distribution maps for the valleriite sample prepared with the initial atomic proportion of precursors Fe 1.8, Cu 2, S 14, Mg 1.5, Al 1, Co 0.2 (160 ºC, 40 h) (sample ***g*** in the article). Figure illustrates that elements, including Co dopant, are uniformly distributed within the vallerite product.

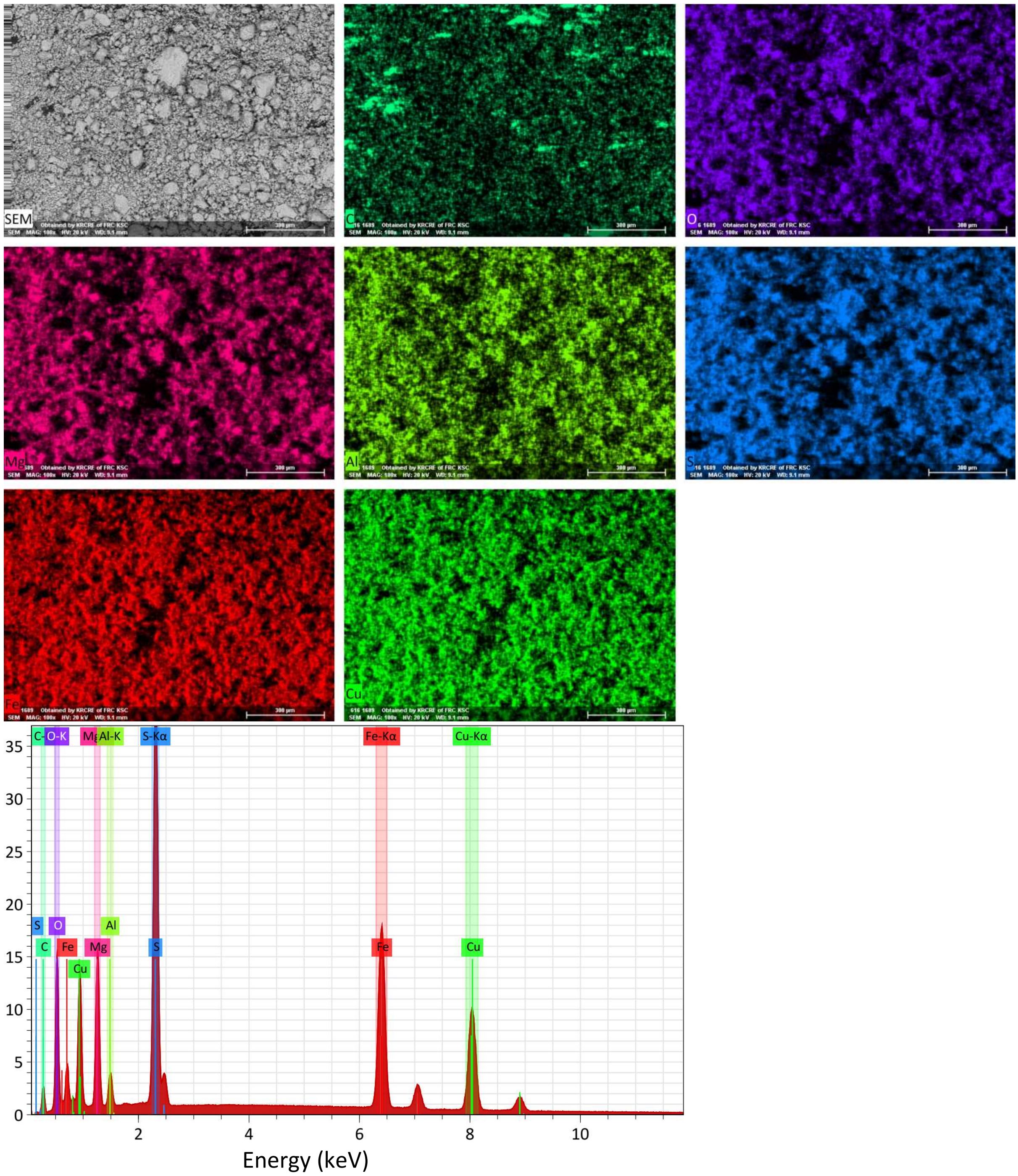

Fig. S4. SEM image, elemental maps, and EDS data (at. %: O 31.7, C 23.2, S 12.3, Cu 10.8, Fe 10.4, Mg 9.8, Al 1.7) for the valleriite sample prepared with the initial atomic proportion of precursors Fe 2, Cu 2, S 14, Mg 2, Al 0.5 (160 ºC, 25 h) (sample ***d*** in the main article).

Table S1. ^{57}Fe Mössbauer fitting parameters for valleriite samples prepared using the atomic precursor ratios listed in the first column and measured at 300 K and 4.2 K. IS is an isomer chemical shift relative to α-Fe (mm/s), QS is a quadrupole splitting (mm/s), W - a width of a doublet line or internal 34 and external 16 lines for sextets (mm/s), H - internal magnetic field (kOe), A - a fraction of a particular iron position.

Conditions of synthesis	300 K						4.2 K						
		IS ±0.02	QS ±0.04	W ±0.04	A ±0.05	Position		IS ±0.02	H ±5	QS ±0.04	W ±0.04	A ±0.05	Position
(a) Fe 2, Cu 1, Mg 2, Al 0, S 10; 33 h	d1	0.33	0.49	0.43	0.69	Fe^{3+}(4S)	s1	0.45	324	-0.07	0.60-0.82	0.24	Fe^{3+}(4S)
	d2	0.37	1.07	0.32	0.27	Fe^{3+}(6OH)	s2	0.46	291	-0.08	0.48-0.98	0.33	Fe^{3+}(4S)
	LS	0.07	0.11	0.15	0.04	Fe(LS)	s3	0.59	279	2.04	0.29-0.73	0.12	Fe^{3+}(6OH)
							s4	0.57	243	2.32	0.19-0.54	0.05	Fe^{3+}(6OH)
							d1	0.43	-	0.52	0.64	0.14	Fe^{3+}(4S)
							d2	0.47	-	1.79	1.70	0.12	Fe^{3+}(6OH)
(b) Fe 2, Cu 1, Mg 2, Al 2, S 14; 33 h	d1	0.33	0.56	0.46	0.73	Fe^{3+}(4S)	s1	0.51	497	-0.35	0.33-0.80	0.19	Fe^{3+}(4S)
	d2	0.39	1.01	0.35	0.23	Fe^{3+}(6OH)	s2	0.47	461	0.20	0.44-0.99	0.20	Fe^{3+}(4S)
	LS	-0.08	0.29	0.15	0.04	Fe(LS)	s3	0.58	337	-0.67	0.32	0.04	Fe^{3+}(4S)
							s4	0.47	310	-0.18	0.44-0.97	0.27	Fe^{3+}(4S)
							s5	0.54	264	0.77	0.60-1.20	0.26	Fe^{3+}(6OH)
							d	0.26	-	0	0.45	0.04	Fe(LS)
(c) Fe 2, Cu 2, Mg 1.5, Al 1.5, S 15; 50 h	d1	0.36	0.63	0.40	0.77	Fe^{3+}(4S)	s1	0.46	499	0	0.65	0.10	Fe^{3+}(4S)
	d2	0.42	1.24	0.26	0.23	Fe^{3+}(6OH)	s2	0.51	457	-0.08	0.60-1.04	0.29	Fe^{3+}(4S)
							s3	0.49	410	0.42	0.24-0.99	0.07	Fe^{3+}(4S)
							s4	0.59	331	-0.64	0.39-0.96	0.18	Fe^{3+}(4S)
							s5	0.62	296	1.10	0.52-1.00	0.30	Fe^{3+}(6OH)
							d	0.23	-	0.76	1.12	0.07	Fe(LS)

(e) Fe 1.8, Cu 2, Mg 1.5, Al 1, **Cr 0.2**, S 15, 50 h	d1	0.33	0.57	0.37	0.51	Fe^{3+}(4S)	s1	0.56	325	-0.52	0.46-9.86	0.41	Fe^{3+}(4S)
	d2	0.39	1.01	0.26	0.14	Fe^{3+}(6OH)	s2	0.58	303	1.07	0.54-0.71	0.38	Fe^{3+}(6OH)
	d3	0.42	1.29	0.27	0.35	Fe^{3+}(6OH)	s3	0.48	261	0.83	0.51-0.81	0.21	Fe^{3+}(6OH)
(f) Fe 1.5, Cu 2, Mg 1.5, Al 1, **Cr 0.5**, S 15, 50 h	d1	0.32	0.54	0.35	0.59	Fe^{3+}(4S)	s1	0.49	464	0	0.32-0.89	0.10	Fe^{3+}(4S)
	d2	0.36	0.80	0.32	0.23	Fe^{3+}(6OH)	s2	0.59	330	-0.37	0.60	0.14	Fe^{3+}(4S)
	d3	0.42	1.24	0.27	0.18	Fe^{3+}(6OH)	s3	0.41	255	0.11	0.41-0.72	0.16	Fe^{3+}(4S)
							s4	0.53	296	0.5	0.46-1.22	0.56	Fe^{3+}(6OH)
							d	0.17	-	0.50	0.36	0.04	Fe(LS)
(h) Fe 1.8, Cu 2, Mg 1.5, Al 1, **La 0.2**, S 15, 50 h	d1	0.27	0.38	0.28	0.14	Fe^{3+}(4S)	s1	0.56	334	-0.61	0.60-0.74	0.30	Fe^{3+}(4S)
	d2	0.27	0.70	0.26	0.25	Fe^{3+}(4S)	s2	0.61	308	0.53	0.37-0.70	0.27	Fe^{3+}(4S)
	d3	0.46	0.75	0.27	0.18	Fe^{3+}(4S)	s3	0.51	275	1.03	0.53-1.02	0.43	Fe^{3+}(6OH)
	d4	0.41	1.26	0.28	0.43	Fe^{3+}(6OH)							