

Supplementary material

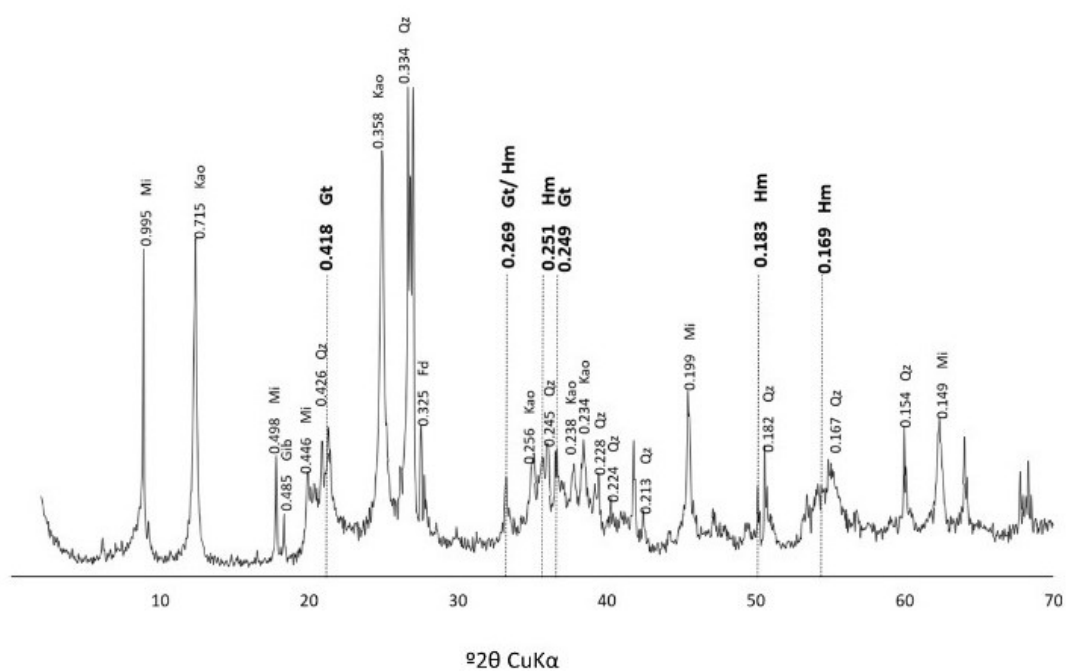


Figure S1. Bulk samples XRD patterns from recently mine tailing deposited at Doce river estuary.

Table S1. Average, standard deviation and p-values from Kruskal-Wallis test.

Variable	Average $\pm$ S.D.						p value	k (Critical value)	K (Observed value)
	Control	Vinasse	Vin + G1	Vin + G2	Vin + G3	Vin + G4			
	378.8	484.4	673.2	639.6	412.1	253.8			
Fe-PY	± 148.9	± 223.9	± 662.5	± 714.7	± 238.7	± 216.1	< 0.001	11.07	19.73
DTMP-Pb	45 ± 29.1	58.6 ± 24.8	18.5 ± 28.8	63.1 ± 19.8	62.4 ± 27.8	68.7 ± 30	< 0.001	11.07	24.52
Fe ²⁺	6.1 ± 2.3	10 ± 6.4	18.5 ± 10	16.9 ± 9.3	19.1 ± 11.6	19.1 ± 12.6	< 0.001	11.07	30.19
H ₂ S	24 ± 7.9	42.3 ± 24.4	33.4 ± 19	44.8 ± 39.1	37.6 ± 21.5	36.7 ± 20.3	0.011	9.49	12.25

Table S2. Reactive Fe (sum of Fe-EX to Fe-OX) and sulfide (H₂S and HS⁻) molar ratio.

S added	Time	Reactive - Fe	Fe/S
mg	days	mg kg ⁻¹	
0	7	32242.6	1289.5
0	14	33800.8	1107.1
0	21	29781.2	1436.6
0	28	29990.1	1531.6
0	35	28990.0	1483.1
2	7	27055.1	795.4
2	14	25862.3	452.0
2	21	26208.5	334.4
2	28	26248.1	1289.5
2	35	30158.4	1504.8
90	7	24387.1	526.0
90	14	28098.8	487.4
90	21	33597.6	2029.0
90	28	26489.9	1230.1
90	35	30466.4	1409.0
439	7	24512.6	472.8
439	14	26625.4	263.8
439	21	28396.2	1538.9
439	28	27093.3	1245.7
439	35	25532.2	1227.0
875	7	25805.1	490.0
875	14	27093.3	395.5
875	21	22497.9	1089.4
875	28	25532.2	1090.4
875	35	23381.3	1123.4
1748	7	22067.7	429.0
1748	14	18061.6	266.6
1748	21	17156.3	870.0
1748	28	18269.4	818.6
1748	35	18270.5	829.9

Table S3. Correlation coefficients (in bold) and p-value of Spearman correlation analysis.

	Time	Fe ²⁺	Sulfides	Fe-EX	Fe-CA	Fe-FR	Fe-LP	Fe-OX	Fe-PY	DOP	Pb-CA	Pb-FR	Pb-LP	Pb-OX	Pb-PY	DTMP-Pb
S_added	0.0000	0.4700	0.1590	0.1180	0.3990	-0.3410	-0.6310	-0.5330	-0.2580	-0.0875	0.2030	-0.1640	-0.6820	-0.0116	0.2730	0.3550
p-value	1.0000	0.0000	0.1350	0.2690	0.0001	0.0011	0.0000	0.0000	0.0144	0.4110	0.0548	0.1210	0.0000	0.9130	0.0093	0.0006
Time		0.3950	-0.6070	0.4200	0.5930	-0.2400	0.1690	-0.1310	-0.0419	-0.0434	0.6030	0.0553	0.0523	0.2330	-0.3080	-0.3360
p-value		0.0001	0.0000	0.0000	0.0000	0.0229	0.1110	0.2190	0.6940	0.6840	0.0000	0.6040	0.6240	0.0273	0.0033	0.0012
Fe ²⁺			-0.2500	0.2210	0.6020	-0.0965	-0.3860	-0.2740	-0.1630	-0.1010	0.4630	-0.0677	-0.3570	0.0826	0.0058	0.0253
p-value			0.0174	0.0362	0.0000	0.3650	0.0002	0.0090	0.1230	0.3430	0.0000	0.5250	0.0006	0.4380	0.9560	0.8120
Sulfides				-0.1260	-0.2830	0.0881	-0.1220	-0.0823	0.0294	0.0906	-0.3480	-0.1250	-0.1570	-0.1560	0.3190	0.4010
p-value				0.2370	0.0070	0.4080	0.2500	0.4400	0.7830	0.3950	0.0008	0.2410	0.1390	0.1420	0.0023	0.0001
Fe-EX					0.5380	-0.0389	0.0102	-0.1520	0.0188	0.0384	0.3010	-0.1430	-0.2000	-0.2050	0.0090	0.0306
p-value					0.0000	0.7150	0.9240	0.1520	0.8600	0.7190	0.0041	0.1770	0.0584	0.0521	0.9320	0.7740
Fe-CA						-0.0589	-0.1100	-0.1570	0.0596	0.0936	0.6820	-0.0887	-0.2550	0.0296	-0.1820	-0.1930
p-value						0.5800	0.3010	0.1380	0.5760	0.3790	0.0000	0.4050	0.0154	0.7810	0.0868	0.0681
Fe-FR							0.0188	0.3310	0.3430	0.2590	-0.0818	0.0558	0.1020	-0.1080	-0.0859	-0.1120
p-value							0.8600	0.0015	0.0010	0.0139	0.4430	0.6010	0.3380	0.3090	0.4200	0.2920
Fe-LP								0.5020	0.2740	0.0740	-0.0048	0.1510	0.6910	0.1850	-0.2360	-0.3430
p-value								0.0000	0.0092	0.4880	0.9640	0.1560	0.0000	0.0815	0.0253	0.0010
Fe-OX									0.2950	0.0011	-0.0356	0.1640	0.4700	0.1120	-0.2030	-0.2830
p-value									0.0049	0.9920	0.7390	0.1220	0.0000	0.2910	0.0550	0.0071
Fe-PY										0.9210	0.1280	-0.0978	0.2290	-0.1210	-0.0034	-0.0944
p-value										0.0000	0.2280	0.3590	0.0299	0.2560	0.9750	0.3760
DOP											0.1720	-0.1570	0.0919	-0.1510	0.0766	-0.0021
p-value											0.1060	0.1390	0.3880	0.1550	0.4720	0.9850
Pb-CA												-0.0959	-0.0092	0.1810	-0.2960	-0.3780
p-value												0.3680	0.9310	0.0873	0.0047	0.0003
Pb-FR													0.0784	0.1420	-0.1230	-0.1600
p-value													0.4620	0.1810	0.2470	0.1320
Pb-LP															0.3100	-0.2830
p-value															0.0030	0.0071
Pb-OX																-0.2080
p-value																0.0489
Pb-PY																
p-value																
DTMP-Pb																
p-value																

Table S4. Pb solid-phase geochemical fractionation in function of S amendment over time.

S added	Time	Pb-EX	Pb-CA	Pb-FR	Pb-LP	Pb-OX	Pb-PY
mg	days	mg kg ⁻¹					
0	7	0.0	0.2	0.1	2.5	1.0	7.0
0	14	0.0	0.2	0.1	1.9	0.9	9.3
0	21	0.0	0.0	4.1	2.1	1.2	3.3
0	28	0.0	0.4	0.5	3.2	1.2	3.8
0	35	0.0	0.6	0.0	3.6	1.6	0.9
2	7	0.0	0.1	0.0	2.0	0.8	7.6
2	14	0.0	0.3	0.0	1.9	1.0	6.1
2	21	0.0	0.4	0.0	1.7	1.0	5.7
2	28	0.0	0.5	0.0	1.7	0.8	6.6
2	35	0.0	0.9	0.0	1.8	0.9	2.7
90	7	0.0	1.0	0.0	2.1	1.1	11.2
90	14	0.0	1.0	0.0	1.5	1.0	2.4
90	21	0.0	0.9	0.1	1.6	1.0	0.1
90	28	0.0	2.8	0.0	2.7	1.4	0.4
90	35	0.0	3.1	0.0	2.8	1.2	0.0
439	7	0.0	0.0	0.0	1.6	0.9	8.7
439	14	0.0	0.6	0.2	1.4	0.9	11.9
439	21	0.0	0.7	0.0	1.2	1.0	7.2
439	28	0.0	0.9	0.2	0.9	1.2	6.1
439	35	0.0	2.5	0.0	1.7	1.1	4.3
875	7	0.0	0.1	0.0	1.1	1.0	7.3
875	14	0.0	0.6	0.0	0.8	1.1	11.4
875	21	0.0	0.7	0.0	0.7	1.0	9.1
875	28	0.0	0.8	0.5	0.7	1.1	6.8
875	35	0.0	1.8	0.0	1.3	0.9	7.5
1748	7	0.0	0.1	0.0	1.5	1.1	8.8
1748	14	0.0	0.3	0.0	0.2	0.8	8.2
1748	21	0.0	0.8	0.0	0.4	1.2	3.3
1748	28	0.0	0.7	0.0	0.6	1.2	15.7
1748	35	0.0	1.1	0.0	0.5	1.1	6.5