



Supplement of

Technical note: assessing pretreatment approaches for serial pyrolysis-oxidation analysis of sedimentary organic carbon

Songfan He et al.

Correspondence to: Huiyuan Yang (yhy_0707@sjtu.edu.cn) and Xingqian Cui (cuixingqian@sjtu.edu.cn)

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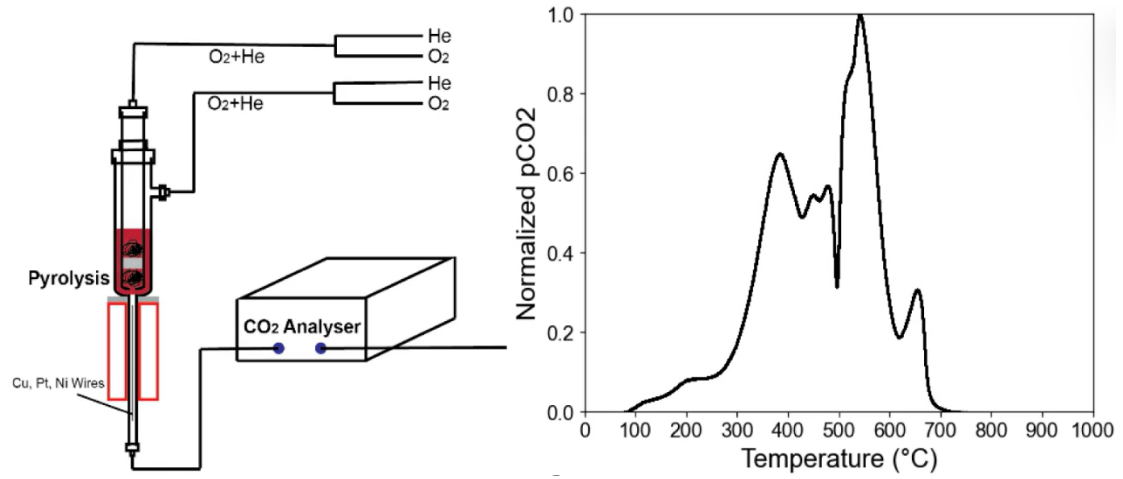


Fig S1: Schematic illustration of the ramped-temperature pyrolysis/oxidation instrument and a representative thermogram.

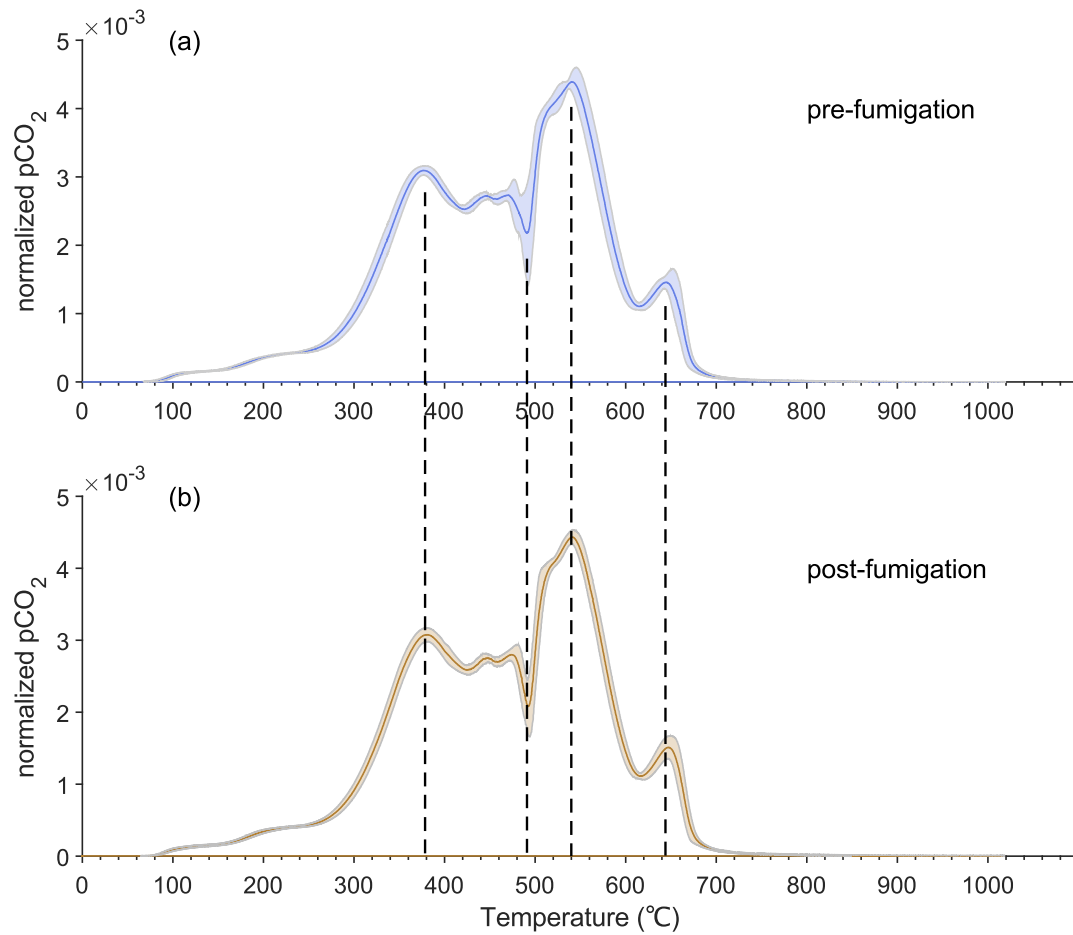


Fig S2: Normalized thermograms of the standard sample (Irati T2) analyzed before and after analyses of acid-fumigated subsamples. No obvious shifts are observed when comparing the thermograms.

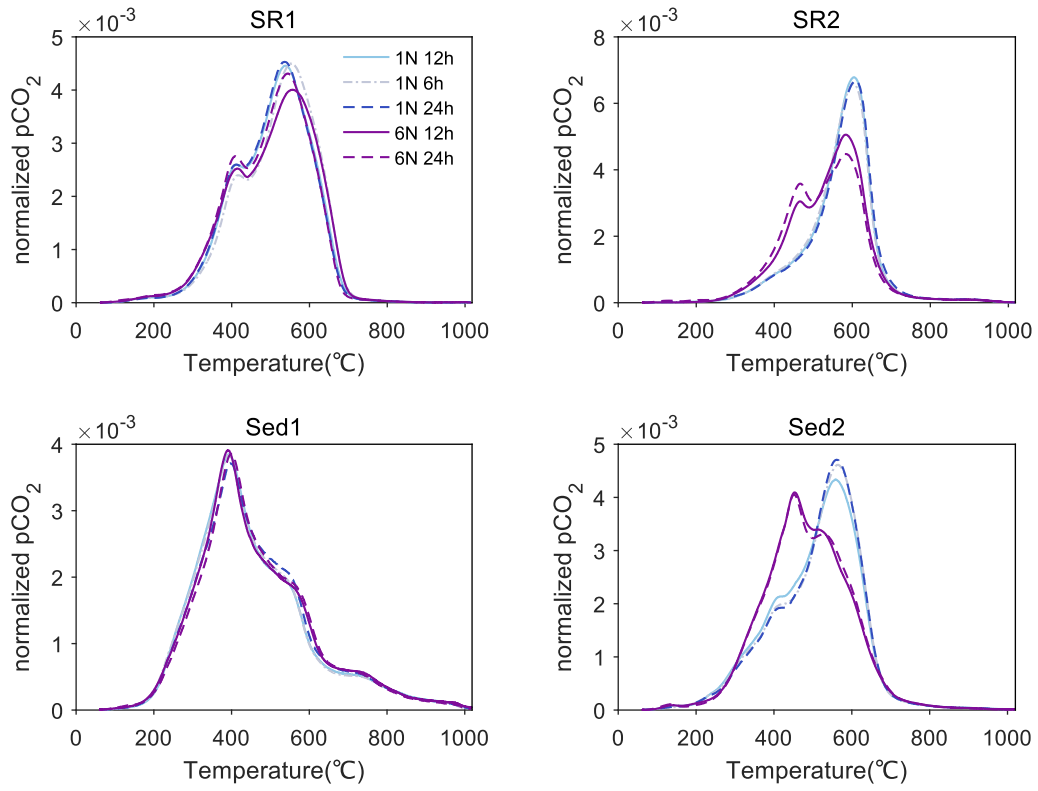


Fig S3: Normalized thermograms of subsamples with different reaction times under 1 N (blue and grey) and 6 N (dark purple) HCl treatments.

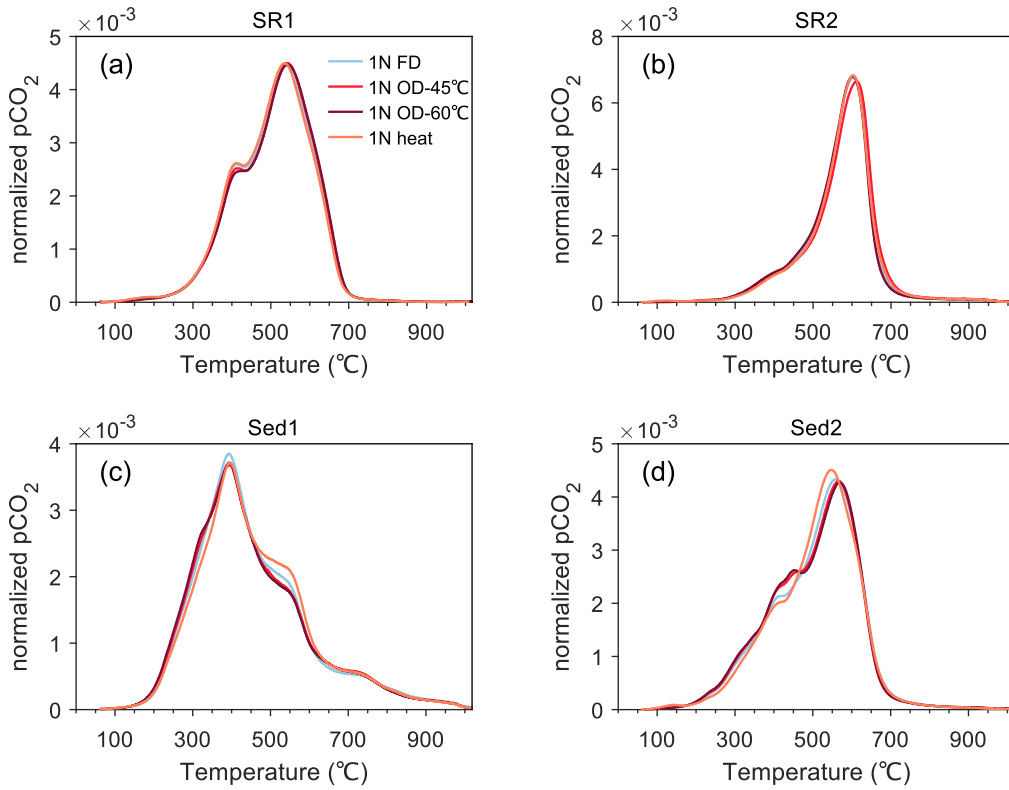


Fig S4: Normalized thermograms of control groups, oven-dried subsamples and heated ones. FD: freeze-dried; OD: oven-dried; heat: heat during decarbonation (60 °C).



Fig S5: The image of outer quartz tubes indicates significant corrosion and a mass of remnants after analyzing acid-fumigated subsamples. From the left to the right, the first is a recently washed quartz tube; the second has undergone combustion of numerous (>20 counts) acid-rinsed samples; Others are the quartz tubes that have been utilized to analyze acid-fumigated samples in this study (SR1, SR2, Sed1, and Sed2). The black remnants likely result from incomplete combustion, whereas the green parts are likely chlorides.

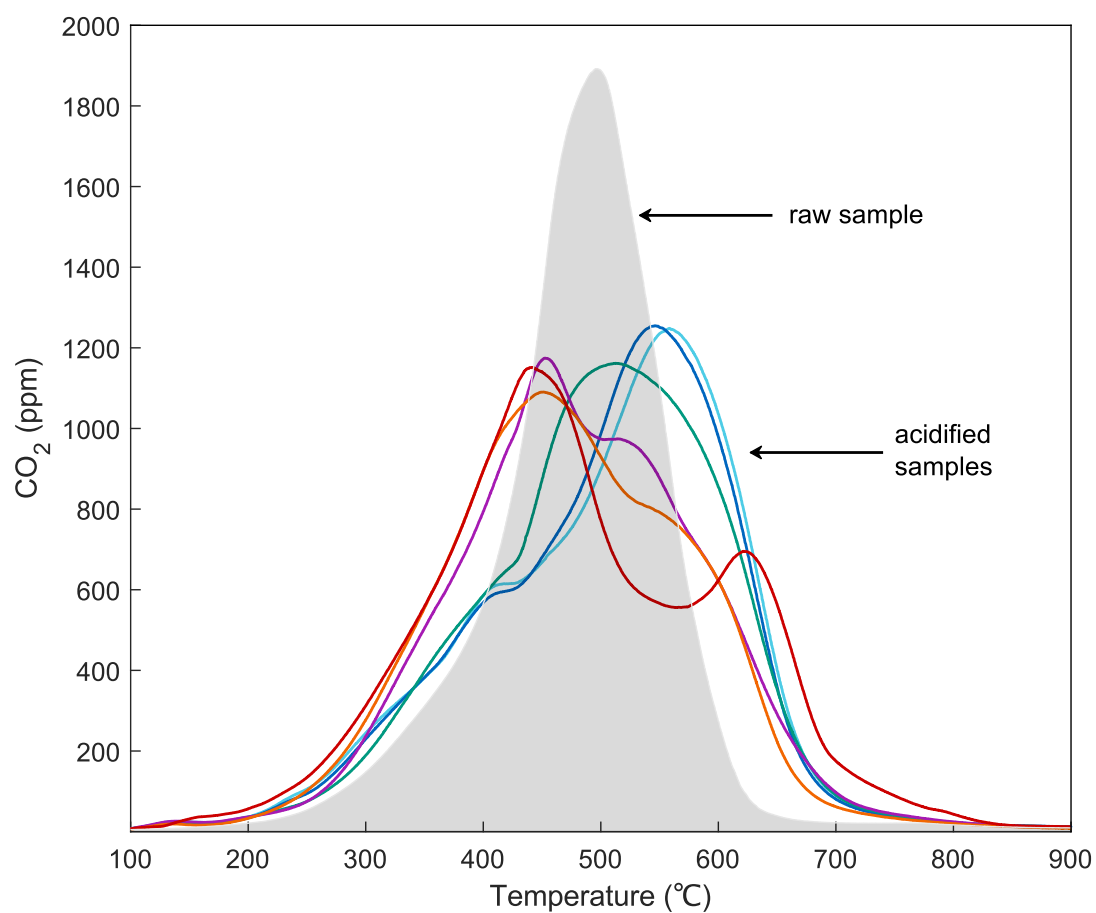


Fig S6: Thermograms of unacidified and acidified Sed2 subsamples. No thermographic spike is observed at high temperatures for the unacidified raw aliquot (gray), suggestive of carbonate decomposition at relatively low temperatures.

Table S1. Combinations of experimental conditions in this study, including acidification methods, HCl concentrations, reaction durations and temperature, as well as drying methods and temperature.

Experimental conditions					
Acidification method	HCl concentration	Reaction temperature	Duration	Drying method	Abbreviation*
Rinsing	1 N	Ambient	12 h	Freeze-drying	EC-1 [§]
Rinsing	2 N	Ambient	12 h	Freeze-drying	EC-2
Rinsing	4 N	Ambient	12 h	Freeze-drying	EC-3
Rinsing	6 N	Ambient	12 h	Freeze-drying	EC-4
Rinsing	1 N	Ambient	6 h	Freeze-drying	EC-5
Rinsing	1 N	Ambient	24 h	Freeze-drying	EC-6
Rinsing	1 N	Ambient	12 h	Oven drying, 45°C	EC-7
Rinsing	1 N	Ambient	12 h	Oven drying, 60°C	EC-8
Rinsing	1 N	60°C	12 h	Freeze-drying	EC-9
Rinsing	6 N	Ambient	24 h	Freeze-drying	EC-10
Fumigation + Rinsing	12 N	Ambient	12 h	Freeze-drying	EC-11
Fumigation	12 N	Ambient	12 h	Oven drying, 60°C	EC-12

*“EC” is the abbreviation of “experimental condition”, and the suffixal numbers are for sorting purpose.

[§]The first line is the control group (1 N HCl). We emphasize it is not necessarily to be the best choice of experimental condition. It is based on the experiment design and is only used to facilitate comparisons between different conditions.

Table S2. Bulk carbon isotopic data and RPO parameters of all subsamples. Bulk parameters include TOC and $\delta^{13}\text{C}_{\text{org}}$; RPO parameters include the average activation energy (μ_E), the standard deviation of activation energy (σ_E), the fractions of OC with activation energy lower than 150 kJ mol⁻¹ ($f_{E<150}$) and lying within 150-185 kJ mol⁻¹ ($f_{150\leq E<185}$). Carbonate contents are included as well. Each row stands for a subsample subjected to a specific experimental condition. See Table S1 for detailed information of different experimental conditions. N. A. indicates not applicable.

Sample ID	Pretreatment	Mass loss [#] (%)	Bulk		RPO			
			TOC (%)	$\delta^{13}\text{C}_{\text{org}}$ (VPDB, ‰)	μ_E (kJ mol ⁻¹)	σ_E (kJ mol ⁻¹)	$f_{E<150}$	$f_{150\leq E<185}$
SR1	EC-1	68.4	0.525	-28.95	188.51	22.97	0.046	0.309
SR1	EC-2	69.5	0.519	-29.26	188.89	23.50	0.047	0.306
SR1	EC-3	70.3	0.519	-28.90	188.91	24.16	0.051	0.307
SR1	EC-4	70.5	0.520	-29.27	189.13	24.94	0.059	0.304
SR1	EC-5	68.7	0.525	-28.95	191.46	23.30	0.040	0.270
SR1	EC-6	68.5	0.522	-29.04	188.01	22.66	0.047	0.311
SR1	EC-7	68.3	0.532	-28.94	189.40	22.88	0.045	0.296
SR1	EC-8	69.1	0.521	-29.01	189.73	22.72	0.044	0.291
SR1	EC-9	68.4	0.535	-28.96	187.83	22.95	0.048	0.316
SR1	EC-10	70.2	0.533	-29.03	186.58	24.26	0.064	0.324
SR1	EC-11	71.6	0.482	-29.03	188.08	24.54	0.058	0.313
SR1	EC-12	N. A.	0.518	-28.94	165.85	19.41	0.174	0.705
SR2	EC-1	8.8	0.586	-24.48	204.40	21.33	0.016	0.135
SR2	EC-2	10.0	0.610	-24.46	203.94	21.27	0.015	0.138
SR2	EC-3	11.0	0.597	-24.27	202.81	22.03	0.019	0.127
SR2	EC-4	11.5	0.604	-24.15	197.39	23.47	0.025	0.243
SR2	EC-5	7.7	0.584	-24.22	203.73	21.64	0.018	0.132
SR2	EC-6	10.0	0.607	-24.43	205.10	22.19	0.019	0.129
SR2	EC-7	8.4	0.580	-24.41	205.48	22.28	0.019	0.128
SR2	EC-8	8.9	0.596	-24.49	202.89	21.71	0.020	0.141
SR2	EC-9	10.7	0.596	-24.37	204.54	22.39	0.019	0.125
SR2	EC-10	11.6	0.604	-24.57	194.20	25.11	0.034	0.297
SR2	EC-11	12.8	0.587	-24.65	193.54	23.57	0.034	0.284
SR2	EC-12	N. A.	0.568	-24.51	180.33	24.71	0.059	0.581
Sed1	EC-1	13.7	0.395	-22.82	175.58	35.14	0.205	0.447
Sed1	EC-2	14.5	0.388	-23.09	175.47	35.25	0.205	0.443
Sed1	EC-3	16.4	0.387	-22.90	176.48	35.33	0.192	0.438
Sed1	EC-4	17.7	0.373	-23.08	177.18	35.55	0.186	0.443
Sed1	EC-5	13.4	0.408	-23.09	175.54	35.28	0.206	0.444

Sed1	EC-6	14.5	0.394	-22.77	177.36	35.23	0.194	0.430
Sed1	EC-7	13.2	0.398	-22.66	175.35	35.71	0.220	0.435
Sed1	EC-8	13.4	0.408	-22.80	174.56	35.58	0.230	0.432
Sed1	EC-9	15.3	0.397	-22.88	177.39	35.15	0.189	0.432
Sed1	EC-10	18.3	0.385	-23.30	178.32	35.22	0.173	0.445
Sed1	EC-11	16.8	0.402	-22.99	175.82	34.95	0.198	0.444
Sed1	EC-12	N. A.	0.440	-23.31	169.29	32.61	0.293	0.388
Sed2	EC-1	13.8	1.224	-26.84	188.64	26.88	0.088	0.269
Sed2	EC-2	15.0	1.153	-26.85	188.38	26.25	0.083	0.267
Sed2	EC-3	17.2	1.131	-27.16	187.51	25.61	0.069	0.323
Sed2	EC-4	18.3	1.144	-27.03	182.93	26.08	0.083	0.425
Sed2	EC-5	13.2	1.210	-26.89	189.99	26.59	0.085	0.244
Sed2	EC-6	14.6	1.147	-26.80	190.39	26.33	0.079	0.240
Sed2	EC-7	13.2	1.175	-27.12	187.66	26.94	0.095	0.289
Sed2	EC-8	13.6	1.152	-26.92	187.60	27.02	0.096	0.291
Sed2	EC-9	15.5	1.179	-27.20	189.16	26.12	0.075	0.263
Sed2	EC-10	17.7	1.182	-26.84	183.56	26.23	0.082	0.418
Sed2	EC-11	20.9	1.128	-27.13	180.57	25.71	0.097	0.461
Sed2	EC-12	N. A.	1.230	-26.87	183.41	30.45	0.111	0.449

[#]This column (mass loss) is calculated by the mass difference before and after acidification and drying.

We do not include EC-12 because calcium chloride remains in solids, leading to weight increase after acidification.