

Supplementary Material for

Optimizing Conditions for the Formation of Calcium Oxalate Trihydrate Using Response Surface Methodology

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Content of listed material

Table ST1. Experimental conditions for the precipitation systems (Runs 1–17): temperature (t), initial pH, and calculated values of ionic strength (I_c), saturation indices (S_i), saturation ratios (S), and ion activity products ($\log IAP$) with respect to calcium oxalate monohydrate (COM) and trihydrate (COT).

Run	Temperature / °C	Ionic strength / mol dm ⁻³	pH	S_i (COM)	S (COM)	$\log IAP$ (COM)	S_i (COT)	S (COT)	$\log IAP$ (COT)
1, 6, 7, 10, 16	36.5	0.05	6.5	2.085	11.028	-6.665	1.653	6.707	-6.667
2	36.5	0.01	5.6	2.234	13.092	-6.516	1.804	7.980	-6.516
3	36.5	0.10	5.6	1.933	9.258	-6.817	1.500	5.623	-6.820
4	36.5	0.10	7.5	1.938	9.311	-6.812	1.505	5.656	-6.815
5	48	0.05	5.6	2.071	10.852	-6.679	1.639	6.599	-6.681
8	48	0.01	6.5	2.236	13.122	-6.514	1.805	7.989	-6.515
9	25	0.10	6.5	1.954	9.484	-6.796	1.521	5.761	-6.799
11	48	0.10	6.5	1.922	9.141	-6.828	1.489	5.553	-6.831
12	48	0.05	7.5	2.076	10.914	-6.674	1.645	6.645	-6.675
13	25	0.05	7.5	2.095	11.156	-6.655	1.663	6.784	-6.657
14	36.5	0.01	7.5	2.239	13.167	-6.511	1.809	8.026	-6.511
15	25	0.01	6.5	2.241	13.198	-6.509	1.811	8.045	-6.509
17	25	0.05	5.6	2.090	11.092	-6.660	1.659	6.753	-6.661

Table ST2. IR vibration bands (in cm⁻¹) of calcium oxalate standards COM and COT.³⁴

COM	COT	Vibrational mode
3483	3528	
3429	3427	$\nu(\text{OH})$
3336	3222	
3258		$2\delta(\text{HOH})$
3058		$\nu(\text{OH})$
1624-1622	1636	$\nu_o(\text{COO}) - \nu_a(\text{CO})$
1384		$\nu_s(\text{CO})$
1368		
1320-1316	1324	$\nu_s(\text{COO})$
958-889		$L(\text{H}_2\text{O})$
782	783	$\nu_s(\text{CO}) + L(\text{H}_2\text{O})$
656	651	$\delta(\text{OCO})$
		$L(\text{HOH})$

ν – stretching; δ – bending; α -antisymmetric; s – symmetric; L - libration.

Table ST3. Phase composition (wt%) of calcium oxalate hydrates and corresponding Rietveld refinement indicators (R_e , R_p , R_{wp} , GoF) for each experimental Run.

Run	Phase composition (wt %)		R_e (%)	R_p (%)	R_{wp} (%)	GoF
	COT	COM				
1	100	0	3.891	9.020	11.991	9.498
2	44.6	55.4	4.117	8.298	11.855	8.291
3	41.2	58.8	4.344	5.566	7.490	2.966
4	100	0	4.481	8.611	14.311	10.198
5	0	100	4.313	6.627	9.290	4.640
6	99.4	0.6	3.957	8.586	12.712	10.318
7	100	0	4.230	10.298	15.670	13.723
8	0	100	4.316	7.244	10.346	5.746
9	100	0	4.128	8.823	12.524	9.203
10	100	0	4.352	8.779	11.765	9.657
11	0	100	4.190	7.763	10.991	6.879
12	0	100	4.351	6.878	9.983	5.266
13	100	0	3.966	9.575	13.336	11.308
14	99.8	0.2	4.210	10.799	16.545	15.448
15	98.8	0.2	4.060	11.792	16.721	16.965
16	100	0	3.879	10.166	15.789	15.998
17	100	0	4.053	10.280	14.771	13.284

Figure SF1. Light microscope images of all precipitated samples. Note the difference in magnification scale (Runs 1, 2, 3, 4, 6, 7, 9, 10, 15, 16, and 17 at 10×; Runs 5, 8, 11, 12, 13, and 14 at 40×).

Figure SF1

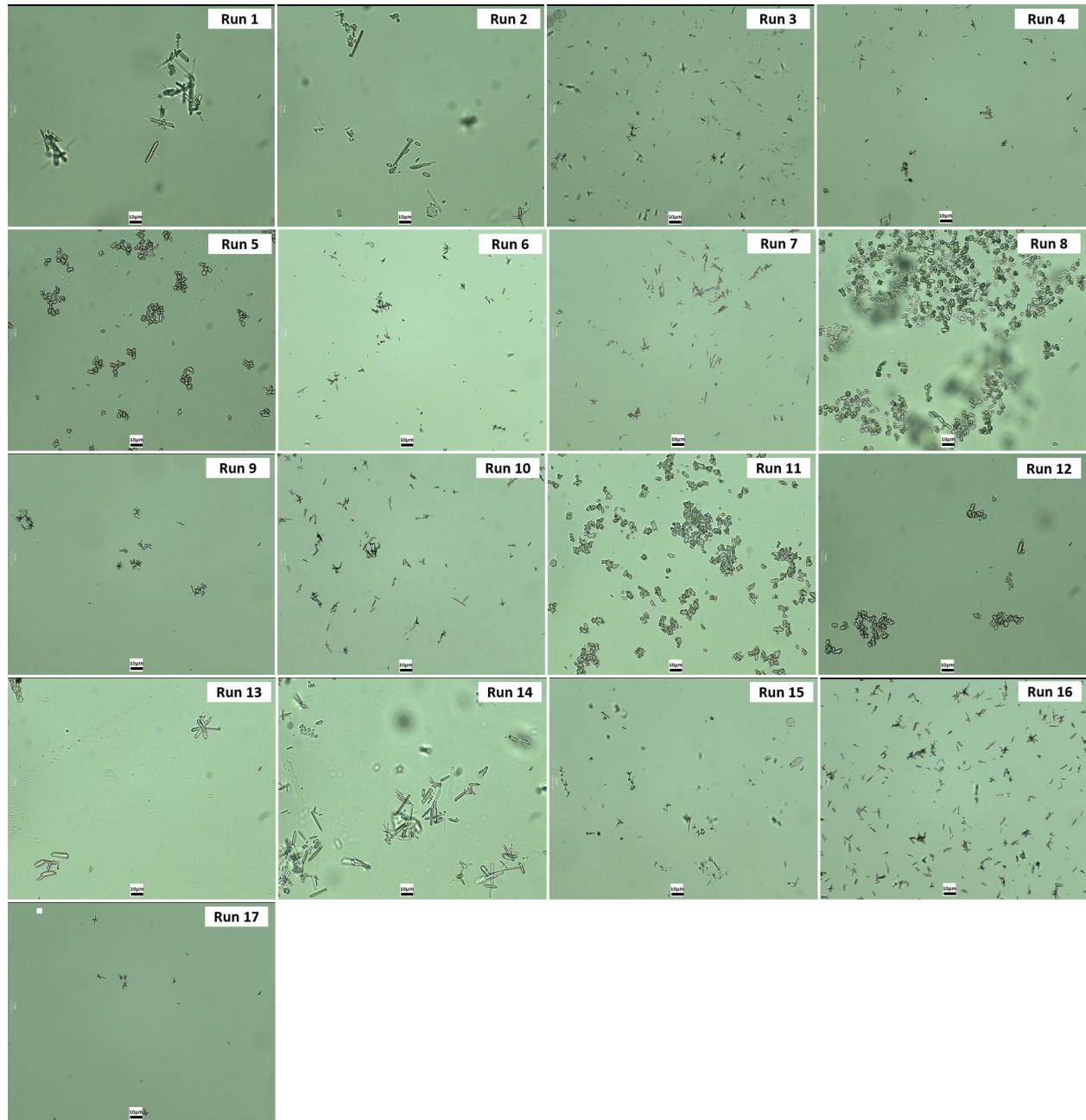


Table ST4. ANOVA for the response surface quadratic models for the COT and COM

Source	Sum of squares	Degree of freedom	Mean square	F-value	p-value
<i>COT (%)</i>					
<i>The recovery</i>					
Model	28213.42	6	4702.24	24.88	< 0.0001
Residual	1889.90	10	188.99		
Lack of fit	1630.70	6	271.78	4.19	0.0933
Pure error	259.20	4	64.80		
Total	30103.32	16			
R^2	0.9372				
<i>COM (%)</i>					
<i>The recovery</i>					
Model	28443.88	6	4740.65	26.20	< 0.0001
Residual	1809.20	8	0.018		
Lack of fit	1629.20	4	0.022	1.60	0.3299
Pure error	180.00	4	0.014		
Total	30253.08	12			
R^2	0.9402				

Table ST5. The regression coefficient of a polynomial function of all response surfaces

Term	Coefficients	Standard error	F-value	p-value
<i>COT</i>				
Intercept	96.4	6.15		
X_1	-49.85	4.86	105.19	< 0.0001
X_2	14.25	4.86	8.60	0.0150
X_3	-0.2500	4.86	0.0026	0.9600
X_1^2	-34.05	6.70	25.83	0.0005
X_2^2	-12.35	6.70	3.40	0.0951
X_3^2	-12.65	6.70	3.57	0.0883
<i>COM</i>				
Intercept	3.00	6.02		
X_1	49.97	4.76	110.44	< 0.0001
X_2	-14.25	4.76	8.98	0.0134
X_3	0.3750	4.76	0.0062	0.9387
X_1^2	34.22	6.56	27.26	0.0004
X_2^2	12.77	6.56	3.80	0.0799
X_3^2	12.83	6.56	3.83	0.0789
$Y = \beta_0 + \beta_1 X_1 + \beta_2 X_2 + \beta_3 X_3 + \beta_{11} X_1^2 + \beta_{22} X_2^2 + \beta_{33} X_3^2$				
X_1 : temperature; X_2 : pH X_3 : ionic strength *Significant at $p \leq 0.05$				

Figure SF2. One-factor graph; effect of each parameter including temperature, pH and ionic strength on the a) COT content and b) COM content

Figure SF2

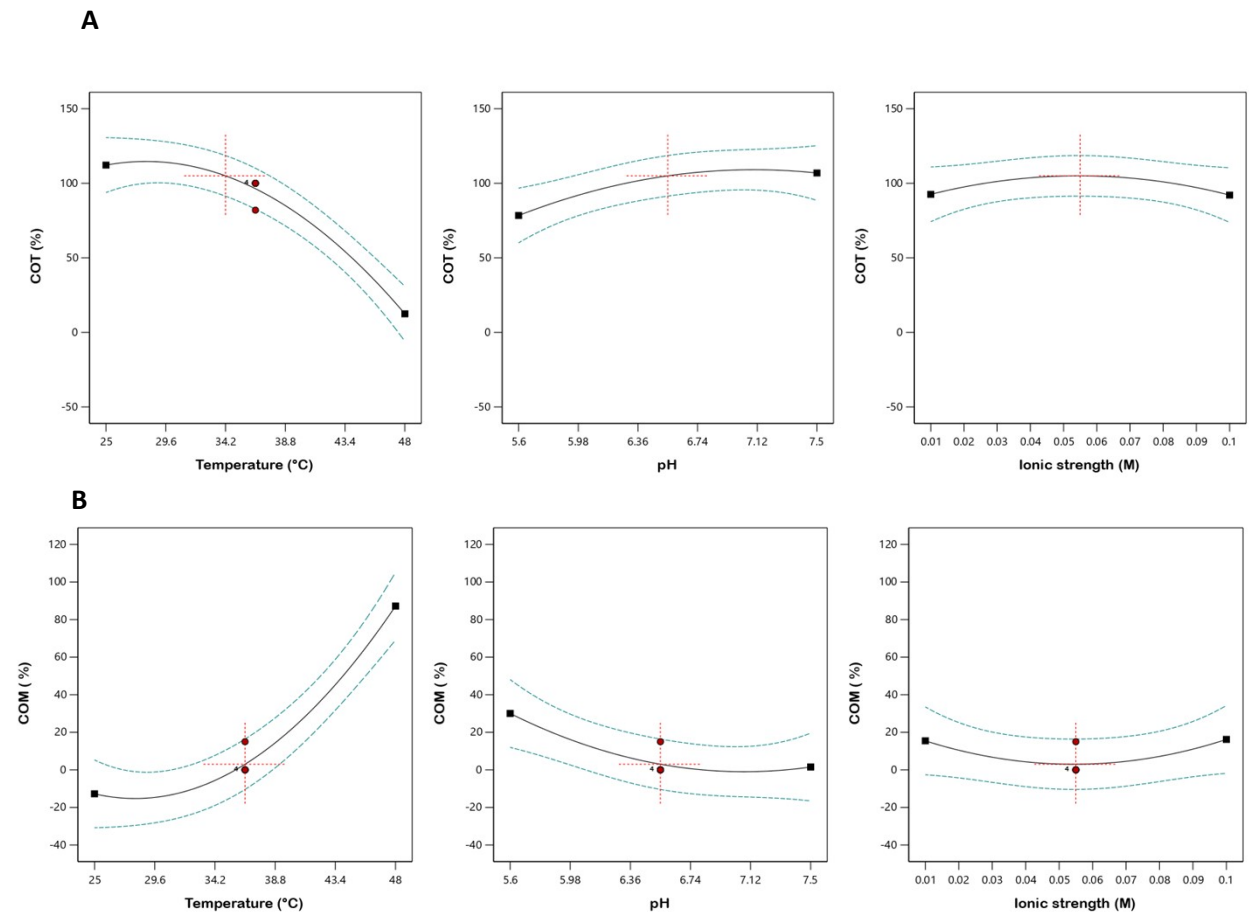


Figure SF3. XRD pattern and its Rietveld refinement of the optimized sample for COT. Rietveld output plot (Black line represents experimental plot, red line calculated plot, black points represent Bragg positions, while blue line represents difference plot).

Figure SF3

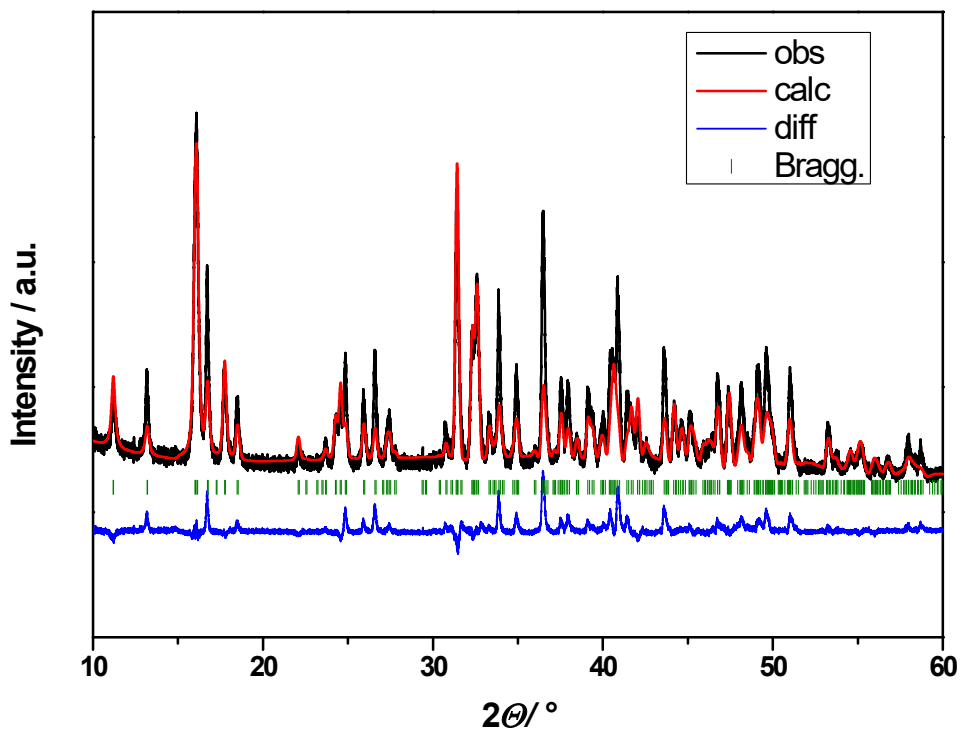


Table ST6. Crystallographic data obtained from Rietveld refinement for the synthesized optimized COT sample.

Phase composition (wt %)	100 %		
Unit cell parameters (Å)	$a = 6.1034(4)$	$b = 7.1566(6)$	$c = 8.4457(6)$
R_B (%)	14.27695		
R_p (%)	12.72697		
R_{wp} (%)	17.23697		
R_e (%)	5.84193		
GoF	8.70581		