



Supplementary Information

Optimization of the obtaining of cellulose nanocrystals from *Agave tequilana* Weber var. Azul by acid hydrolysis

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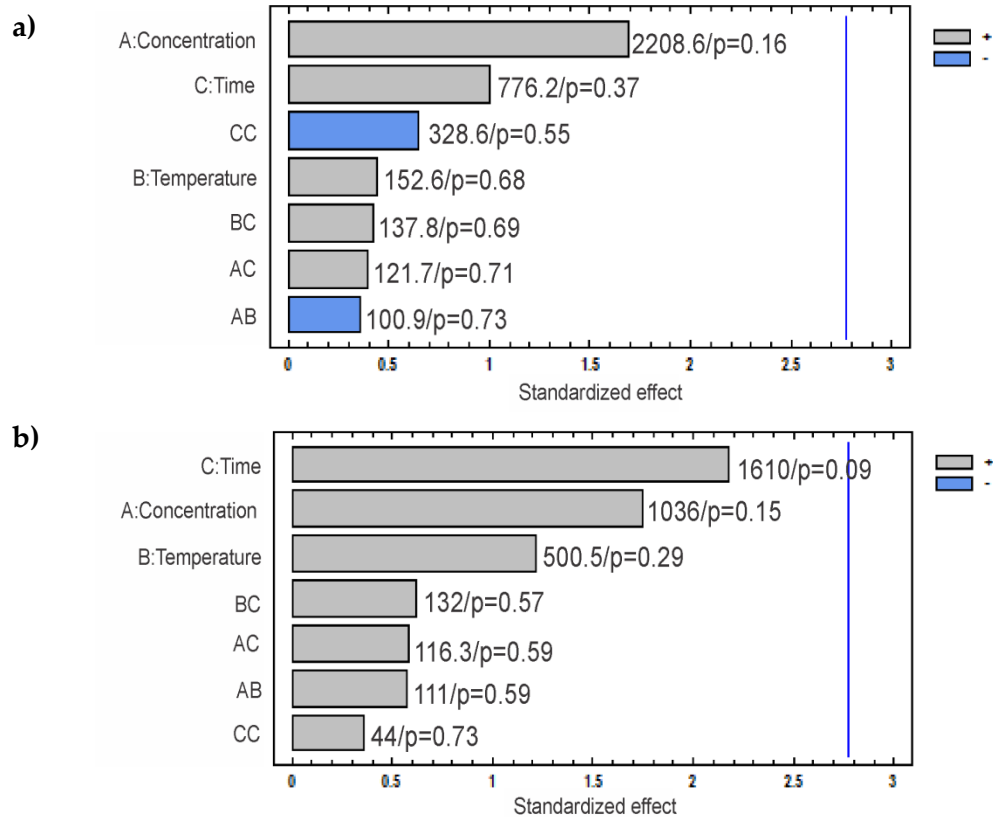


Figure S1. (a) Pareto standardized effect for CNC-H yield production; (b) Pareto standardized effect for CNC-S yield production. The sum of squares and p-value is shown at the right side of each bar.

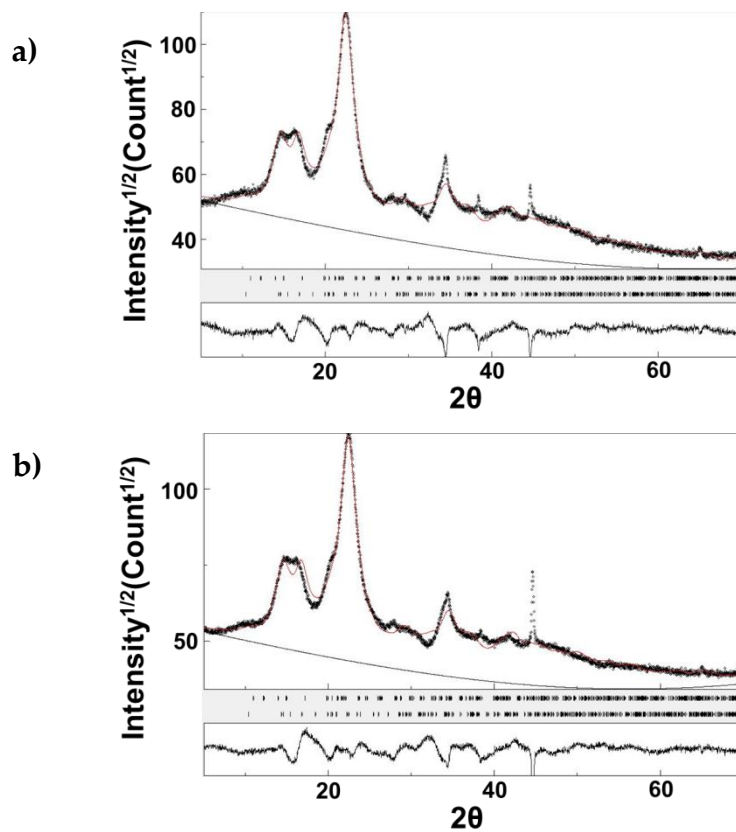


Figure S2. Rietveld analysis for (a) CNC-S (E3S), (b) CNC-H (E10H)

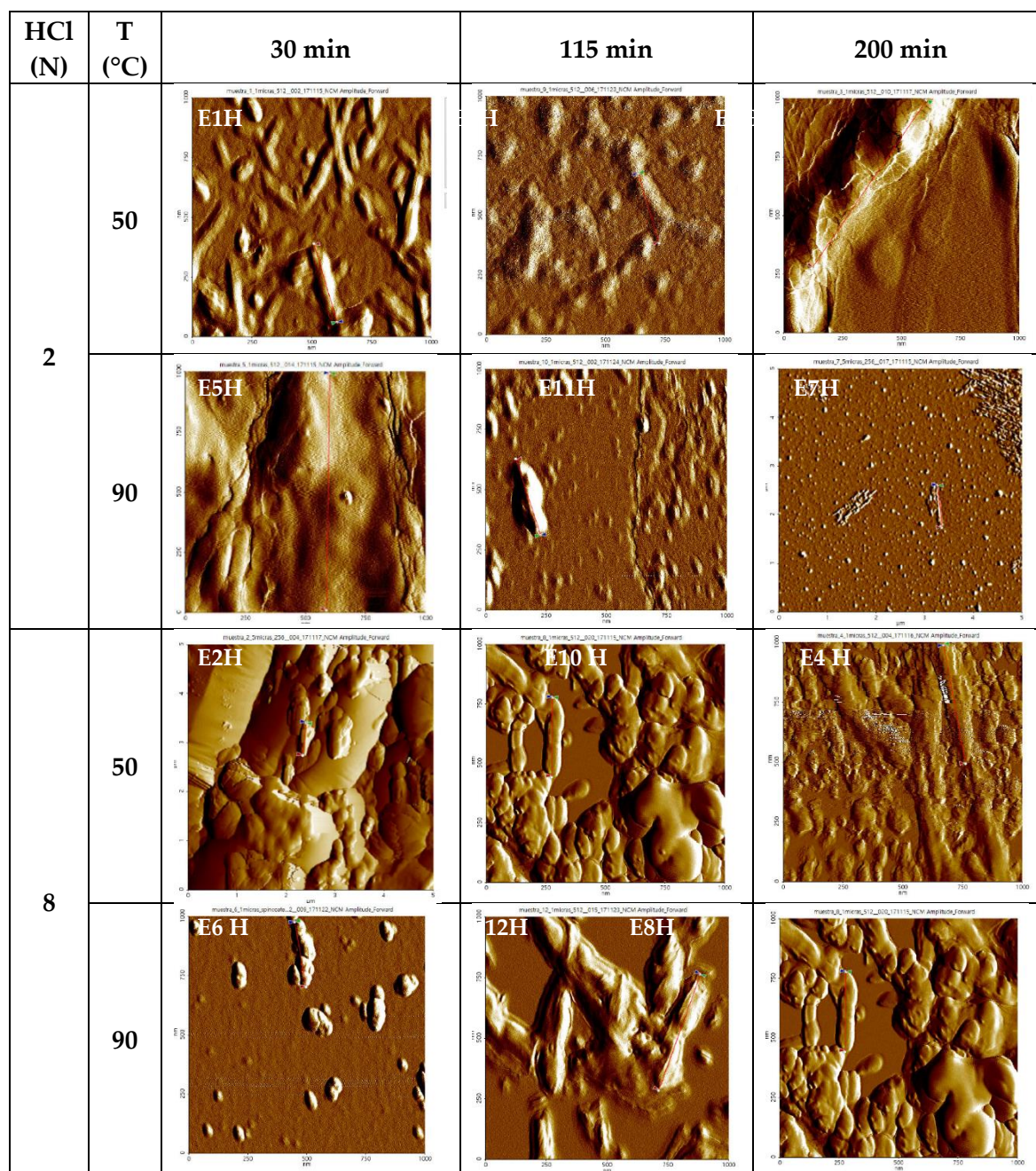


Figure S3. AFM 2D images of the 12 CNC-H samples

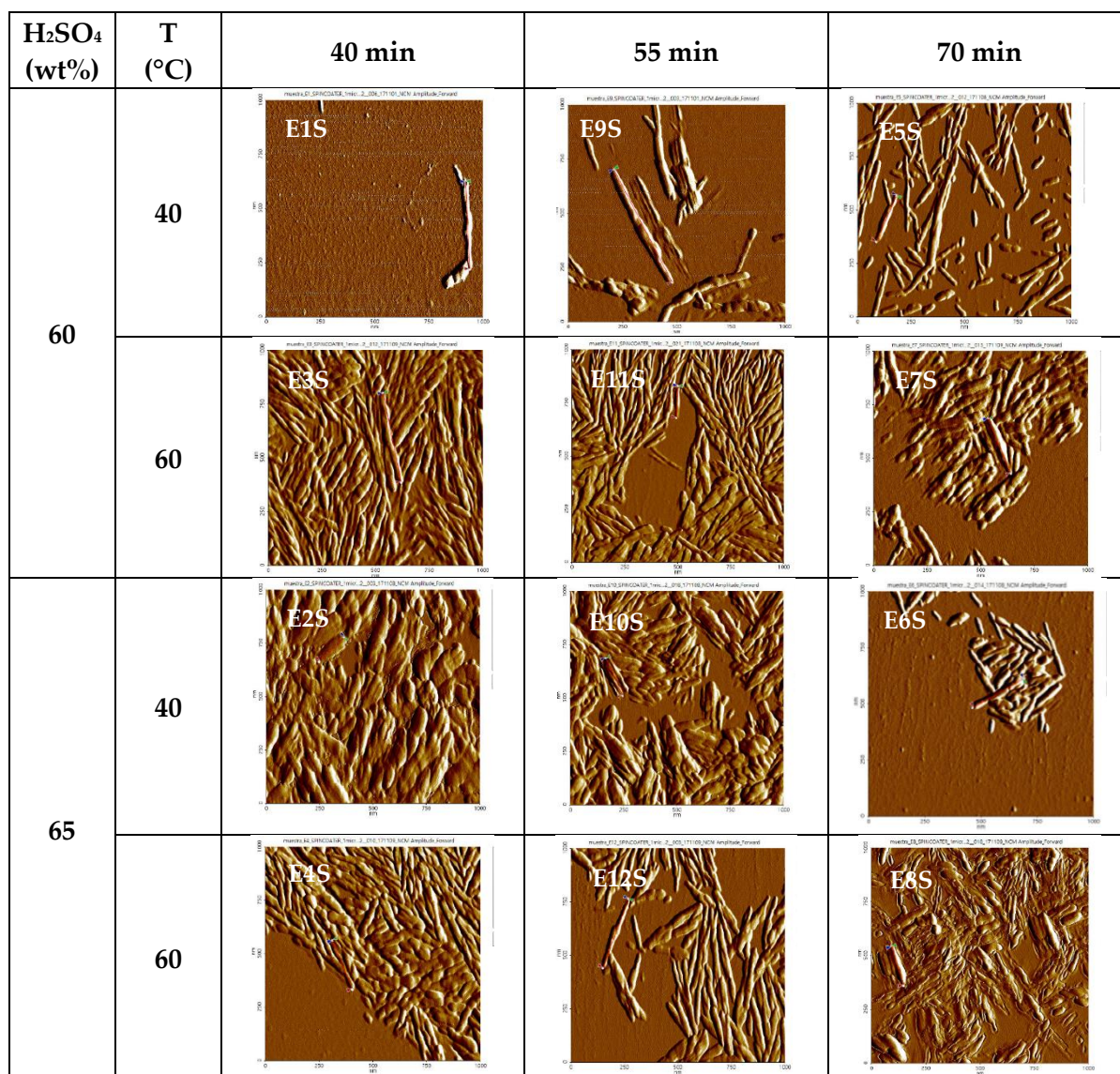


Figure S4. AFM 2D images of the 12 CNC-S samples

**Table S1.** DLS and yield results for HCl hydrolysis

Hydrolysis Conditions			Sample		DLS			
HCl (N)	T (°C)	t (min)	ID	Yield (%)	Size (d.nm)	PdI	Peak 1	
							Size (d.nm)	%
2	50	30	E1H	4.2	412±82	0.30±0.09	447±87	98±2
		115	E9H	34	462±56	0.32±0.07	499±59	98±2
		200	E3H	22	620±40	0.26±0.05	627±58	98±2
	90	30	E5H	30	606±48	0.27±0.04	738±169	99±1
		115	E11H	33	565±20	0.34±0.06	614±22	100
		200	E7H	36	619±65	0.45±0.07	526±92	88±2
8	50	30	E2H	32	794±245	0.50±0.22	617±231	100
		115	E10H	90	482±50	0.45±0.04	376±62	99±1
		200	E4H	37	402±66	0.34±0.05	437±69	97±2
	90	30	E6H	40	387±87	0.26±0.11	373±58	99±1
		115	E12H	33	323±18	0.24±0.06	322±34	99±1
		200	E8H	90	329±6	0.19±0.05	355±18	100

Table S2. DLS and yield results for H₂SO₄ hydrolysis conditions

Hydrolysis conditions			Sample	Results							
H ₂ SO ₄ (wt%)	T (°C)	t (min)	ID	Yield	[AG]	DLS					
				Yield (%)	$\frac{mmol}{Kg}$	Size (d.nm)	PdI	Peak 1		Peak 2	
								Size (d.nm)	%	Size (d.nm)	%
60	40	40	E1S	4.5	10	366 ± 20	0.31 ± 0.06	379 ± 201	97 ± 2	-	-
		55	E9S	34	15	343 ± 37	0.45 ± 0.12	386 ± 39	85 ± 7	50 ± 2	6 ± 1.5
		70	E5S	32	35	210 ± 22	0.50 ± 0.04	470 ± 110	75 ± 12	73 ± 24	24 ± 13
	60	40	E3S	23	15	206 ± 140	0.74 ± 0.14	260 ± 126	79 ± 11	38 ± 32	18 ± 10
		55	E11S	31	31	362 ± 20	0.85 ± 0.21	552 ± 41	63 ± 8	93 ± 22	12 ± 0.2
		70	E7S	37	69	435 ± 53	0.70 ± 0.19	473 ± 60	87 ± 4	75 ± 22	11 ± 3
65	40	40	E2S	29	21	117 ± 3	0.36 ± 0.11	177 ± 69	93 ± 5	-	-
		55	E10S	37	72	111 ± 6	0.58 ± 0.05	356 ± 125	58 ± 6	52 ± 10	38 ± 10
		70	E6S	42	162	125 ± 5	0.61 ± 0.12	306 ± 141	68 ± 11	48 ± 17	28 ± 10
	60	40	E4S	37	38	158 ± 40	0.79 ± 0.16	370 ± 109	78 ± 7	47 ± 16	14 ± 5
		55	E12S	32	80	222 ± 76	0.68 ± 0.18	326 ± 76	78 ± 7	40 ± 22	12 ± 4
		70	E8S	96	166	222 ± 13	0.69 ± 0.24	329 ± 25	87 ± 8	33 ± 9	10 ± 1.5

**Table S3.** Total sulfate groups

Experiment	Total Sulfate Groups (mmol/Kg)
E1S	10.34
E2S	54.77
E3S	20.73
E4S	38.18
E5S	35.32
E6S	68.85
E7S	162.01
E8S	166
E9S	15.45
E10S	31.16
E11S	71.74
E12S	79.51

Table S4. AFM measurements of CNC-H

Hydrolysis conditions			Sample	AFM					
HCl (N)	T (°C)	t (min)	ID	Dimensions			Aspect ratio		
				L (nm)	H (nm)	W (nm)	L/H	L/W	W/H
2	50	30	E1H	216 ± 73	8.9	69 ± 17	24 ± 8	3.6 ± 1.9	7.8 ± 1.9
		115	E9H	302 ± 109	9.1	77 ± 16	33 ± 12	85 ± 3	8.5 ± 2.9
		200	E3H	867	8.8	-	98	-	-
	90	30	E5H	520 ± 225	8.7	65 ± 7	60 ± 26	8.5 ± 4.4	7.5 ± 0.8
		115	E11H	490 ± 82	9.1	109 ± 24	54 ± 9	4.9 ± 1.8	12 ± 2.6
		200	E7H	828 ± 193	8.7	68 ± 17	95 ± 22	13.7 ± 6.3	7.8 ± 2.6
8	50	30	E2H	829 ± 282	9.1	253 ± 70	91 ± 31	3.9 ± 2.2	28 ± 8
		115	E10H	324	9.1	103 ± 14	36	3.2 ± 0.4	11 ± 1.5
		200	E4H	292 ± 147	9.0	87 ± 24	32 ± 16	4.1 ± 2.8	9.7 ± 2.7
	90	30	E6H	277 ± 72	8.9	58 ± 11	31 ± 8	5.2 ± 2.2	6.5 ± 1.2
		115	E12H	350 ± 133	9.1	103 ± 46	38 ± 14	5 ± 3.5	11 ± 5.1
		200	E8H	266 ± 107	8.6	88 ± 15	31 ± 12	3.3 ± 1.8	10 ± 1.7

**Table S5.** AFM measurements of CNC-S

Hydrolysis conditions			Sample	AFM					
H ₂ SO ₄] (wt%)	T (°C)	t (min)	ID	Dimensions			Aspect ratio		
				L (nm)	H (nm)	W (nm)	L/H	L/W	W/H
60	40	40	E1S	404	9.3	37 ± 5	43	11 ± 1.5	4 ± 0.5
		55	E9S	370 ± 146	9.1	36 ± 6	41 ± 16	13 ± 4.2	4 ± 0.7
		70	E5S	209 ± 116	9.0	31 ± 9	23 ± 13	10 ± 4.8	3.5 ± 1
	60	40	E3S	240 ± 31	9.0	38 ± 10	27 ± 3	7.3 ± 2.3	4.2 ± 1.1
		55	E11S	185 ± 59	9.0	54 ± 13 25 ± 5	21 ± 6	4.3 ± 1.6 9.2 ± 3	6 ± 1.4 2.8 ± 0.6
		70	E7S	221 ± 76	8.9	40 ± 12	25 ± 8	7.4 ± 3.2	4.5 ± 1.3
65	40	40	E2S	214 ± 85	8.9	73 ± 11	24 ± 9	3.7 ± 3.1	8.2 ± 1.2
		55	E10S	158 ± 70	8.7	49 ± 18	18 ± 8	4.9 ± 2.5	5.6 ± 2.1
		70	E6S	137 ± 68	9.1	33 ± 7	26 ± 7	8.8 ± 2.9	3.6 ± 0.8
	60	40	E4S	169 ± 60	8.7	44 ± 12	19 ± 7	5.1 ± 2.1	5.1 ± 1.4
		55	E12S	244 ± 113	8.7	35 ± 6	28 ± 13	9.1 ± 3.2	4 ± 0.7
		70	E8S	149 ± 59	9.2	39 ± 12	16 ± 6	5.3 ± 2.4	4.2 ± 1.3