

Supplementary Information file for

The effect of sulfate half-ester groups on cellulose nanocrystal periodate oxidation

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Nuclear Magnetic Resonance Spectroscopy

Table S1. Results of spectral fitting for the C1, C4 and oxidized compounds region of CP/MAS ¹³C NMR spectrum of cellulose (cf. Figure 5).

	Assignment	d (ppm)	FWHM (Hz)
C1	Crystalline	108.4	243
	Crystalline	106.1	248
	Crystalline	105.3	175
	Crystalline	104.8	316
	Crystalline	104.2	177
	Non-Crystalline	102.1	255
	Cellulose oligomer	100.5	255
	Oxidized compounds	103.1	323
	Oxidized compounds	99.8	273
	Oxidized compounds	97.6	307
C4	Oxidized compounds	94.9	321
	Oxidized compounds	91.0	429
	Crystalline	89.2	91
	Crystalline	88.7	87
	Crystalline	88.2	316
	Crystalline	87.9	122
	Amorphous	84.2	209
	Amorphous	83.4	464
	Amorphous	82.7	159
	Cellulose oligomer	80.9	198
Cellulose oligomer	79.3	174	

Elemental analysis

The sulfate content of the CNCs was higher previous to oxidation than the sodium content indicating that not all sulfate half-ester groups were in Na⁺ form. The Na/S ratio increased with oxidation indicating that the use of the sodium periodate facilitated a cation exchange of the remaining sulfate groups. However, it could also be a result of decreasing sulfate group content or that the oxidation results in other functionalities than aldehydes only.

Table S2. Elemental analysis of CNCs with various degree of oxidation (DO) expressed as wt%. The standard deviation was less than 0.02 in all cases.

Derivate	S	H	O	Na	Na/S	O/S
CNC	1.09	5.60	52.20	0.39	0.35	48.11
DAC DO 11%	0.71	5.92				
DAC DO 27%	0.69	5.85				
DAC DO 52%	0.66	5.65	54.78	0.51	0.77	82.99
desulfCNC	0.29	6.04	52.42			183.93
desulfDAC DO 11%	0.30	5.85				
desulfDAC DO 32%	0.26	5.60				
desulfDAC DO 48%	0.24	5.79	53.84			229.09

Table S3. Bonferroni-Holm test comparing the dimensions of the CNC derivatives. Each row represents a comparison between derivate 1 and derivate 2. The p-value and the corrected alpha (rejection criteria) for each individual hypotheses to avoid family-wise error are included. If the p-value is smaller than the corrected alpha the difference is statistically significant.

Derivate 1	Derivate 2	p-value	Alpha	Difference
CNC	DAC	2.79E-10	0.01	YES
CNC	desulfCNC	0.42	0.05	NO
desulfCNC	desulfDAC	0.02	0.02	NO
DAC	desulfDAC	1.07E-11	0.01	YES
CNC	desulfDAC	0.10	0.03	NO
DAC	desulfCNC	1.55E-8	0.01	YES