SUPPORTING INFORMATION

Influence of Reaction Variables on the Surface Chemistry of Cellulose Nanofibers Derived from Palm Oil Empty Fruit Bunches

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Contents

Table S1. Multilevel Factorial Design 2^4

No. Experiments	Identification samples	Experimental parameters	
		Reaction time [min]	Cellulose:NaClO molar ratio
1	TOCN 30-1:1	30	1:1
2	TOCN 30-1:5	30	1:5
3	TOCN 30-1:8	30	1:8
4	TOCN 30-1:10	30	1:10
5	TOCN 60-1:1	60	1:1
6	TOCN 60-1:5	60	1:5
7	TOCN 60-1:8	60	1:8
8	TOCN 60-1:10	60	1:10
9	TOCN 90-1:1	90	1:1
10	TOCN 90-1:5	90	1:5
11	TOCN 90-1:8	90	1:8
12	TOCN 90-1:10	90	1:10
13	TOCN 120-1:1	120	1:1
14	TOCN 120-1:5	120	1:5
15	TOCN 120-1:8	120	1:8
16	TOCN 120-1:10	120	1:10

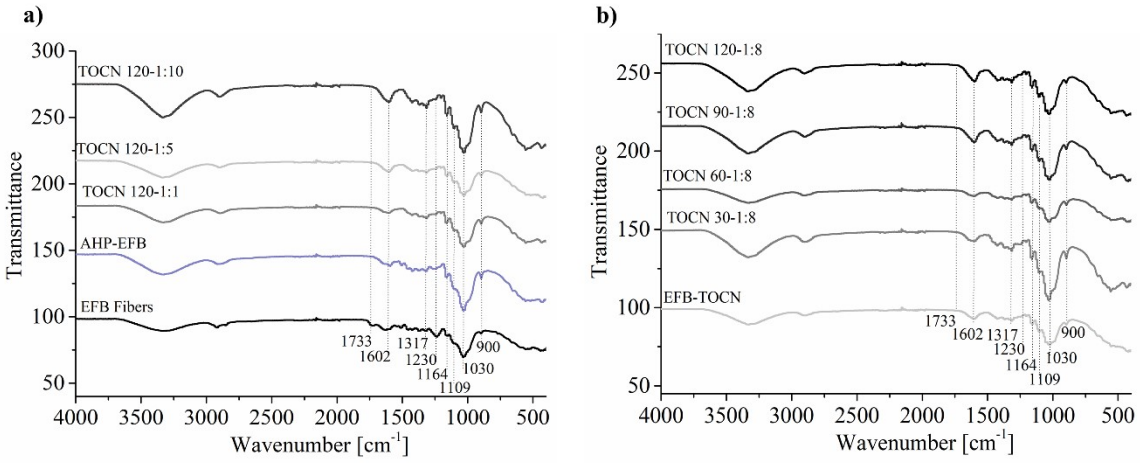

Figure S1. FTIR-ATR spectra of TOCN isolated from Palm Oil Empty Fruit Bunches (EFB). a) Effect of changes in cellulose:NaClO ratios for a reaction time of 120 min for AHP-EFB-TOCN, and b) Effect of changes in reaction time for AHP-EFB-TOCN.

Table S2. Signal assignment infrared spectroscopy

Wave number [cm^{-1}]	Functional group	Reference
1733	C=O stretch of acetyl and hemicellulose	1
1602	Stretching C=O in carboxylic acids with sodium	2,3
1317	Stretch flexion CH_2 in cellulose	
1230	Asymmetric axial deformation =C-O- in the ether, ester, and phenol group of lignin	4
1164	Asymmetrical C-O-C stretch of the pyranose ring	5
1109	C-O-C stretch of cellulose	4
1030	Cellulose C-O and C-H stretching vibrations	
900	specific C-H anomeric vibration for β-glycosides	5,6

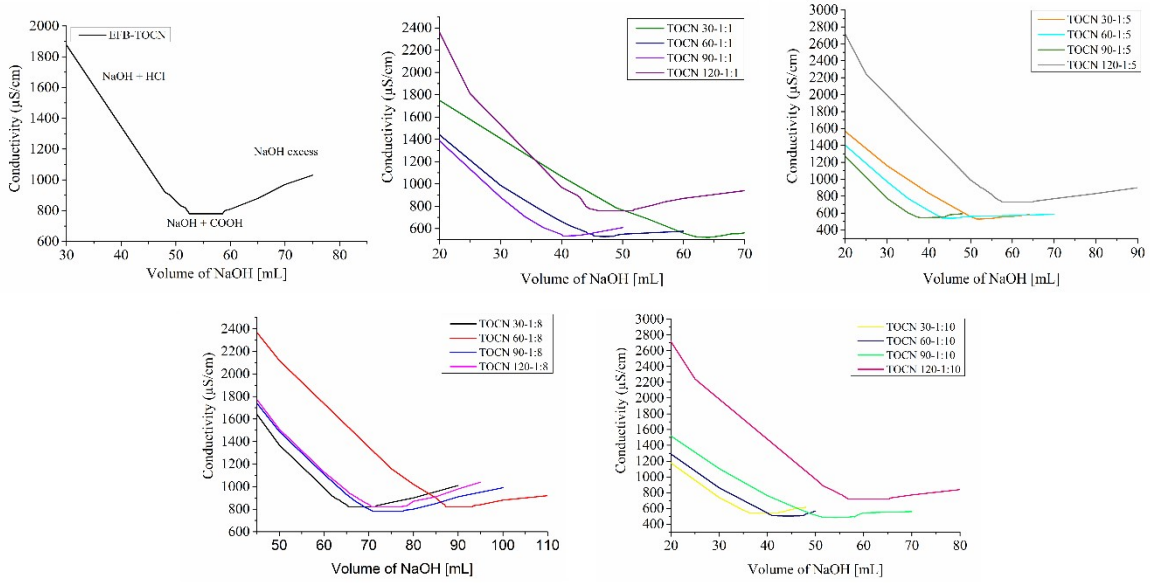

Figure S2. Conductimetric titration graphs of oxidized cellulose suspensions

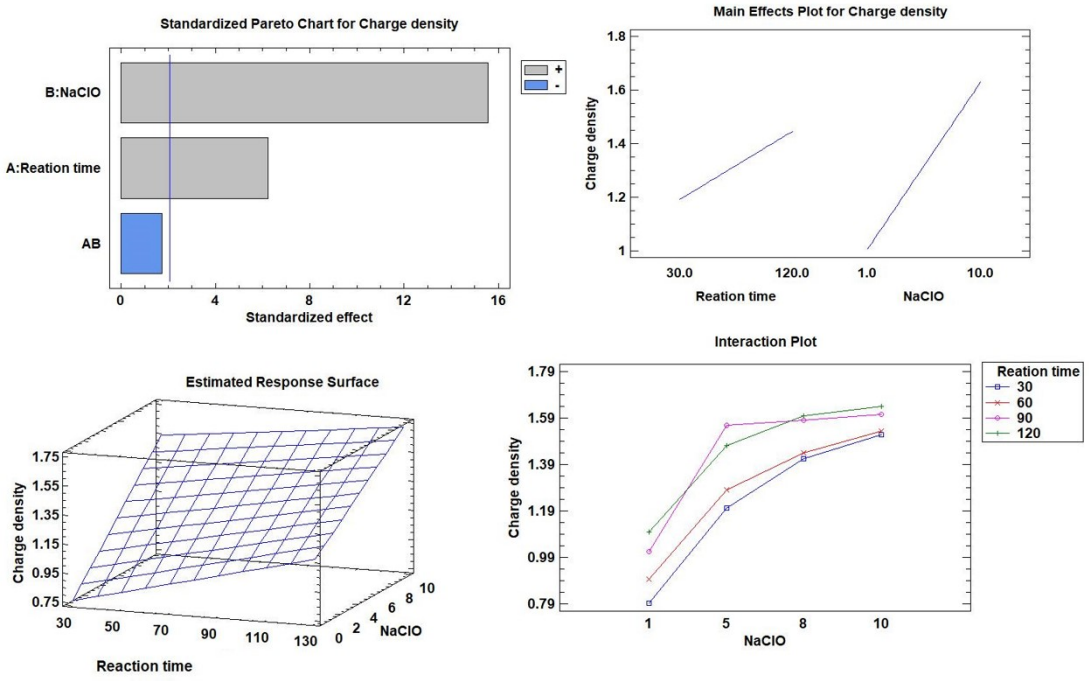

Figure S3. Statistical analysis of charge density a) Pareto chart, b) Main effects plot, c) Estimated response surface, and d) Interaction plot.

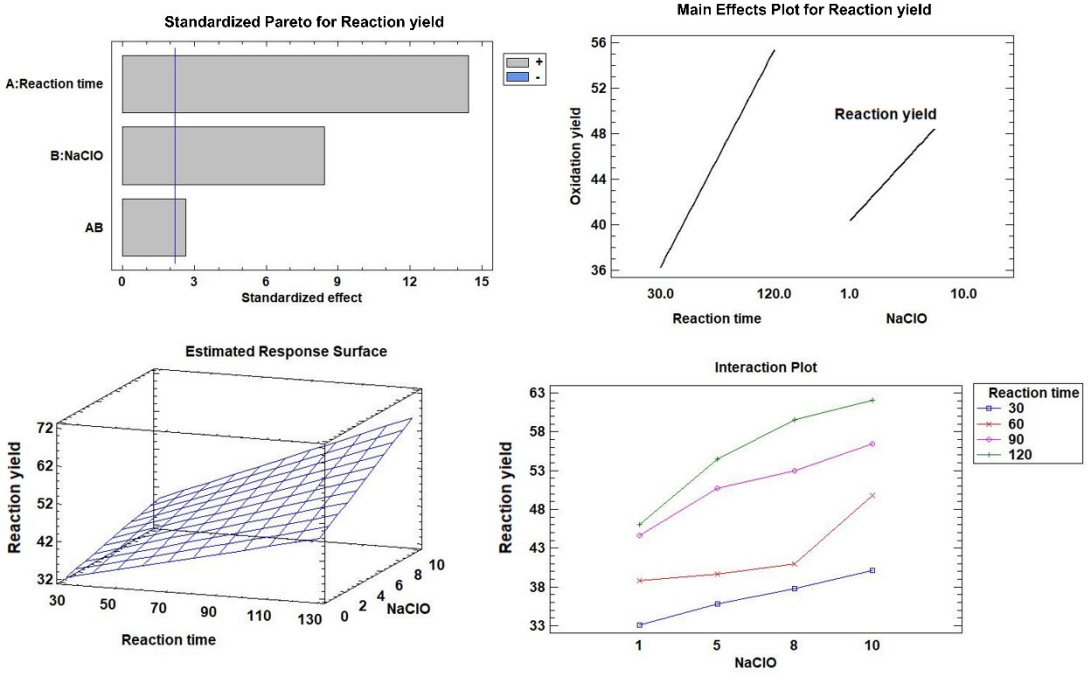

Figure S4. Statistical analysis of reaction yield [%] a) Pareto chart, b) Main effects plot, c) Estimated response surface, and d) Interaction plot.

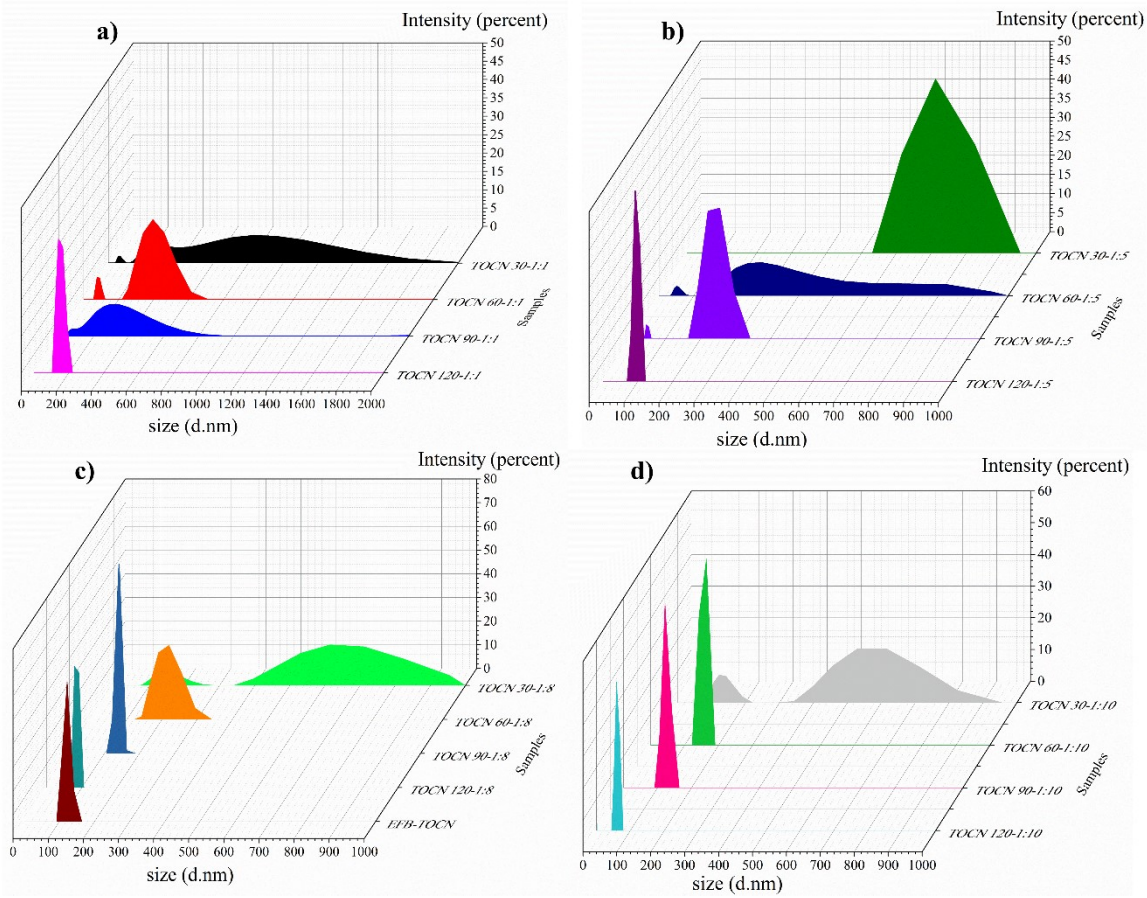

Figure S5. Effect of Time reaction on size distribution of AHP-EFB-TOCN a) 1:1, b) 1:5, c) 1:8, and d) 1:10 cellulose:NaClO ratio.

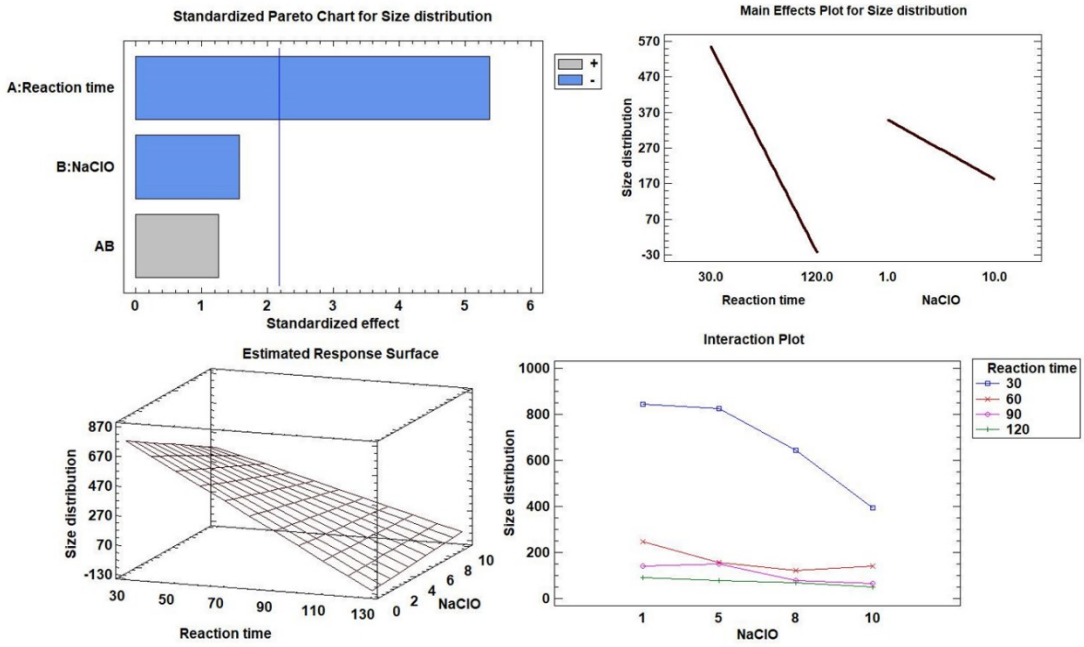

Figure S6. Statistical analysis of size distribution a) Pareto chart, b) Main effects plot, c) Estimated response surface, and d) Interaction plot.

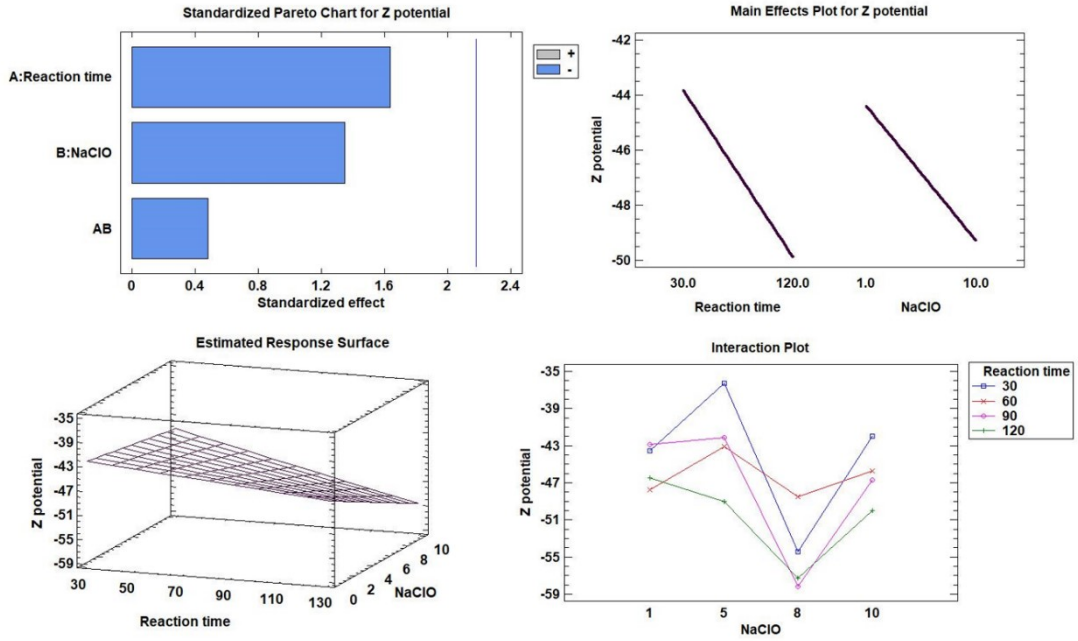

Figure S7. Statistical analysis of Z potential a) Pareto chart, b) Main effects plot, c) Estimated response surface, and d) Interaction plot.

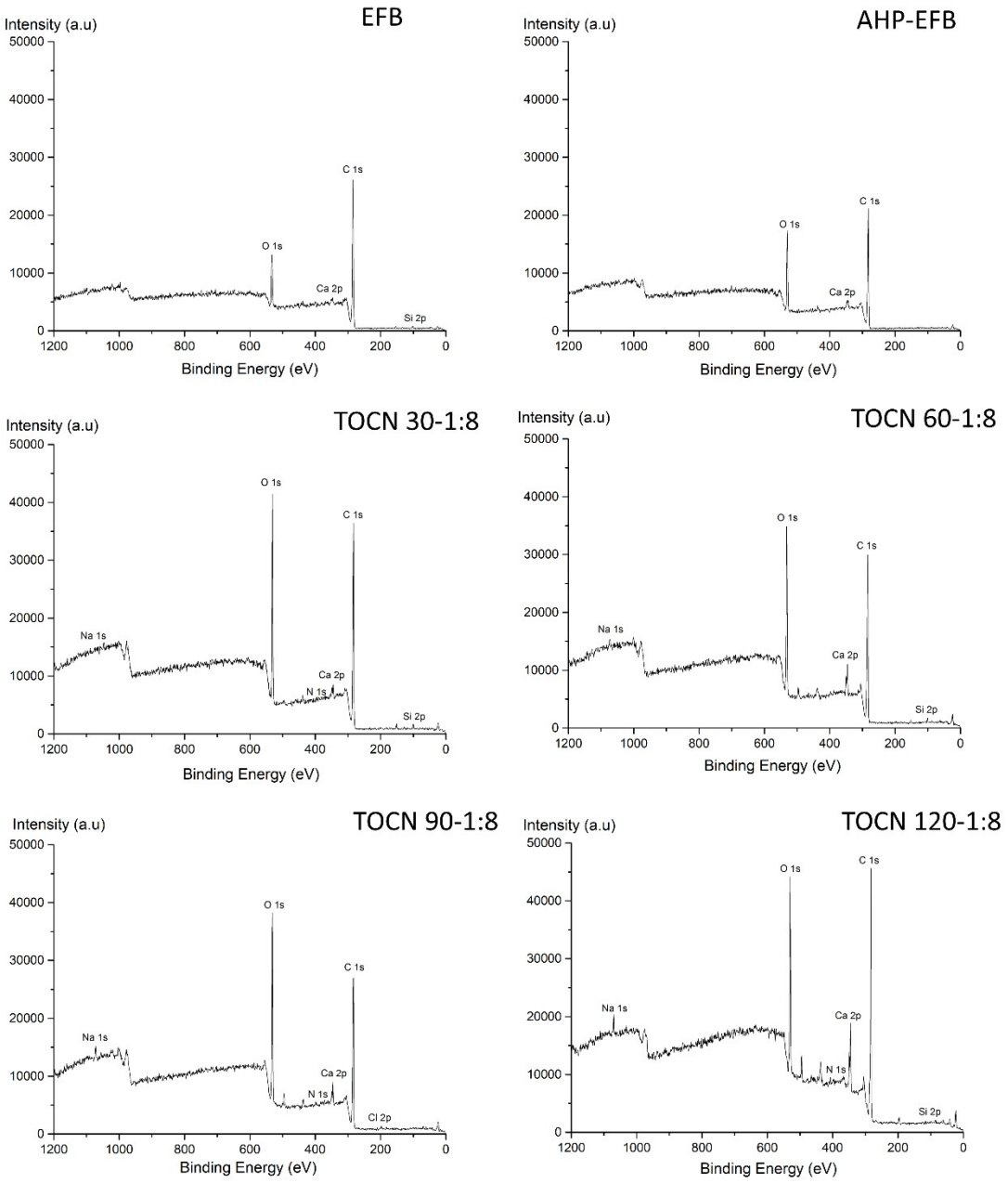

Figure S8. Spectra XPS a) (EFB, EFB-AHP, TOCN 30-1:8, TOCN 60-1:8, TOCN 90-1:8 and TOCN 120-1:8) and b)

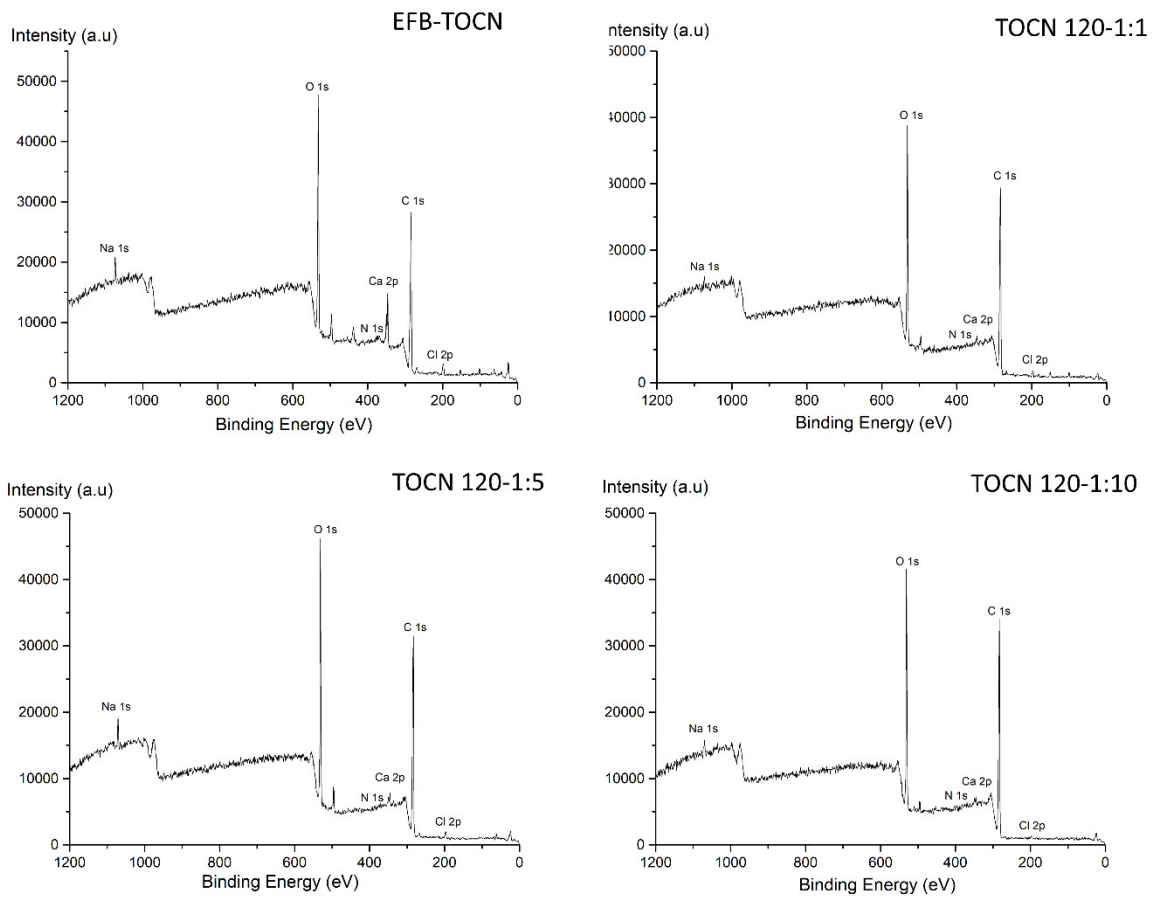

Figure S9. Spectra XPS b. Spectra XPS (EBF-TOCN, TOCN 120-1:1, TOCN 120-1:5, and TOCN 120-1:10)

Table S3. General spectra and the positions and values of FWHM

Atom		XPS data	EFB	EFB-AHP	EFB-TOCN	TOCN 30-1:8	TOCN 60-1:8	TOCN 90-1:8	TOCN 120-1:8	TOCN 120-1:1	TOCN 120-1:5	TOCN 120-1:10
C	C-H	Position [eV]	284.8	284.8	284.8	284.8	284.8	284.8	284.8	284.8	284.8	284.8
		FWHM	2.37	1.91	1.28	1.30	1.35	1.26	1.20	1.30	1.14	1.21
	C-C-O	Position [eV]	-	286.8	286.5	286.5	286.5	286.5	286.4	286.5	286.5	286.4
		FWHM	-	1.91	1.28	1.30	1.35	1.26	1.20	1.30	1.14	1.21
	O-C-O	Position [eV]	287.9	288.6	288.2	287.9	288.1	288.0	288.3	287.9	287.9	287.7
		FWHM	2.37	1.55	1.28	1.30	1.35	1.26	1.20	1.30	1.14	1.21
	C-COO-	Position [eV]	-	-	289.5	288.8	289.4	289.3	289.5	289.0	288.7	288.6
		FWHM	-	-	1.28	1.30	1.35	1.26	1.20	1.30	1.14	1.21
	O	Position [eV]	532.5	532.6	532.3	532.9	532.7	532.8	531.9	533.0	532.9	532.8
		FWHM	2.90	2.52	2.43	1.99	2.4	2.17	2.15	1.88	1.81	1.90
Ca	3/2	Position [eV]	347.5	347.2	347.3	347.5	347.3	347.3	347.3	347.4	347.39	347.3
		FWHM	2.32	1.94	1.48	1.48	1.57	1.54	1.44	1.5	1.46	1.77
	1/2	Position [eV]	351.1	350.9	350.9	351.1	350.9	350.9	350.9	351.0	351.0	350.9
		FWHM	2.20	1.95	1.56	1.59	1.56	1.55	1.52	1.50	1.50	1.83
	Si	Position [eV]	101.9	-	-	101.9	101.9	-	101.9	-	-	-
		FWHM	1.94	-	-	1.36	1.42	-	0.98	-	-	-
	Na	Position [eV]	-	-	1073.9	1072.9	1073.8	1073.7	1072.2	1074.3	1072.6	1072.1
		FWHM	-	-	1.83	2.18	2.74	2.39	2.25	1.63	1.68	2.22
Cl	3/2	Position [eV]	-	-	198.4	-	-	198.8	-	197.8	198.2	198.2
		FWHM	-	-	1.87	-	-	2.49	-	1.56	2.31	2.12
	1/2	Position [eV]	-	-	200.02	-	-	200.4	-	199.4	199.8	199.8
		FWHM	-	-	1.28	-	-	1.84	-	1.43	2.09	0.98
	N	Position [eV]	-	-	398.7	399.9	-	398.7	398.3	400.0	399.8	399.9
		FWHM	-	-	0.78	1.50	-	0.50	0.1	2.34	2.50	1.83

Parity Plot

The parity plot was performed with the experimental data obtained in the C 1S spectra, focusing on the atomic percentages obtained for the signals C-C-O, O-C-O/C= O and C-COO-. The contributions were then summed and divided by the corresponding ratio for AHP-EFB 5/6 and 1/6 for C-C-O and O-C-O and for oxidised cellulose TOCN 4/6, 1/6 and 1/6 for C-C-O, O-C-O/C= O and C-COO- respectively. Table S4 presents the data obtained for EFB-AHP and EFB-TOCN.

Table S4. Theorical value parity plot

	C 1s	Experimental	Ratio	Operation	Theoretical
EFB-AHP	C-C-O	16.84	5/6	(20.74/(5/6)	17.28
	O-C-O	3.90	1/6	(20.74)/(1/6)	3.46
	C-COO-	0.00	-	-	-
	Total Carbon	20.74	-	-	-
EFB-TOCN	C-C-O	18.15	4/6	(28.63) /(4/6)	19.09
	O-C-O	6.60	1/6	(28.63) /(1/6)	4.77
	C-COO-	3.88	1/6	(28.63) /(1/6)	4.77
	Total Carbon	28.63	-	-	-

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