Electronic Supplementary Material (ESI) for Green Chemistry.

Sonochemical production of nanoscaled crystalline cellulose using organic acids

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The present information corresponds to the supplementary material of the article with the same title. In this file, more information regarding the Fourier Transform Infrared Analysis, Atomic Force Microscopy, Thermogravimetric Analysis, as well as crystallinity analysis, are displayed. The purpose of the current material is to contribute to the better elucidation of the article mentioned above.

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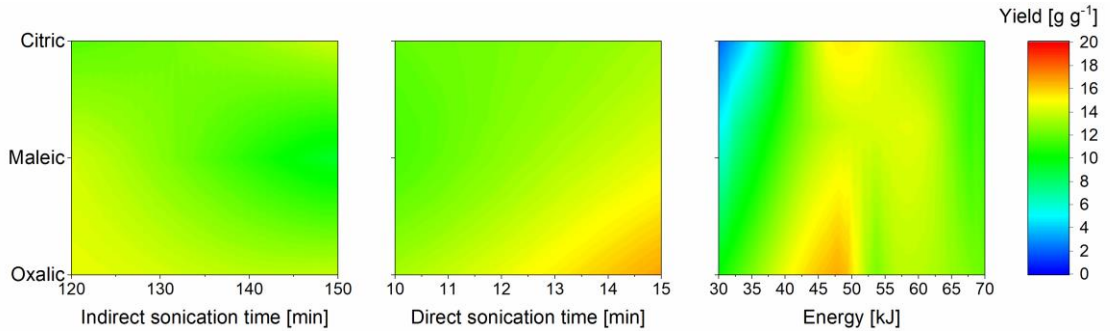

Figure S1. Surface response of yields to direct and indirect sonication, as well as to the accumulated energy delivered to the system.

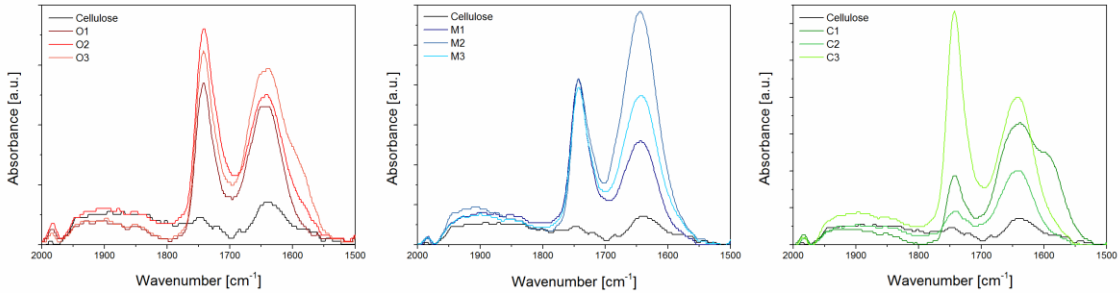

Figure S2. FTIR absorbance in the 2000-1500 cm^{-1} region.

Table S1. Assignment and the bands position in the 3700 – 2800 cm^{-1} region for all the cellulose samples[1–4]

Assignment	Cellulose	O1	O2	O3	M1	M2	M3	C1	C2	C3
O(2)H...O(6) intramolecular hydrogen bonds stretching vibration	3419	3416	3415	3417	3417	3417	3417	3415	3415	3415
O(3)H···O(5) intramolecular stretching vibration	3342	3343	3343	3343	3344	3344	3344	3344	3344	3346
O(6)H···O(3) intermolecular in cellulose I_β stretching vibration	3271	3278	3276	3276	3276	3276	3278	3279	3279	3274
O(6)H···O(3) intermolecular in cellulose I_α stretching vibration	3213	3222	3222	3222	3221	3221	3221	3221	3221	3221
asymmetric CH_3 stretching vibration	2963	2966	2967	2967	2967	2967	2967	2967	2967	2969
asymmetric CH_2 stretching vibration	2944	2943	2942	2942	2940	2940	2940	2943	2944	2944
symmetric CH_3 stretching vibration	2903	2905	2908	2905	2905	2920	2908	2904	2904	2907
symmetric CH_2 stretching vibration	2860	2859	2854	2856	2856	2852	2856	2858	2858	2853

Table S2. Assignment and the bands position in the 1850 – 700 cm^{-1} region for all the cellulose samples [1–4]

Assignment	Cellulose	O1	O2	O3	M1	M2	M3	C1	C2	C3
C=O anhydride stretching vibration		1806	1806	1808	1804	1808	1811	1810	1801	1801
C=O bond in carbonyl, ester and acetyl groups stretching vibration		1736	1739	1746	1744	1740	1738	1735	1738	1742
C=O in carboxylic acids stretching vibration		1673	1687	1682	1682	1683	1682	1681	1683	1882
Water molecules stretching vibration	1639	1638	1629	1643	1638	1635	1638	1635	163	1640
carboxylate ($–COO^-$) asymmetrical stretching vibration		1563	1573	1568	1566	1569	1567	1564	1567	1562
C–H deformation vibration	1464	1461	1459	1461	1461	1492	1461	1460	1461	1464
C–H deformation vibration	1431	1429	1428	1424	1427	1430	1427	1428	1428	1427
C–H deformation vibration	1374	1372	1372	1371	1372	1378	1372	1371	1371	1370
C–H stretching vibration	1318	1318	1318	1317	1317	1319	1317	1317	1317	1315
C–O stretching vibration	1277	1277	1277	1278	1278	1268	1278	1276	1278	1278
C–O–C stretching mode of the pyranose ring	1243	1241	1242	1242	1242	1225	1242	1241	241	1241
C–O–C stretching mode of the pyranose ring	1205	1205	1206	1206	1205	1208	1205	1205	1205	1205
C–O–C stretching vibration in cellulose C-O from esters	1168	1163	1163	1164	1163	1164	1163	1163	1163	1162
glucose ring stretching vibration	1118	1114	1114	1115	1114	1118	1114	1113	1113	1114
C–O stretching mainly from C(3)–O(3)H	1062	1062	1063	1063	1063	1063	1063	1063	1063	1063
C–O stretching ring	1025	1027	1027	1026	1028	1032	1028	1028	1028	1026
C–O stretching vibration	982	991	991	991	990	990	991	991	991	991
β-glucosidic linkage between the sugar units	939	930	928	930	930	930	930	929	929	932
β-glucosidic linkage between the sugar units in hemicelluloses and celluloses	893	897	899	895	896	907	896	897	897	893
O-H out of plane bending in cellulose I_α	753	760	760	760	760	757	760	759	759	759
O-H out of plane bending in cellulose I_β	715	713	713	713	712	712	712	712	712	712

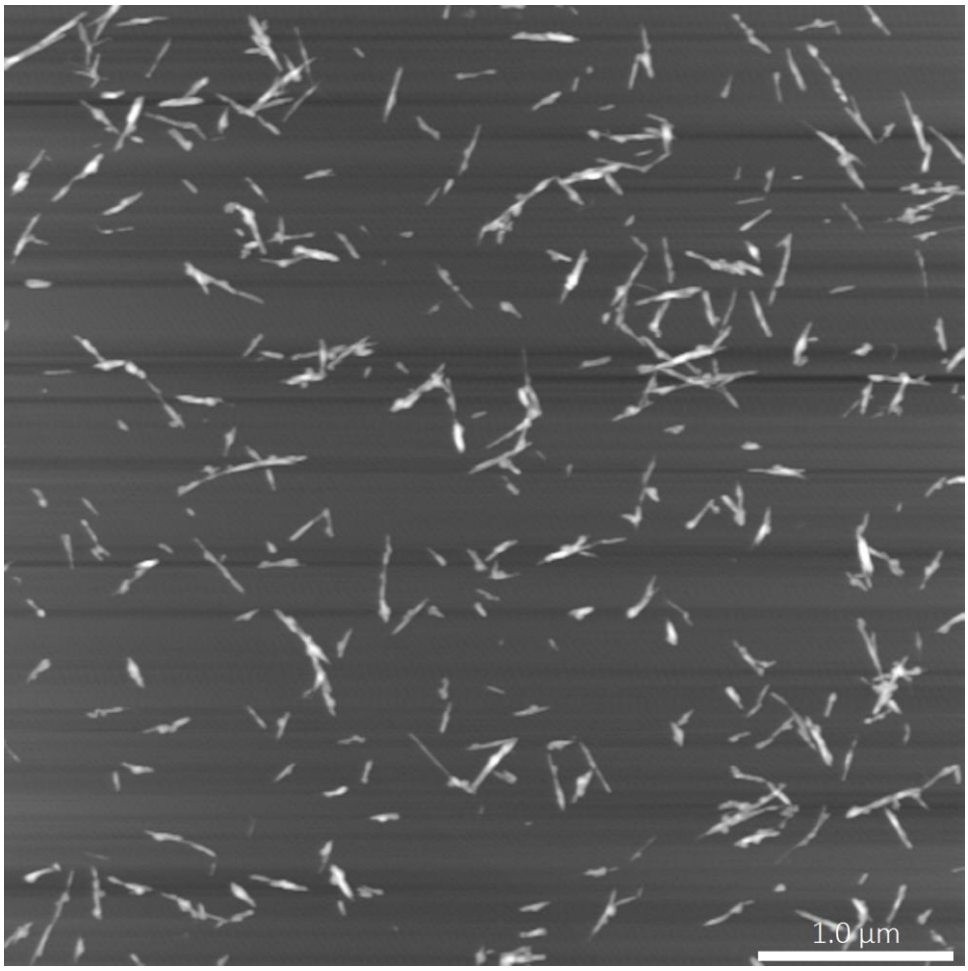

Figure S3. AFM image of S1 sample.

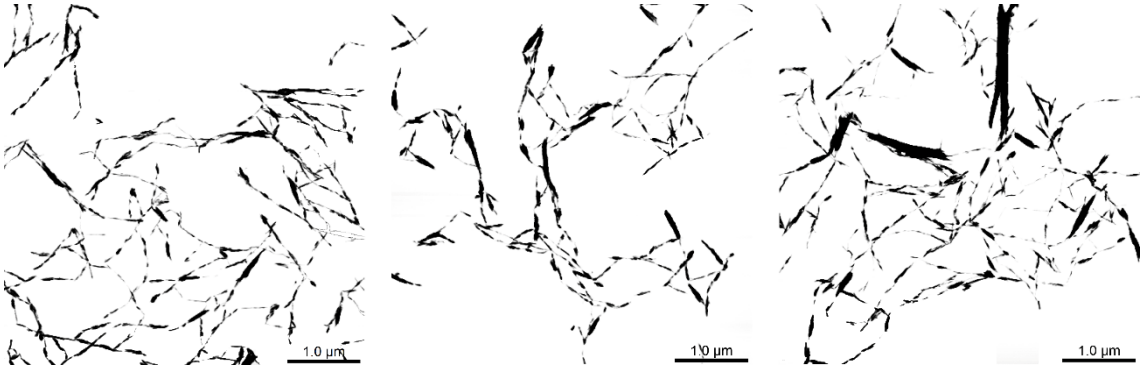

Figure S4. AFM two-tone images of three C1 samples.

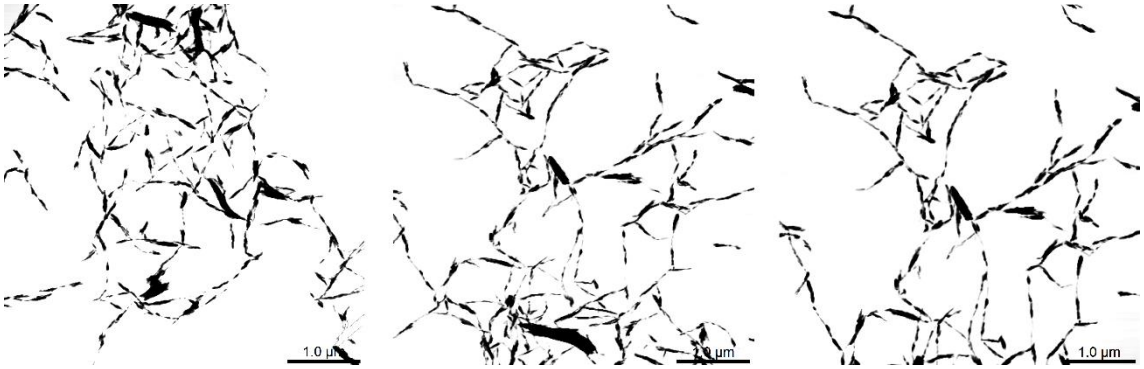

Figure S5. AFM two-tone images of three C2 samples.

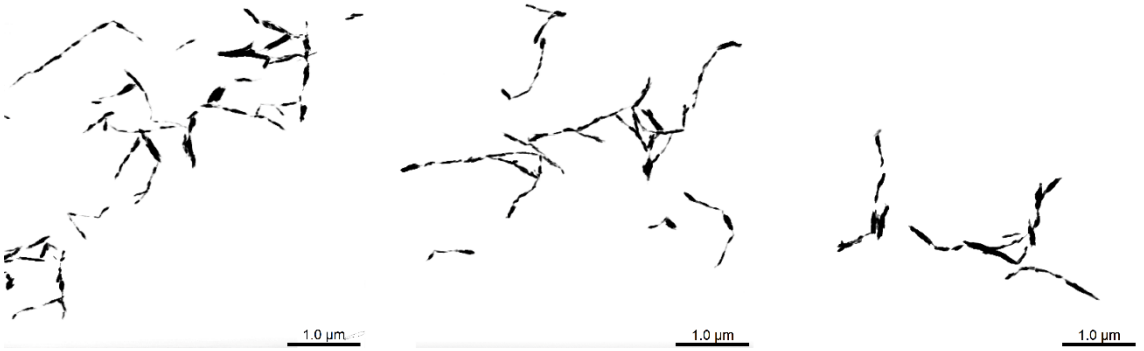

Figure S6. AFM two-tone images of three C3 samples.

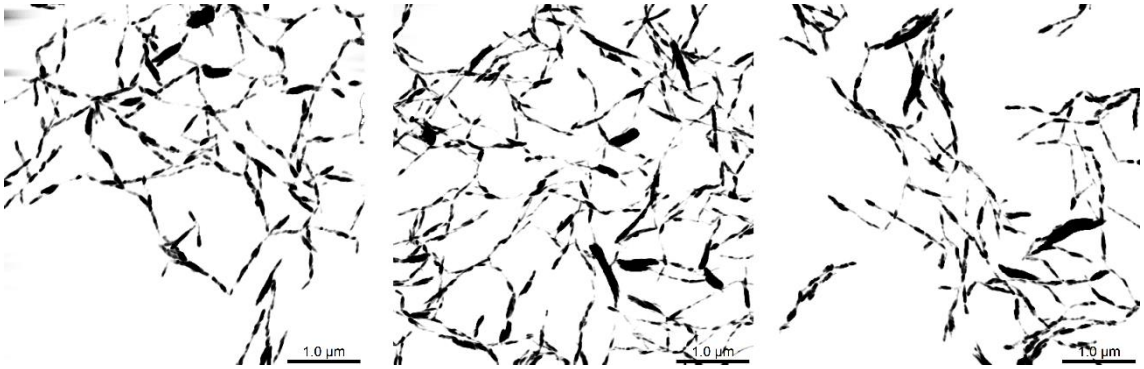

Figure S7. AFM two-tone images of three M1 samples.

Figure S8. AFM two-tone images of three M2 samples.

Figure S9. AFM two-tone images of three M3 samples.

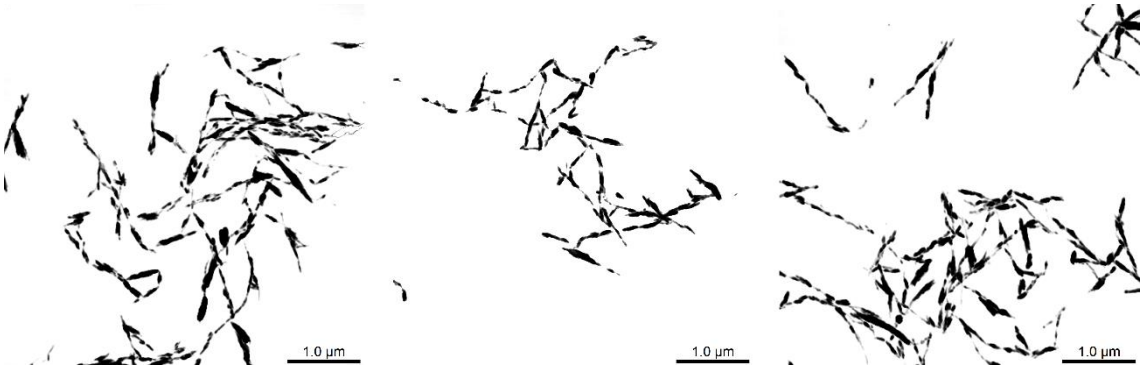

Figure S10. AFM two-tone images of three O1 samples.

Figure S11. AFM two-tone images of three O2 samples.

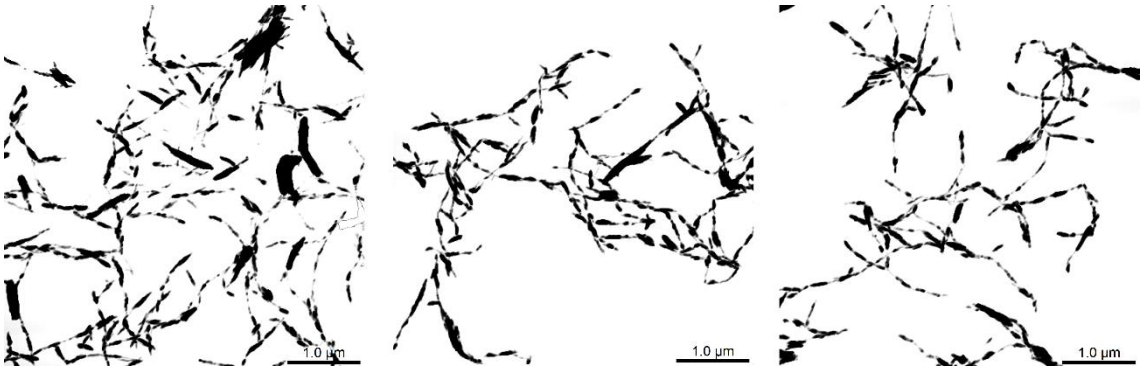

Figure S12. AFM two-tone images of three O3 samples.

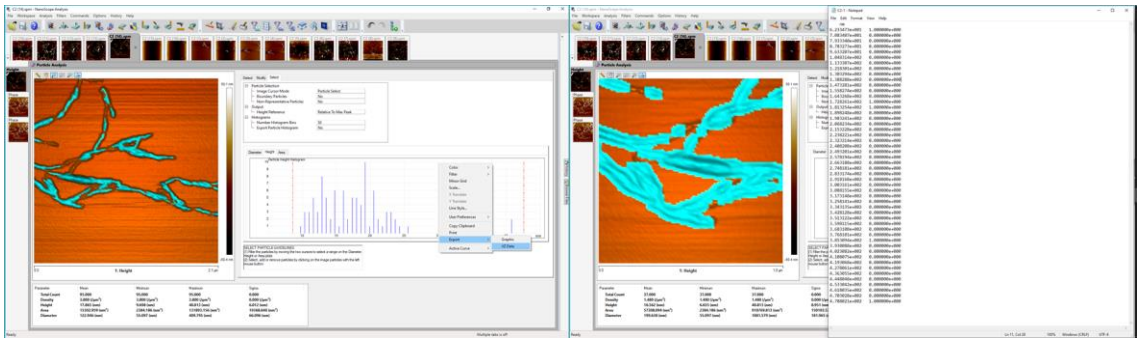

Figure S13. Parameters for automatic measurement with Bruker NanoScope Analysis.

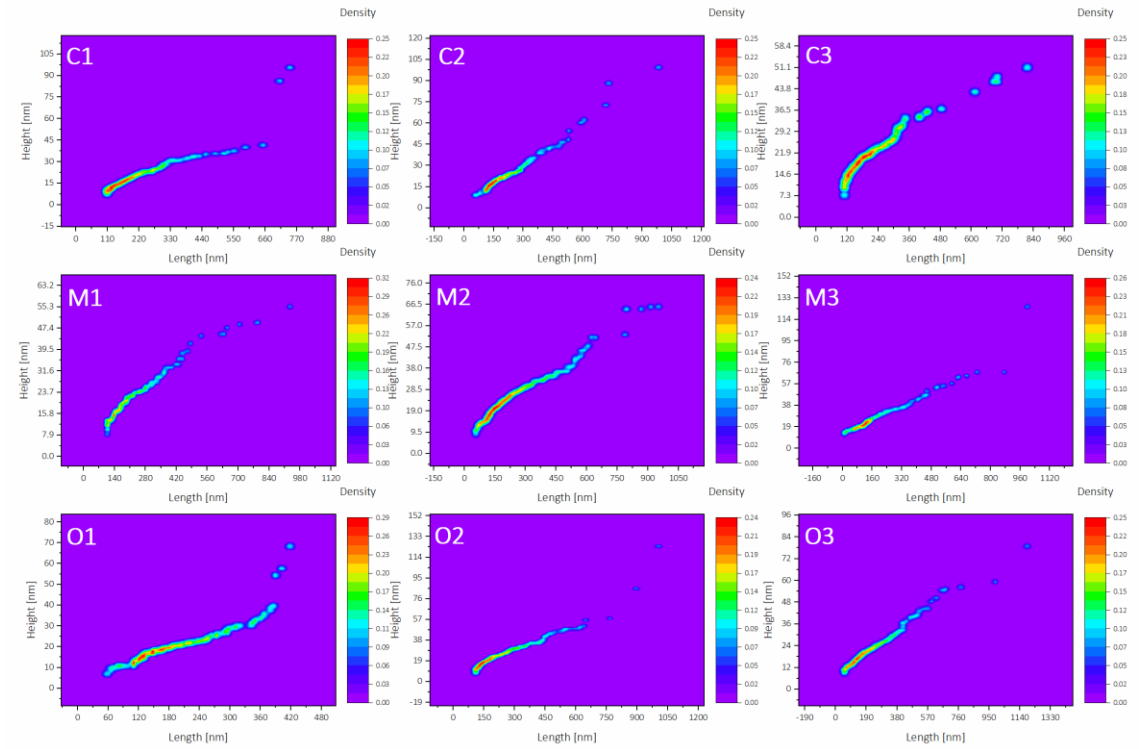

Figure S14. 2D kernel density plot for width vs. height of the nanocelluloses.

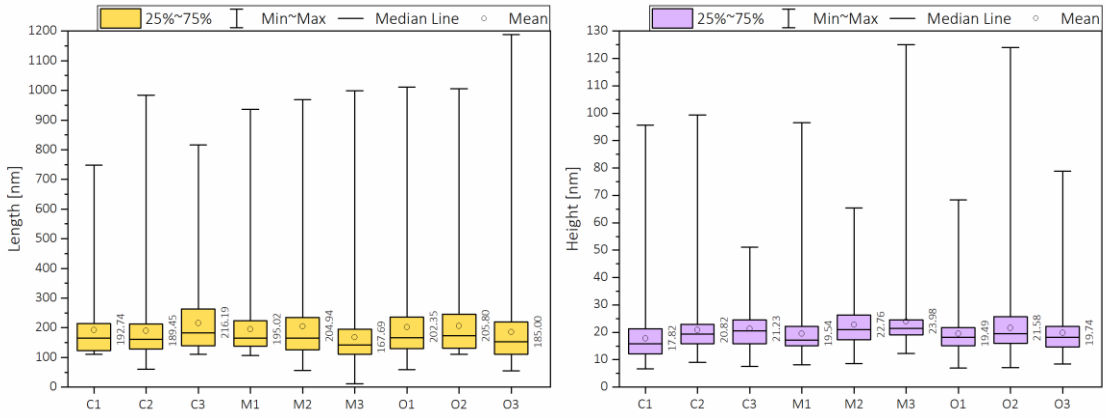

Figure S15. Box chart of length and height as measured automatically, the box contains specimens within the 25%~75% quartiles, with mean values besides.

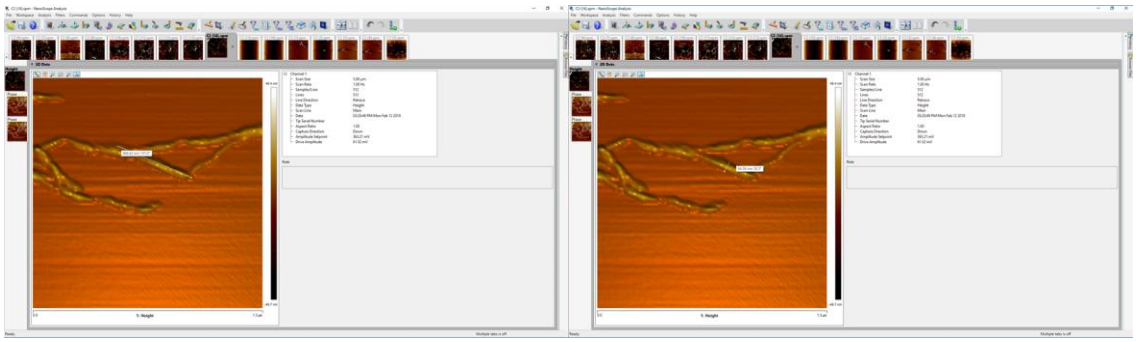

Figure S16. Parameters for manual measurement of the nanocelluloses using Bruker NanoScope Analysis.

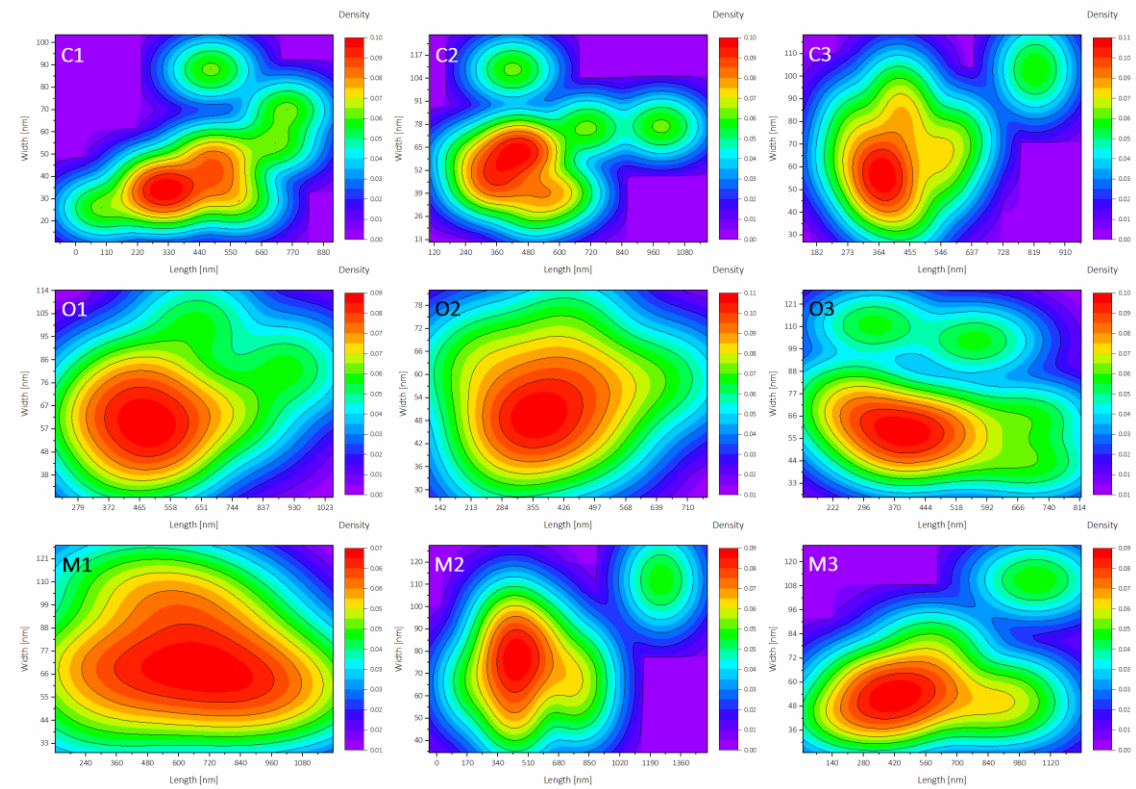

Figure S17. 2D kernel density plot for width vs. length of the nanocelluloses.

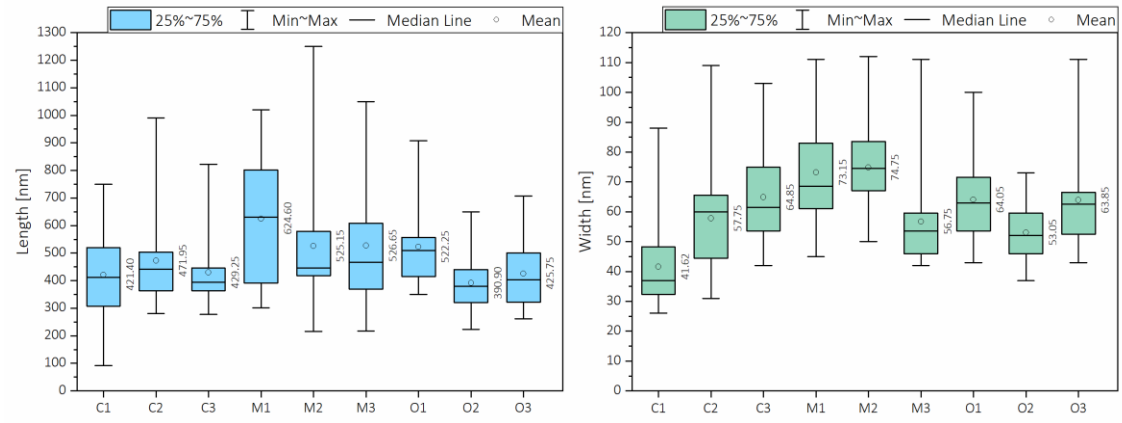

Figure S18. Box chart of length and height as measured manually, the box contains specimens within the 25%~75% quartiles, with mean values besides.

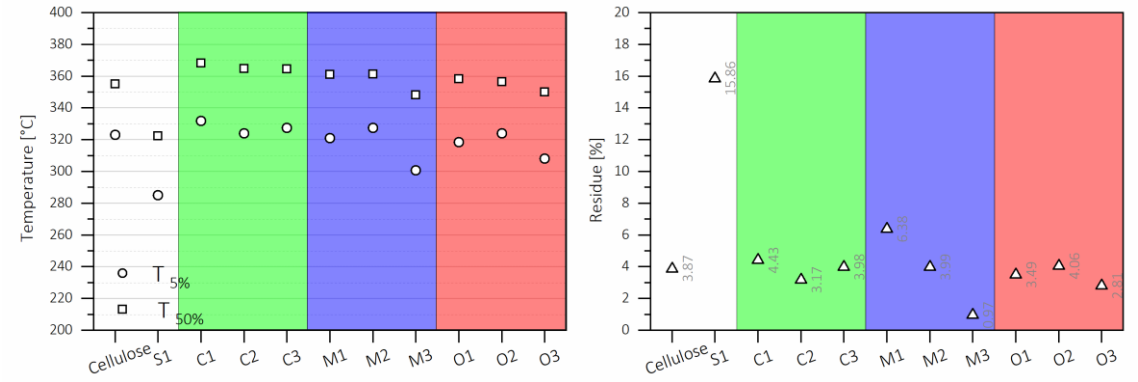

Figure S19. Transition temperatures and carbon residue as obtained from TG analysis.

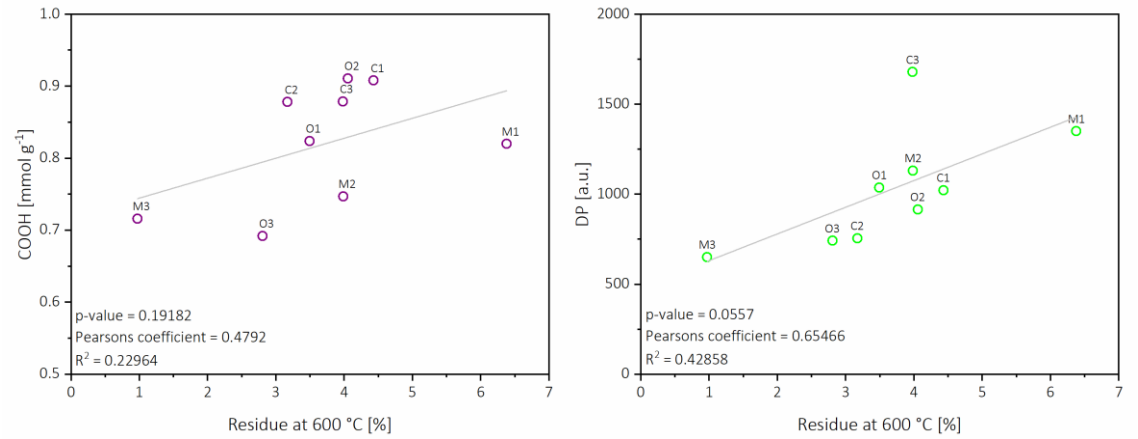

Figure S20. Correlation between residues at 600 °C and carboxylic content and degree of polymerization.

References

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