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Supplementary Information

Ozonolysis of regular and crosslinked lignin nanoparticles: closing the loop

Alexandros E. Alexakis* and Mika H. Sipponen*

Department of Materials and Environmental Chemistry, Stockholm University, 10691 Stockholm, Sweden
E-mail: alexandros.alexakis@mmk.su.se, mika.sipponen@mmk.su.se

* Corresponding authors

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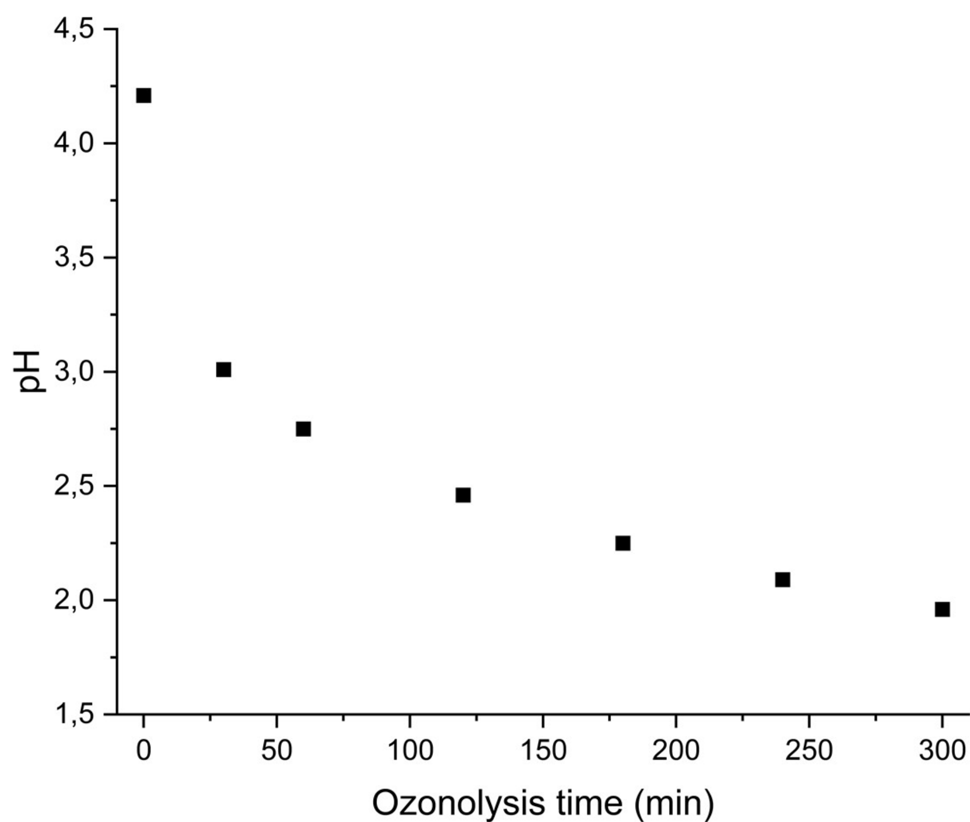

Figure S1. Evolution of pH of the LNP dispersion during ozonolysis without pH adjustment.

Table S1. Dry content of LNP and HLNP dispersions after ozonolysis. The volume of the ozonated dispersion is shown in parenthesis.

Ozonolysis time (min)	**LNP (50 mL)**	**LNP (10 mL)**	**HLNP (10 mL)**
0	4.7	4.7	2.7
300	8.9	7.9	5.3

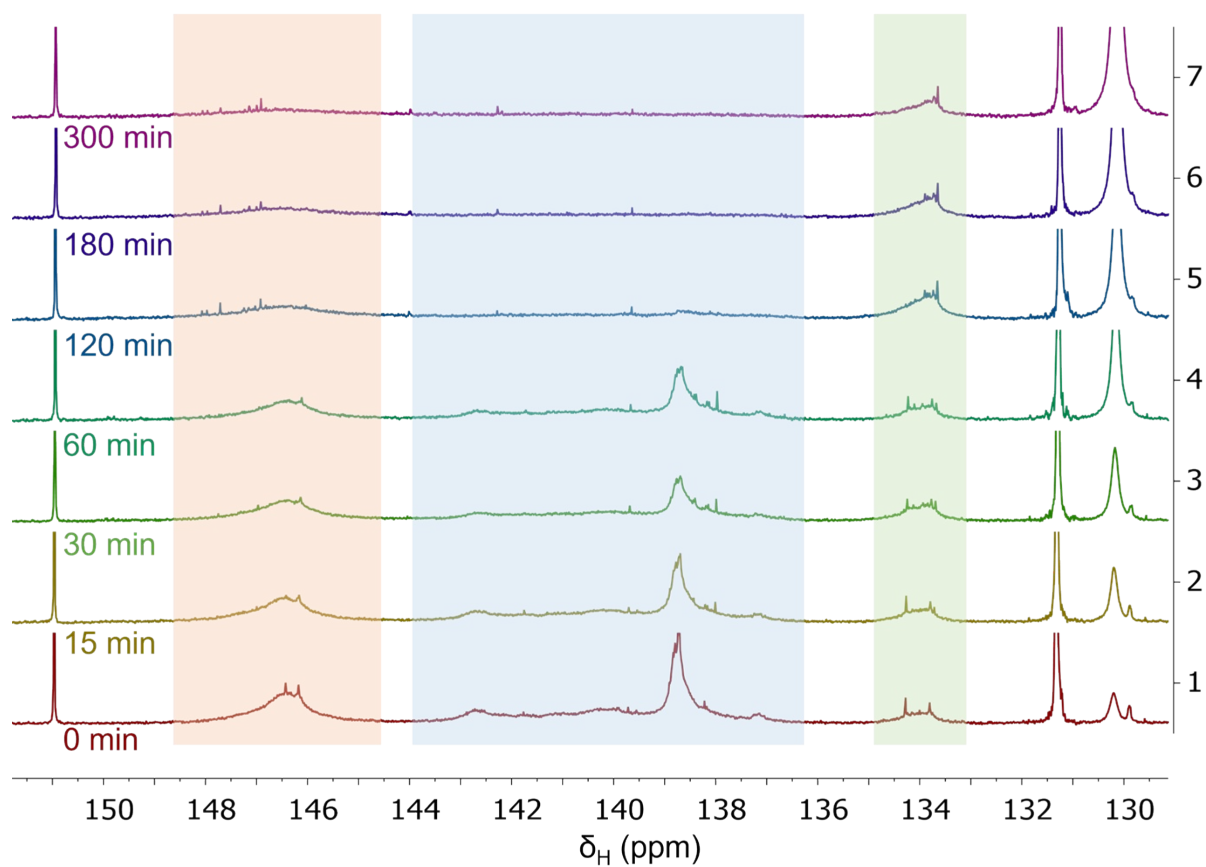

Figure S2. ^{31}P-NMR of the lyophilized LNPs aver ozonolysis. The ozonolysis time is shown on the bottom left of each curve which is color coded to match the respective curve. Three regions are also highlighted to mark the aliphatic -OH (orange, left), phenolic -OH (blue, middle) and carboxylic acid (green, right).

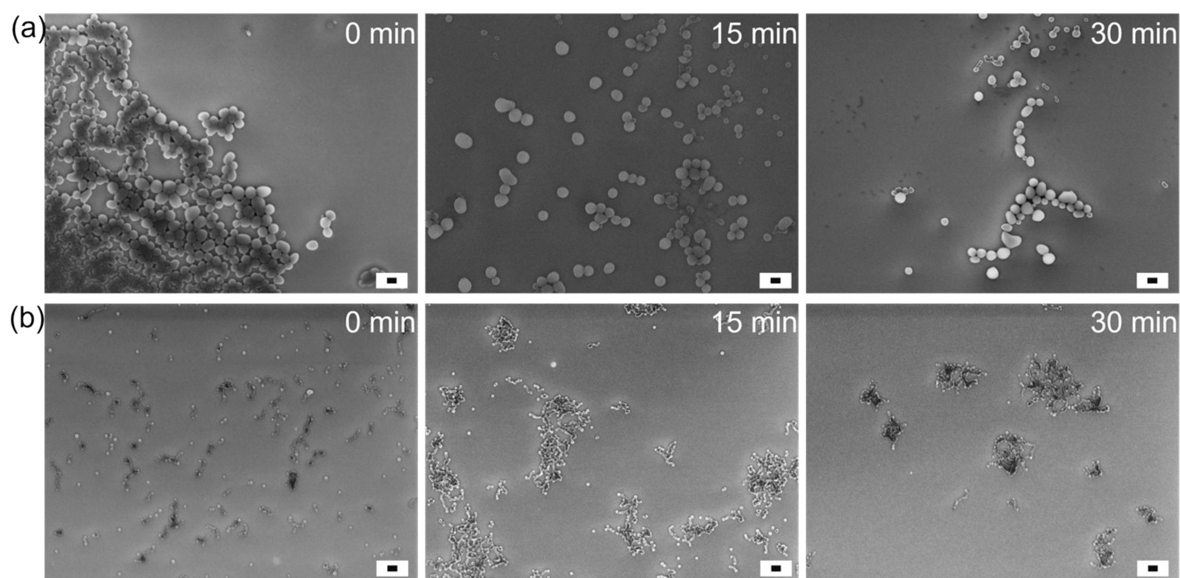

Figure S3. Low magnification SEM images of the ozonated LNPs (a) and HLNPs (b). The scale bar in all SEM images is 100 nm.

Figure S4. Digital photos of the 50 mL LNP (a) and 10 mL HLNP (b) dispersions after ozonolysis.

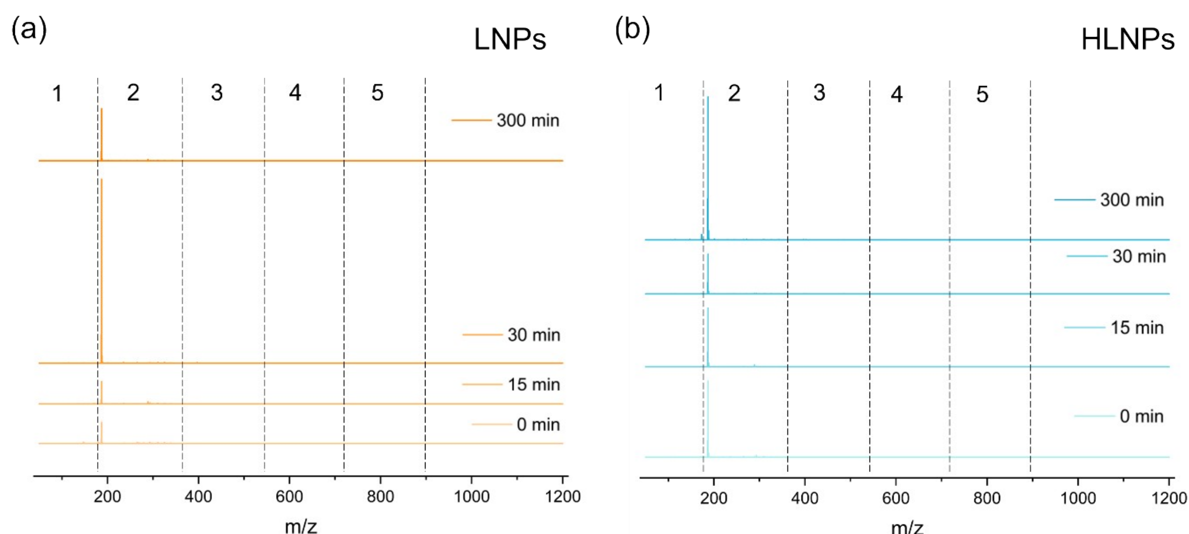

Figure S5. LC-MS data for the ozonated LNPs (a) and HLNPs (b) from the negative mode. The dashed lines indicate the different regions where monomers (1), dimers (2), trimers (3), tetramers (4) and oligomers (5) can be found.

LC-MS sample analysis method

Identification of the small molecule degradation products was out of scope of the present work, but further studies could attempt to use chromatographic analysis to identify short-chain organic acids among other oxidized species. To increase reliability when comparing signal intensity for samples at different time points, ion mobility separation was used in combination with the mass spectrometric separation. However, it has to be noted that variability in the signal intensity that is due to ion suppression cannot be reduced with this method. No ion fragmentation was provoked in this pilot study as the aim was primarily to see whether differences can be observed in the mass spectra, depending on the ozonation time, without attempting to identify individual compounds. Progenesis QI detected ions forming drift time peaks and compared the area of those peaks across the sample set. These features were filtered in order to focus on those that 1) had an at least 2 times larger drift time peak area than the blank and 2) showed an increasing or decreasing time trend within the sample set.