Supplementary Information

Simple process for lignin nanoparticle preparation

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Determination of residual THF in the lignin nanoparticle dispersions

The amount of THF in the lignin nanoparticle (LNP) dispersions after dialysis with water was quantified by headspace gas chromatography-mass spectrometry (GC-MS) and ^{1}H-NMR spectroscopy.

Figure S1 presents a GC chromatogram of a freshly prepared LNP dispersion. The experiments were carried out with a TRACE 1300 Gas Chromatograph ISQ-MS instrument equipped with a TriPlus RSH Autosampler (Thermo Scientific) using headspace sample preparation method. The peaks of toluene (added to the sample as an internal standard) and THF were identified from the mass spectra in addition to retention time. Integration of peak areas and comparison with a control sample of known THF concentration revealed that the amount of residual THF in the LNP dispersion after dialysis was negligible, i.e., below 1 ppm (0.74 µg/ml).
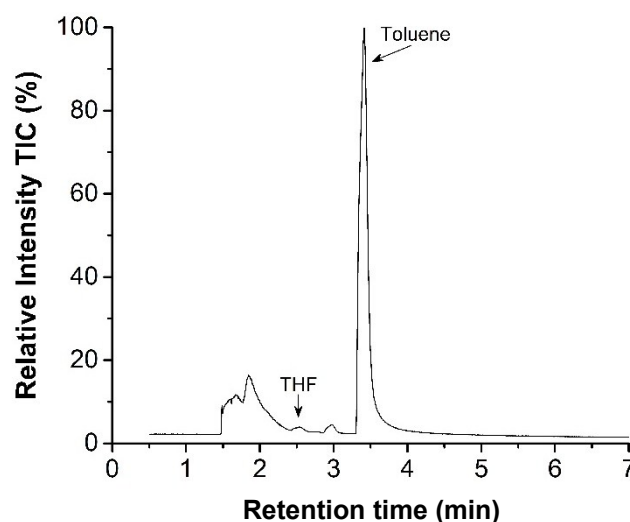

Figure S1. GC chromatogram of a LNP dispersion. The peaks of toluene (internal standard) and THF are indicated.

^{1}H-NMR analyses were carried out using a Bruker AVANCE 400 MHz instrument. The ^{1}H-NMR spectrum of a LNP dispersion after solvent exchange from H_2O to D_2O by centrifugation (twice) followed by dissolution in deuterated DMSO showed negligible traces of THF (very small peak at 1.76 ppm in Figure S2). By comparing the areas of the aromatic peaks and the 1.76 ppm THF peak of Figure S2 with the corresponding spectrum of a control sample of known THF concentration (1:1 THF:lignin weight ratio), the THF:lignin weight ratio in the sample was estimated to be 0.00051:1 (0.051 wt-%), equivalent to a THF concentration in the LNP dispersion below 1 µg/ml, in agreement with the result obtained by GC-MS.
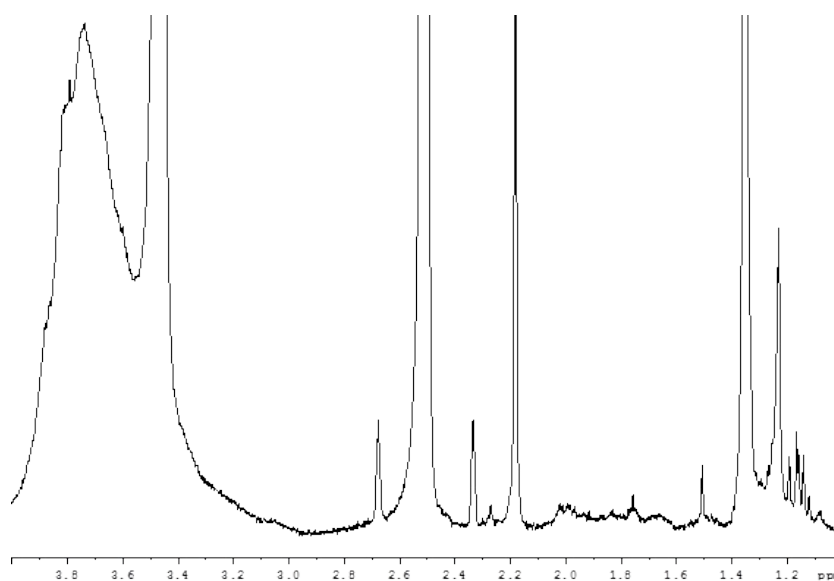

Figure S2. ^{1}H-NMR spectrum of LNP after solvent exchange (D_2O) and dissolution in deuterated DMSO.