Electronic Supplementary Information

Producing wood-based nanomaterials by rapid fractionation of wood at 80°C using a recyclable acid hydrotrope

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Fig. S1 Image showing re-crystallizaed *p*-TsOH acid (top) in undiluted spent liquor (bottom) at ambient temperature. The bottle was set upside down to differentiate the lignin containing *p*-TsOH crystals (dark color from lignin) from the dissolved lignin liquor (also dark color).

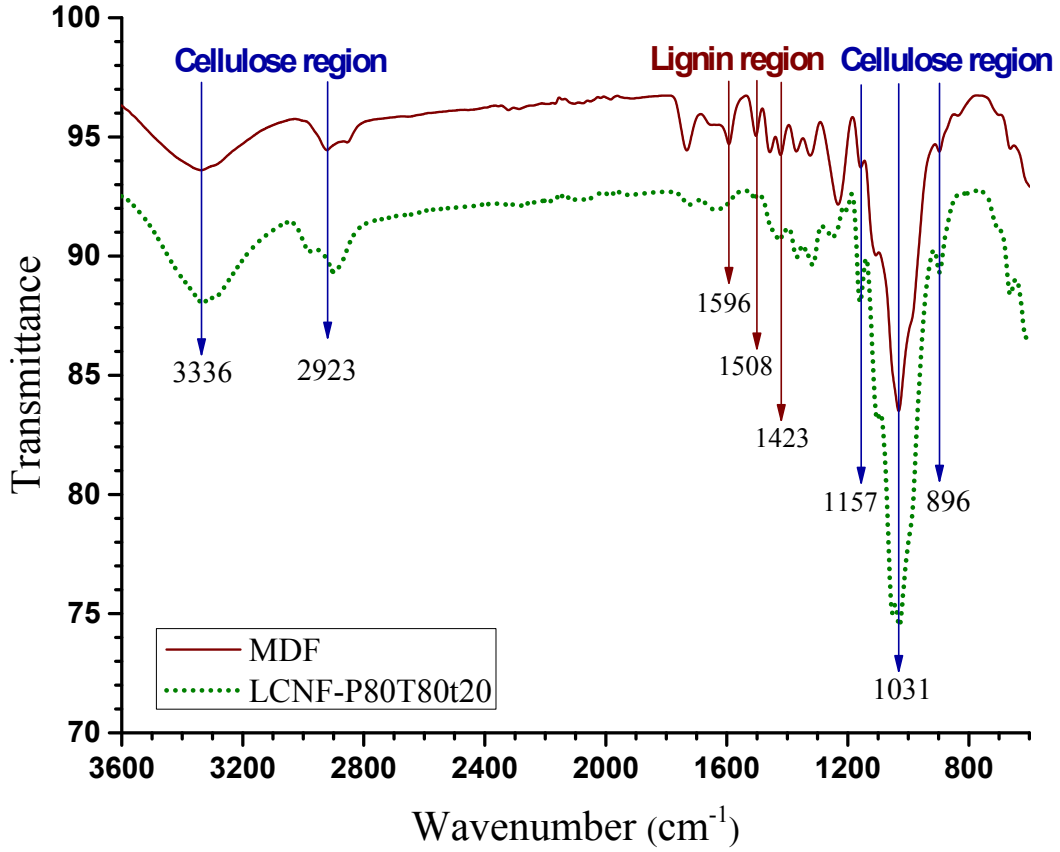

Fig. S2 Comparison of FTIR absorption spectra between the MDF fiber and the LCNF-P80T80t20 (from the most severe *p*-TsOH treatment with lignin removal of approximately 85%). Identified peaks (cm^{-1}) for cellulose: 3336 – O-H stretching in aliphatic and phenolic OH; 2923 – C-H stretching in methyl and methylene groups; 1157 – C-O-C stretching; 1031 – aromatic C-H in-plane deformations in G units and C-O deformations in primary alcohols; 896 – C-H deformation vibration. For lignin: 1596 – C=C benzene ring vibration; 1508 – aromatic skeletal vibration; 1423 – aromatic skeletal vibrations.

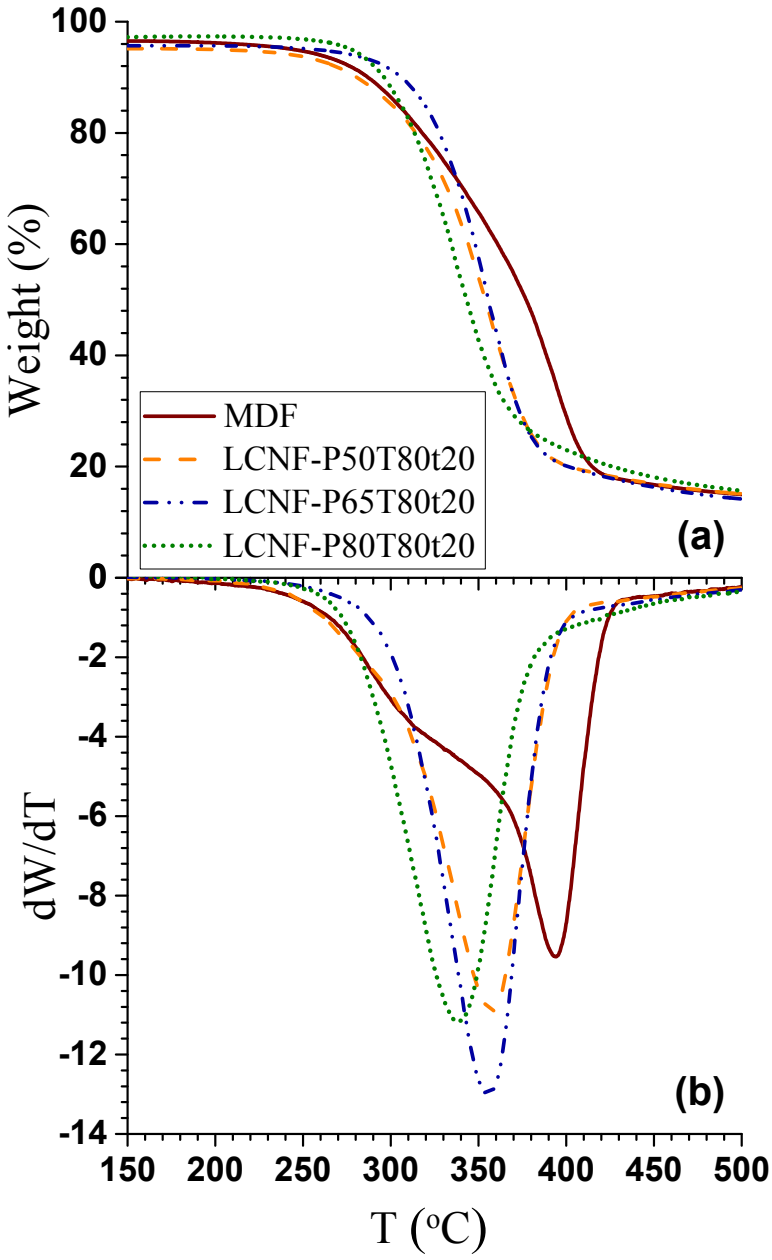

Fig. S3 Thermogravimetric analysis results of three LCNF samples in comparison with those of the MDF. (a) weight loss; (b) temperature derivative weight loss.

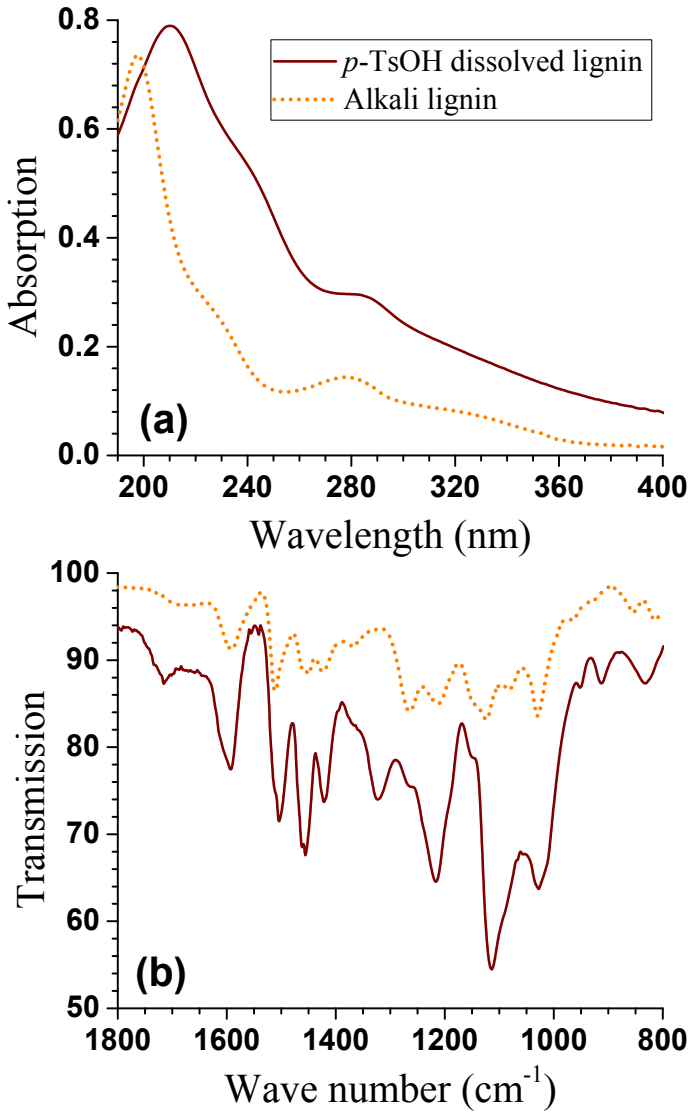

Fig. S4 Comparisons of *p*-TsOH dissolved lignin (dialyzed LNP suspension from P80T80t20) UV (a) and FTIR (b) absorption spectra with their corresponding spectrum from a commercial alkali lignin (Sigma CAS 8068-05-1).

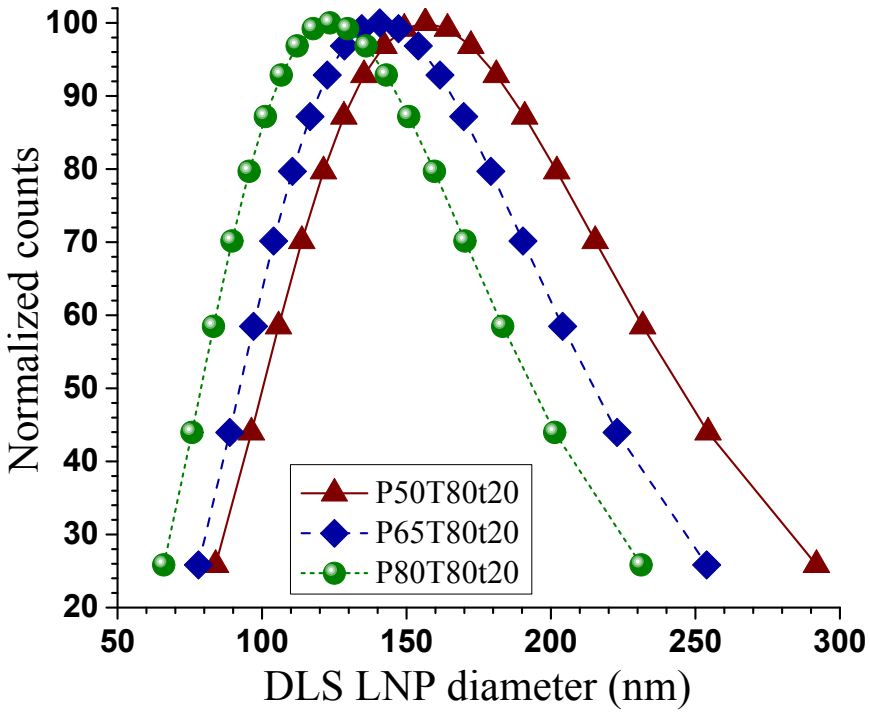

Fig. S5 Effect of fractionation severity on dynamic light scattering measured lignin nanoparticle (LNP) size distributions.

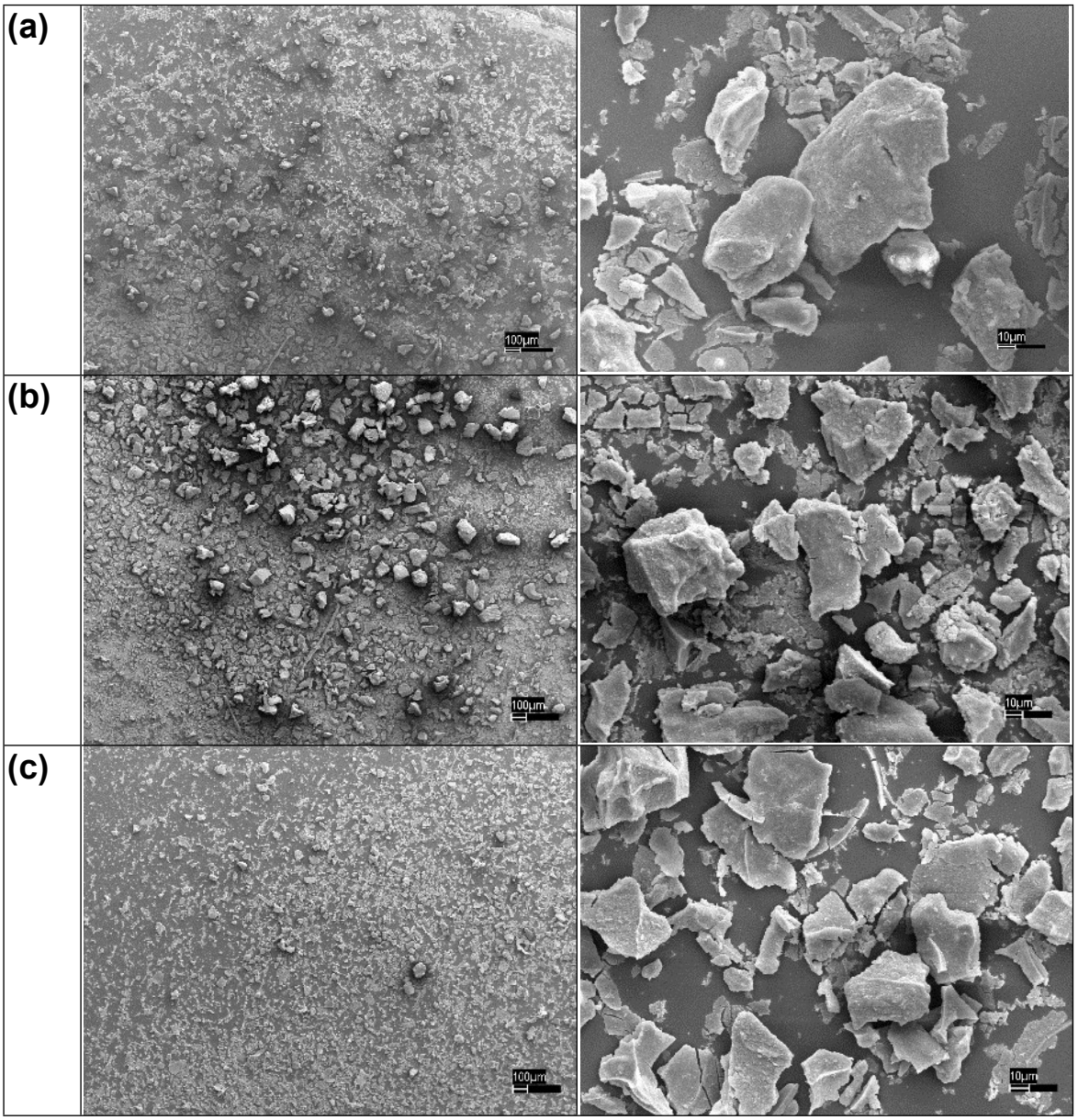

Fig. S6 SEM images of freeze-dried lignin nanoparticles (LNP) obtained from different fractionation severities. (a) P50T80t20; (b) P65T80T20; and (c) P80T80t20. Scale bars = 100 µm (left panel) and = 10 µm (right panel).

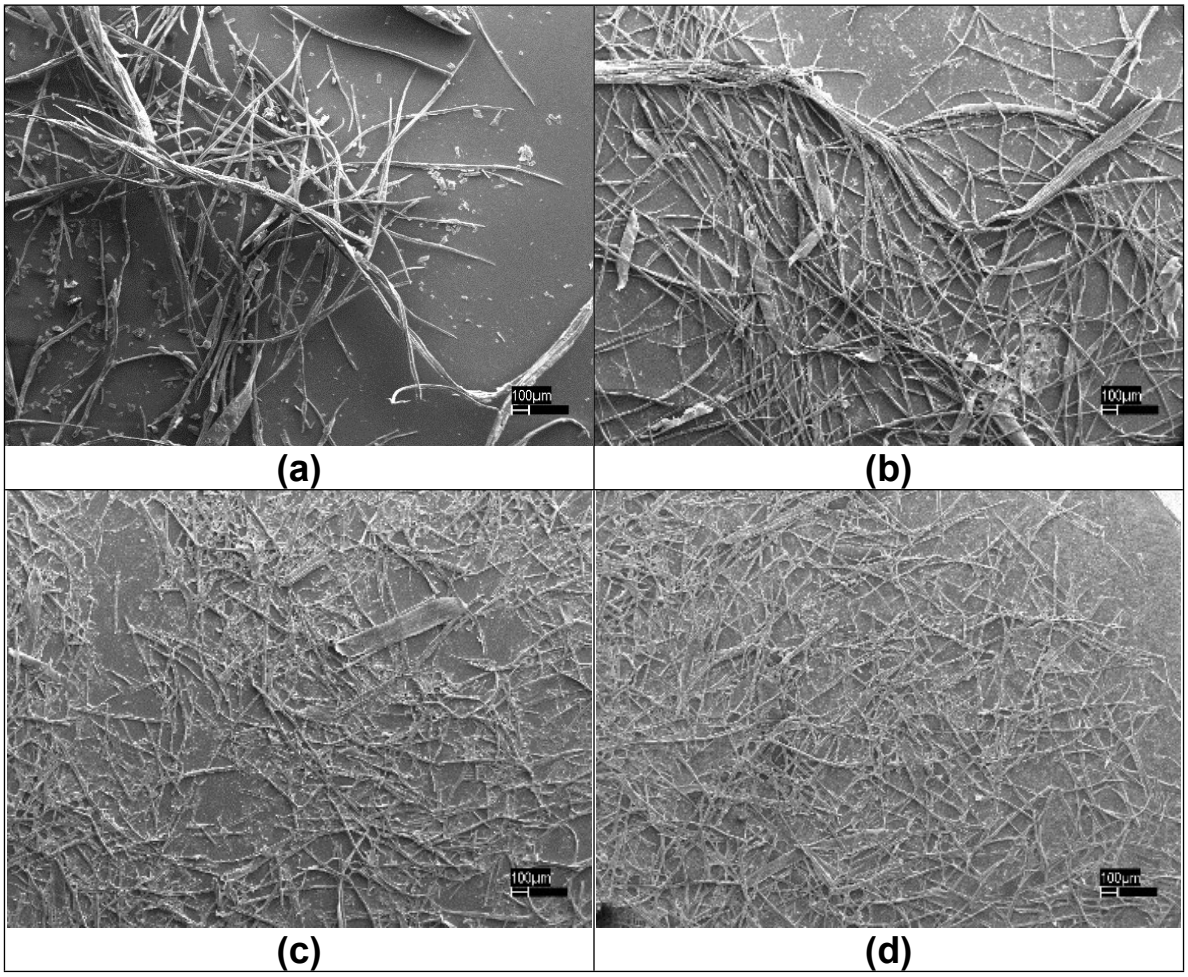

Fig. S7 SEM images of *p*-TsOH fractionated (washed) water insoluble solids (WIS) along with the feed medium density fiberboard fibers (MDF). All scale bars = 100 µm. (a) MDF; (b) WIS-P50T80t20; (c) WIS-P65T80t20; (d) WIS-P80T80t20.

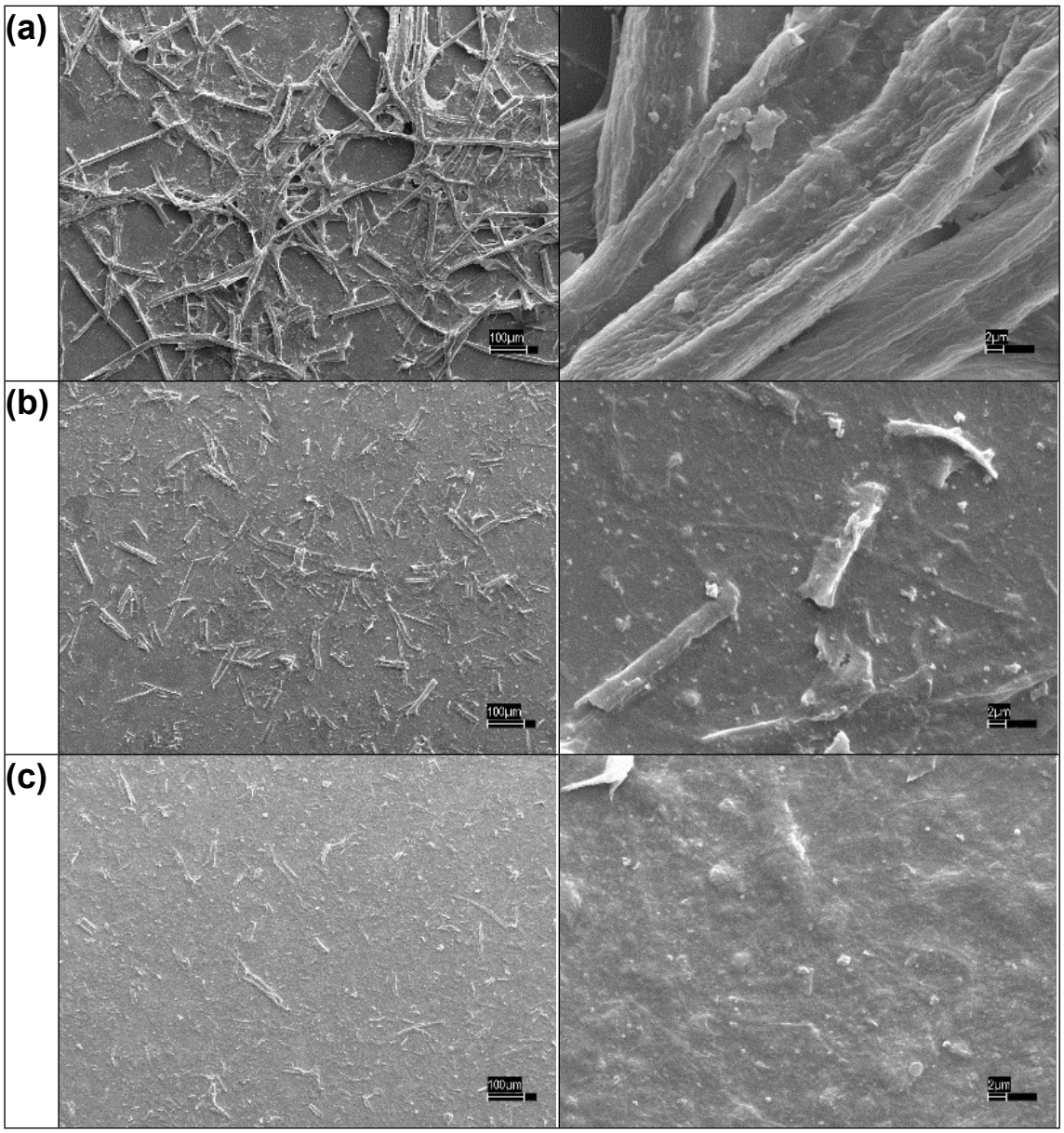

Fig. S8 SEM images of cellulose microfibrils produced from fractionated lignocellulosic solid residue (LCSR) under different severities. LCSR were from dialyses of the washed water insoluble solids (WIS) to separate the lignocellulosic crystalline fibrils (LCCNF). The fibrils shown were from mechanical fibrillation only through the 200 µm chamber for 3 passes at 40 MPa in the microfluidizer. Scale bars = 100 µm (left panel) and = 2 µm (right panel). (a) P50T80t20; (b) P65T80t20; (c) P80T80t20