Niobium pentachloride in a biphasic catalytic system for valorization of corn cob biomass

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General Techniques

Analytical grade commercial solvents and reagents were purchased from Sigma-Aldrich, and used as received. NMR spectra were recorded at 25 °C in DMSO-d_6 on a Bruker AvanceCore spectrometer, operating at 400 MHz for ^{1}H and 100 MHz for ^{13}C or on a Varian Mercury 300 spectrometer operating at 300 MHz for ^{1}H and 75 MHz for ^{13}C. All chemical shifts are reported in parts per million (ppm) and were measured relative to the solvent in which the sample was analyzed (DMSO δ = 2,50 for ^{1}H NMR). The Spectrum mass was determined for gas chromatography coupled to a mass spectrometer using a SHIMADZU GCMS-QP2010C Ultra mass spectrometer and method with the following specifications: column RTx-5 MS, 30 m, DI 0.25 mm; carrier gas helium; injector temperature: 220 °C; oven temperature was: 40 °C (2 min), ramped to 5 °C min^{-1} up to 100 ° C (held for 5 min), ramped to 30 °C min^{-1} up to 200 ° C (held for 5 min).

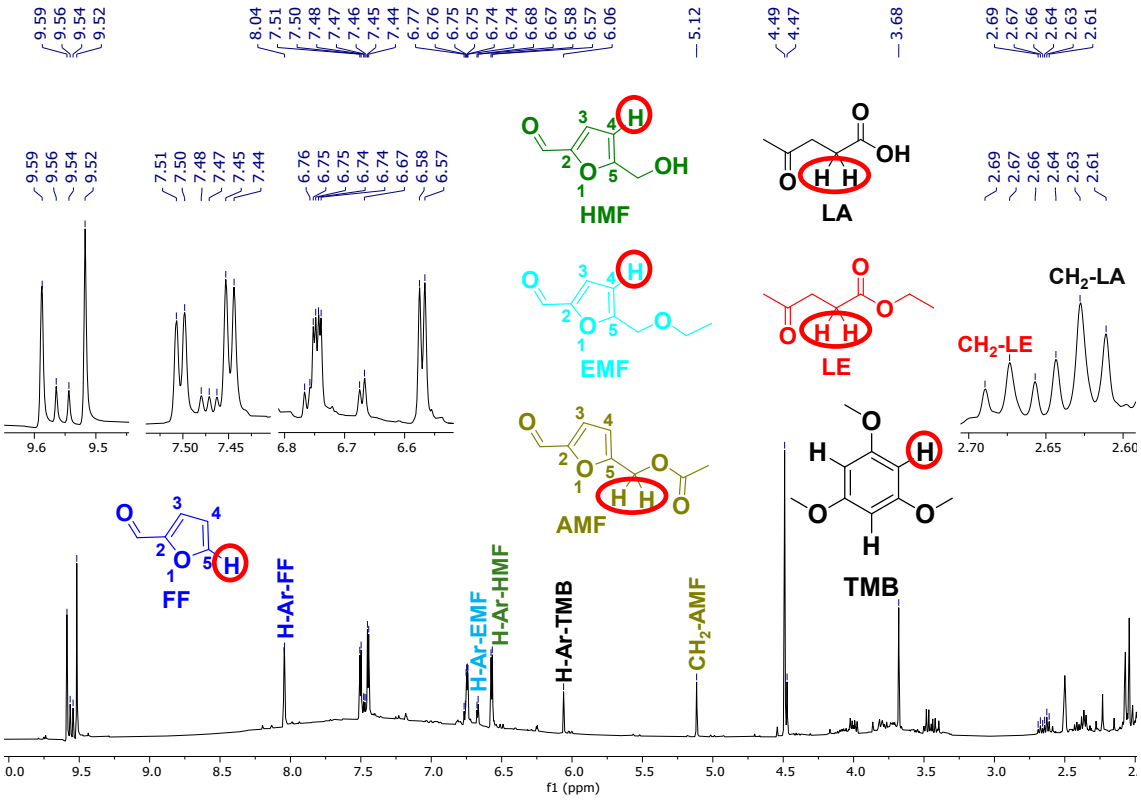

Fig. S1 ^{1}H NMR spectrum clipping (400 MHz, DMSO-d_6, 25 °C) of the crude obtained from the organic phase of the reaction of conversion of corn cob biomass into FF, HMF, EMF, AMF, LE and LA using niobium pentachloride as catalyst (12.5% wt), 200 °C, 3 h.

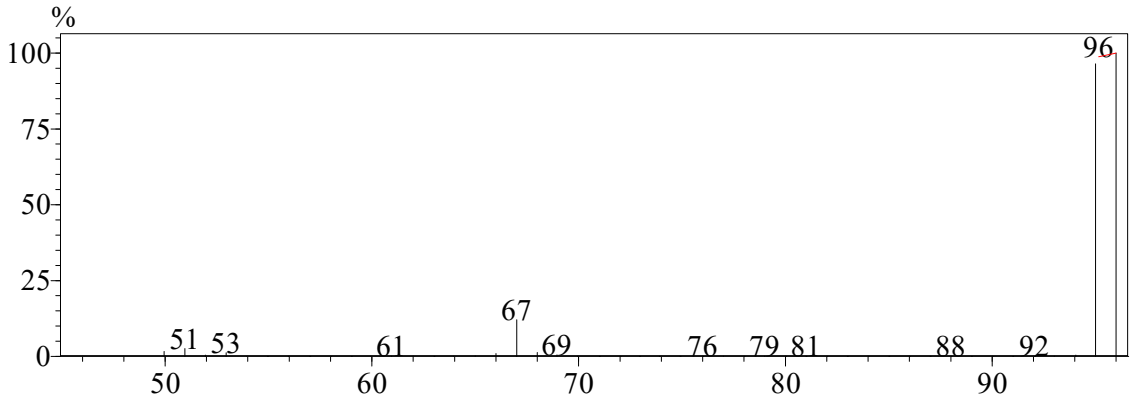

Fig. S2 Mass spectrum (IE 70 eV) of furfural.

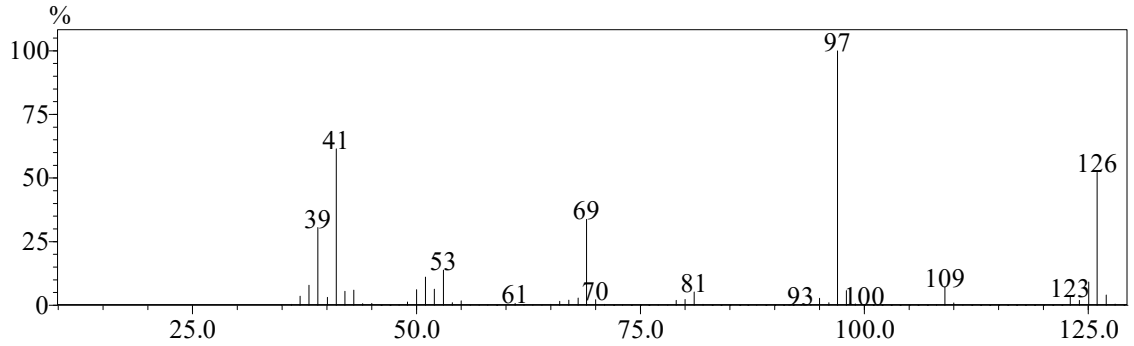

Fig. S3 Mass spectrum (IE 70 eV) of 5-hydroxymethylfurfural.

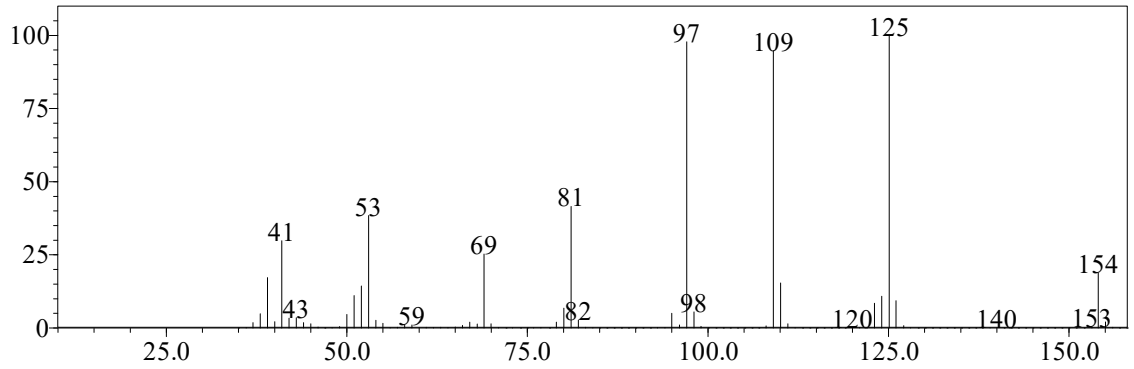

Fig. S4 Mass spectrum (IE 70 eV) of 5-ethoxymethylfurfural.

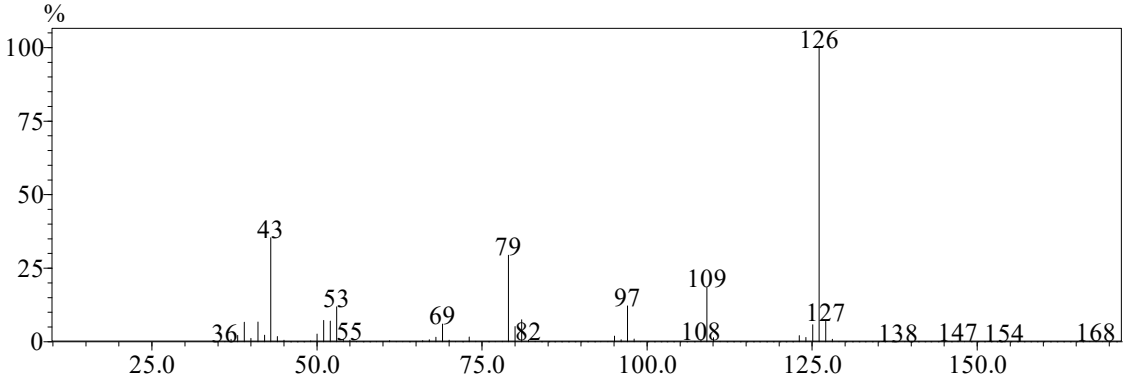

Fig. S5 Mass spectrum (IE 70 eV) of 5-acetoxymethylfurfural.

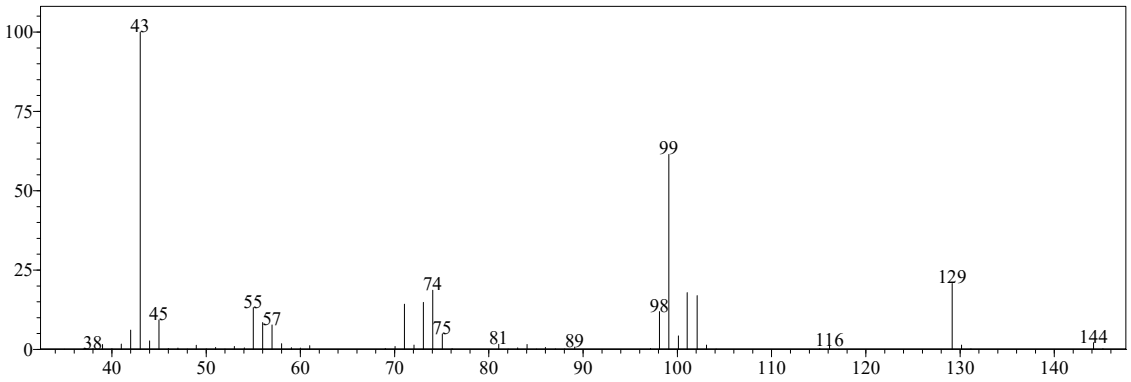

Fig. S6 Mass spectrum (IE 70 eV) of ethyl levulinate.

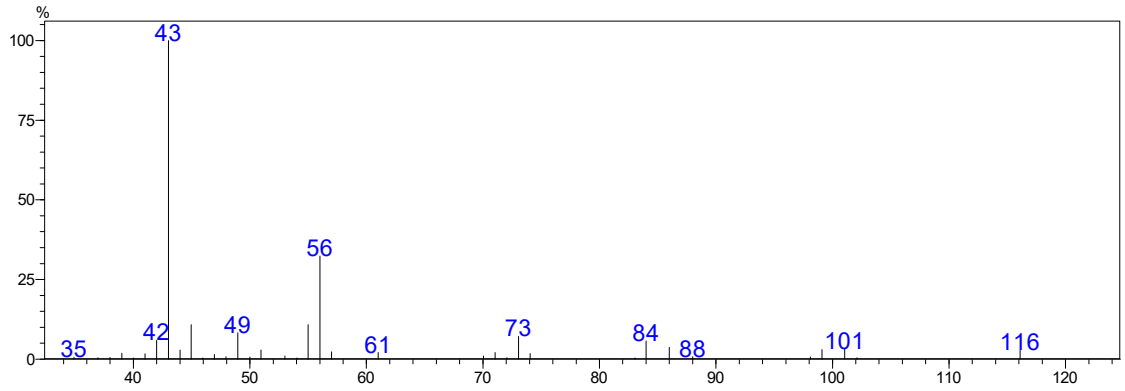

Fig. S7 Mass spectrum (IE 70 eV) of levulinic acid.

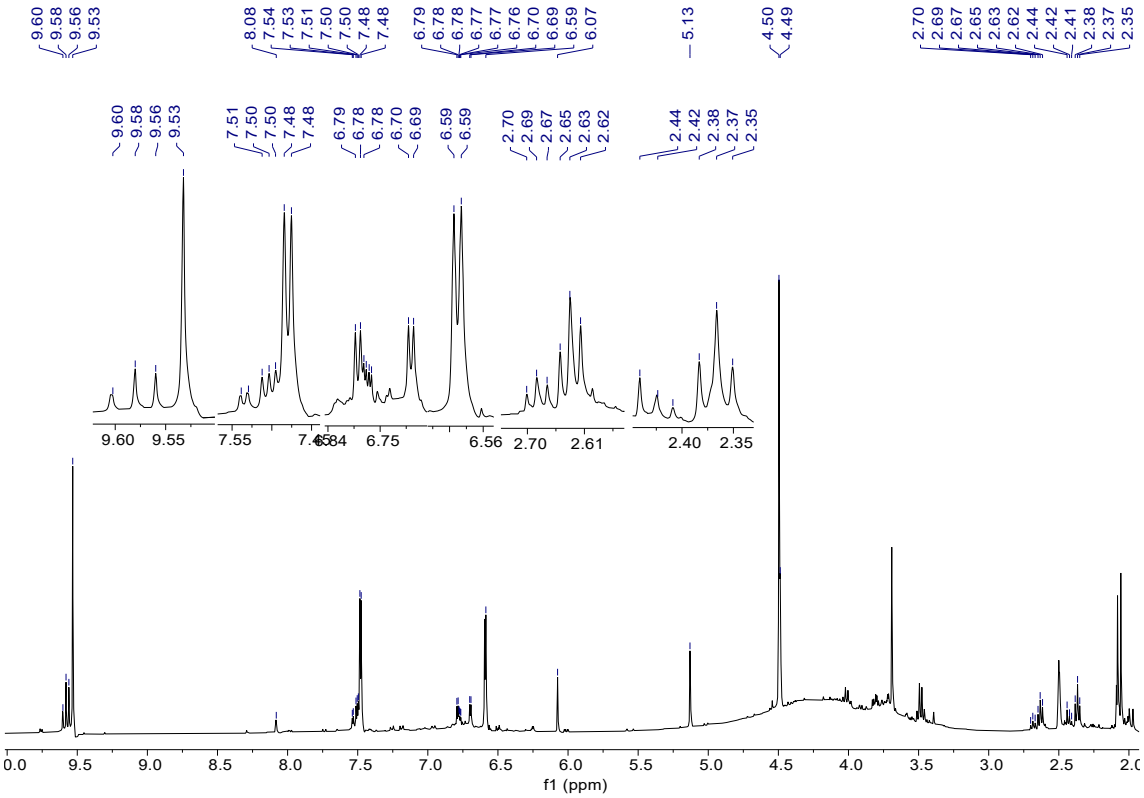

Fig. S8 ^{1}H NMR spectrum clipping (400 MHz, DMSO-d_6, 25 °C) of the crude obtained from the organic phase of the reaction of conversion of bamboo biomass into FF, HMF, EMF, AMF, LE and LA using niobium pentachloride as catalyst (12.5% wt), 200 °C, 3 h.

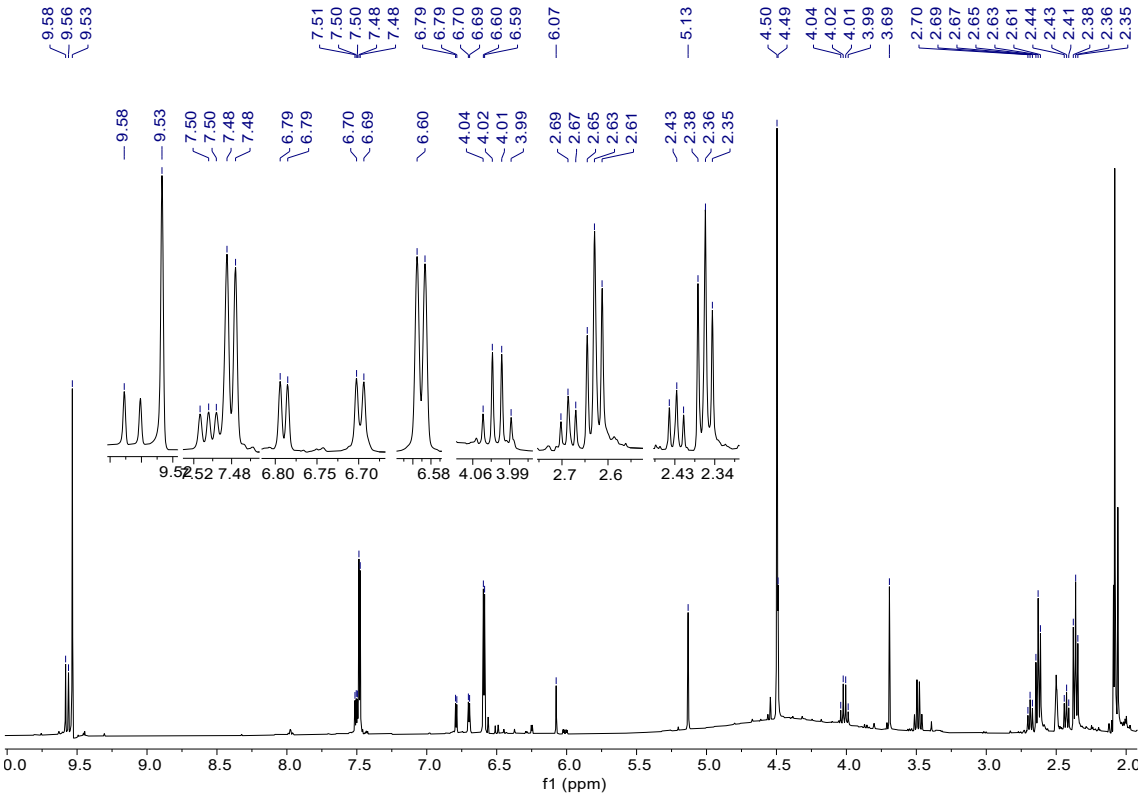

Fig. S9 ^{1}H NMR spectrum clipping (400 MHz, DMSO-d_6, 25 °C) of the crude obtained from the organic phase of the reaction of conversion of microcrystalline cellulose into HMF, EMF, AMF, LE and LA using niobium pentachloride as catalyst (12.5% wt), 200 °C, 3 h.

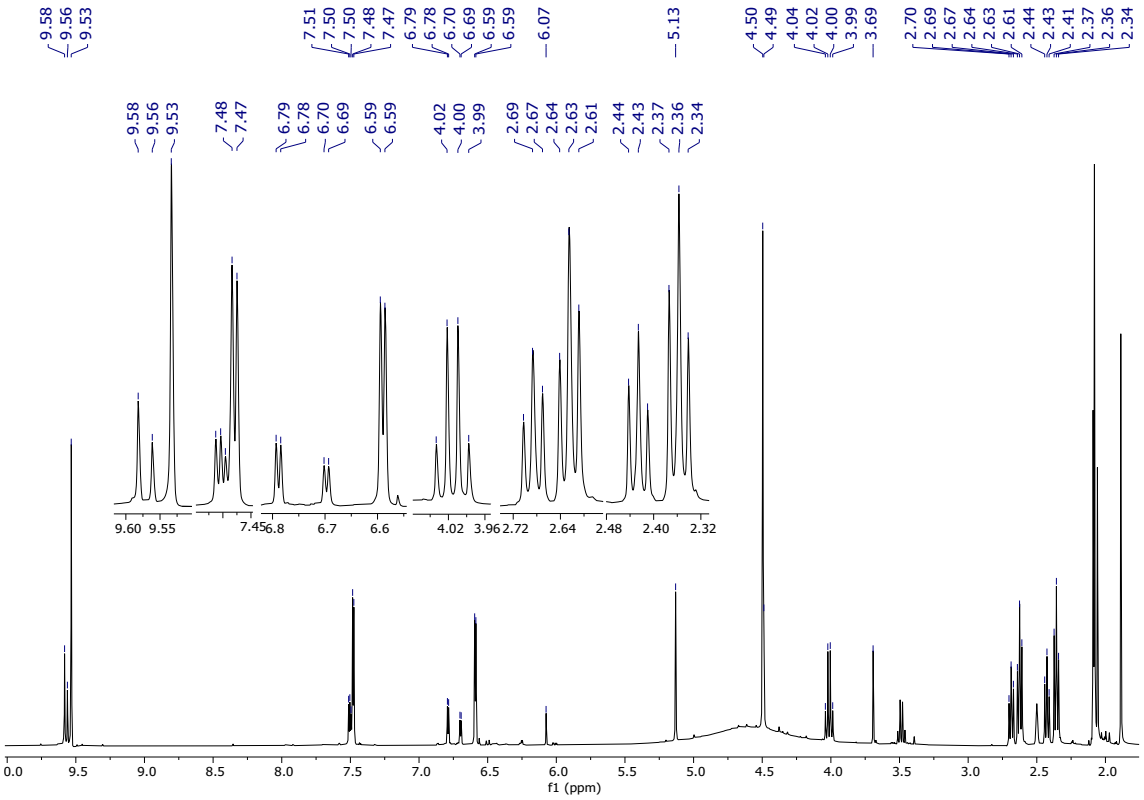

Fig. S10 ^{1}H NMR spectrum clipping (400 MHz, DMSO-d_6, 25 °C) of the crude obtained from the organic phase of the reaction of conversion of inulin *dahlia tubers* into HMF, EMF, AMF, LE and LA using niobium pentachloride as catalyst (12.5% wt), 200 °C, 3 h.

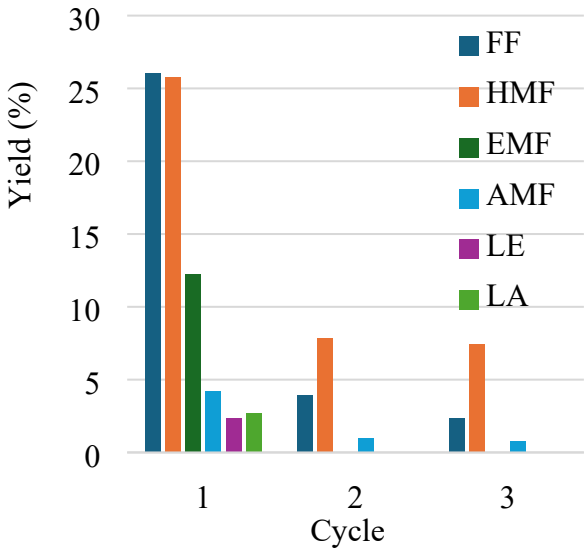

Fig. S11 Products yields (^{1}H NMR analysis) in experiments evaluating reuse of catalytic system. *Reagents and conditions*: 100.0 mg of substrate, 12.5% wt of niobium

pentachloride, 2.0 mL of saturated aqueous NaCl solution, 6.0 mL of ethyl acetate, autoclave reactor, 180 min and 200 °C.