Conversion of wheat straw to furfural and levulinic acid in a concentrated aqueous solution of betaïne hydrochloride

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Analysis of furfural after its extraction with diethyl ether when wheat straw was converted at 180°C for 1 hour under microwave irradiations (250 W): Two technics were employed LC-MS and GC-MS analyses.

- LC-MS QTof Impact HD (Bruker Daltonique) , selected-ion monitoring (*SIM*)

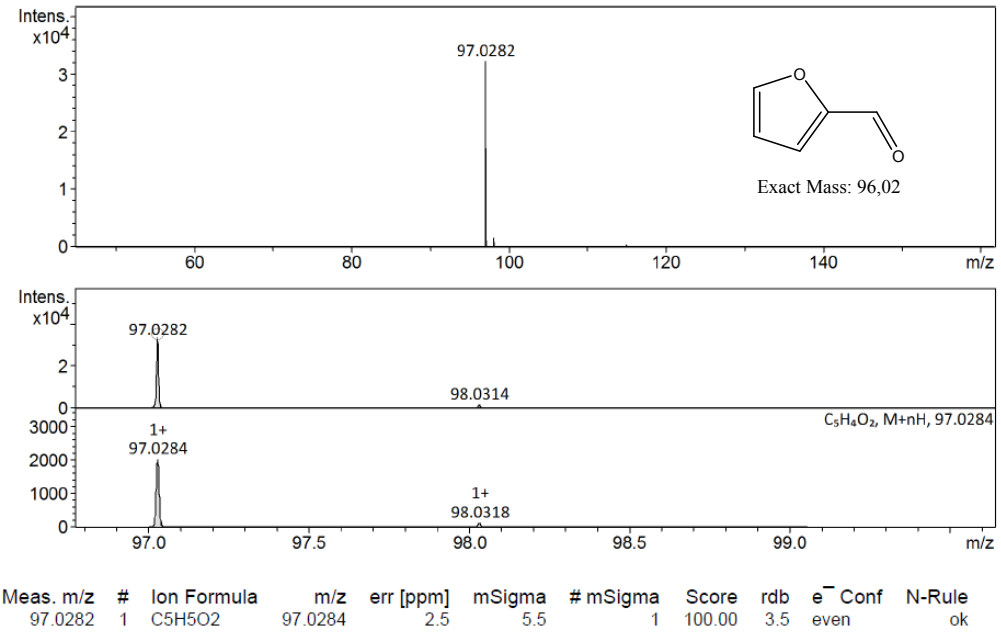


Fig. S1 LC-MC analysis of furfural after its extraction

- GC-MS

GC/Mass Q-TOF "exact mass"

GC: 7890A Agilent, column HP 5 ms 30X0.25X0.25

Mass: 7200 Agilent

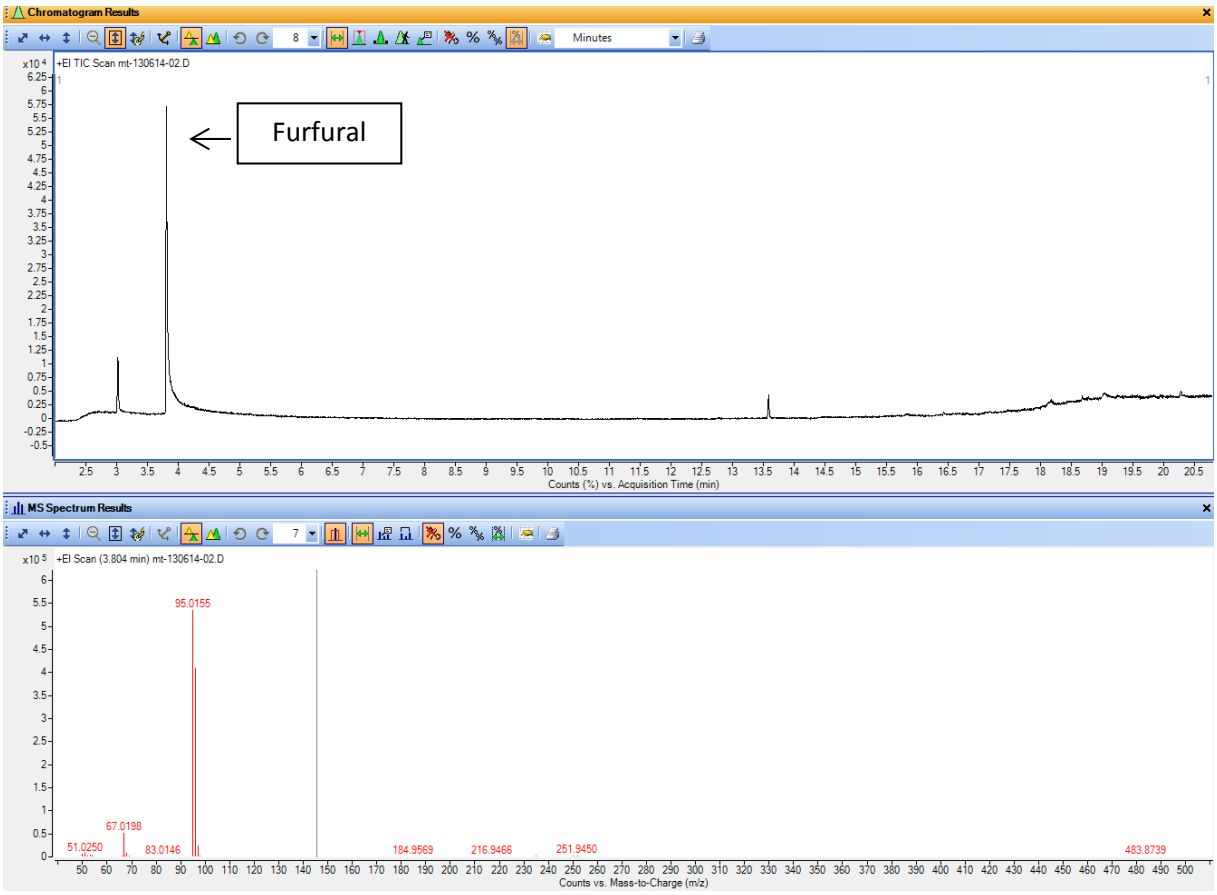
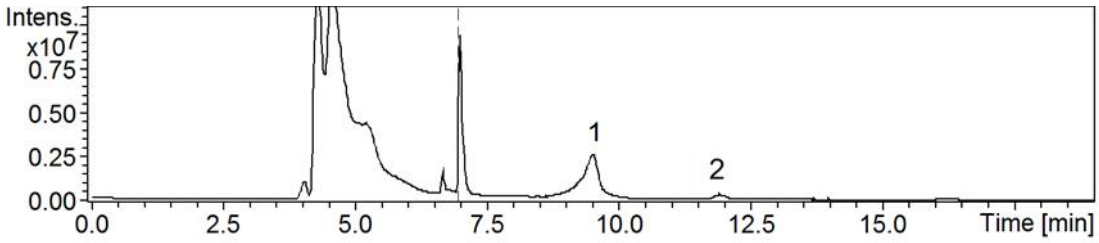


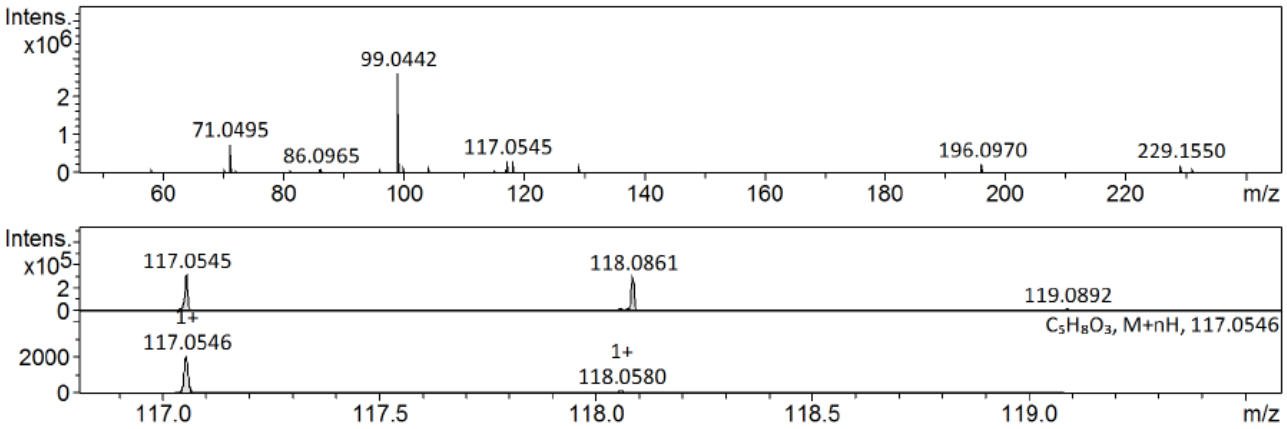
Fig. S2 GC-MC analysis of furfural after its extraction

Analysis of levulinic acid and 5-hydroxymethylfurfural by LC-MC analysis when wheat straw was converted at 180°C for 1 hour under microwave irradiations (250 W):



Source Type	ESI	Ion Polarity	Positive	Set Nebulizer	0.7 Bar
Focus	Active	Set Capillary	3800 V	Set Dry Heater	200 °C
Scan Begin	50 m/z	Set End Plate Offset	-500 V	Set Dry Gas	4.0 l/min
Scan End	800 m/z	Set Charging Voltage	2000 V	Set Divert Valve	Waste
		Set Corona	0 nA	Set APCI Heater	0 °C

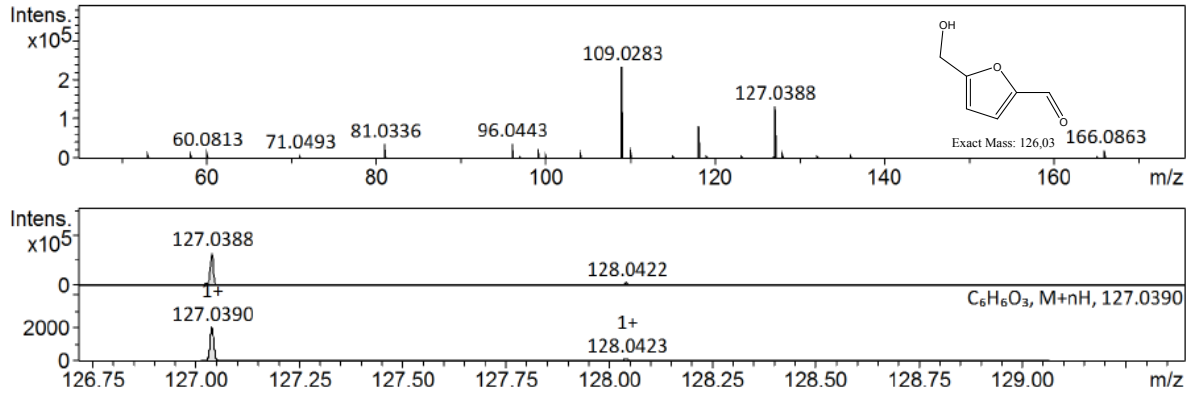
Levulinic acid (1):



Meas. m/z	#	Ion Formula	m/z	err [ppm]	mSigma	# mSigma	Score	rdb	e¯ Conf	N-Rule
71.0495	1	C4H7O	71.0491	-4.9	1.8	1	100.00	1.5	even	ok
99.0442	1	C5H7O2	99.0441	-1.7	3.4	1	100.00	2.5	even	ok
117.0545	1	C5H9O3	117.0546	1.2	4.4	1	100.00	1.5	even	ok

Fig. S3 LC-MC analysis of levulinic acid

5-Hydroxyméthylfurfural (2):



Meas. m/z	#	Ion Formula	m/z	err [ppm]	mSigma	# mSigma	Score	rdb	e⁻ Conf	N-Rule
109.0283	1	C6H5O2	109.0284	1.4	0.5	1	100.00	4.5	even	ok
127.0388	1	C6H7O3	127.0390	1.7	1.2	1	100.00	3.5	even	ok

Fig. S4 LC-MC analysis of 5-hydroxyméthylfurfural

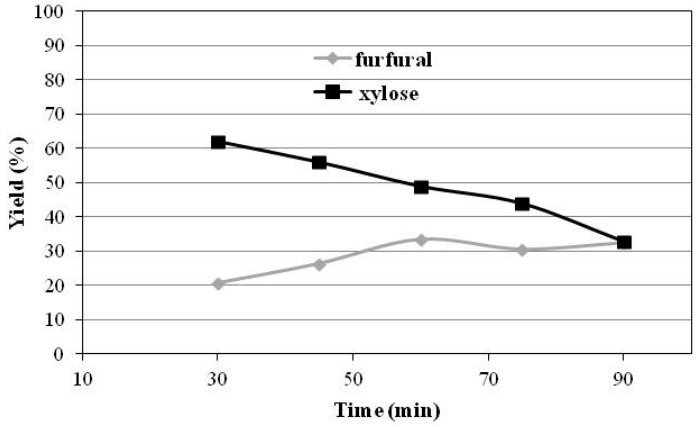


Fig. S5 Tandem hydrolysis/dehydration of xylan originated from beechwooch in the presence of an aqueous solution BHC. Xylan and BHC content of 10 wt% and 20 wt% respectively, 150°C

<u>^{1}H NMR spectra :</u> ^{1}HNMR are recorded on a 400 MHz Bruker Advance DPX spectrometer using DMSO as solvent.

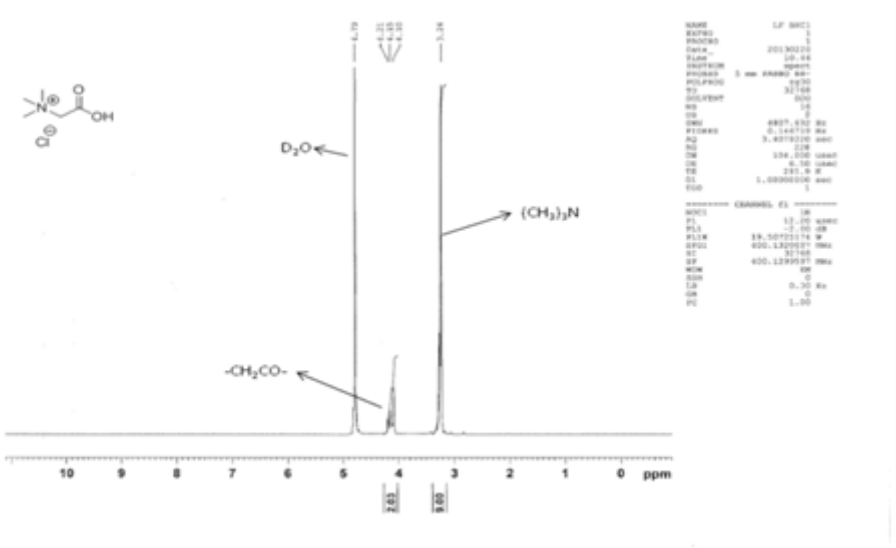


Fig. S6 ^{1}H NMR spectra of BHC recovered after the 6th cycle of recycling

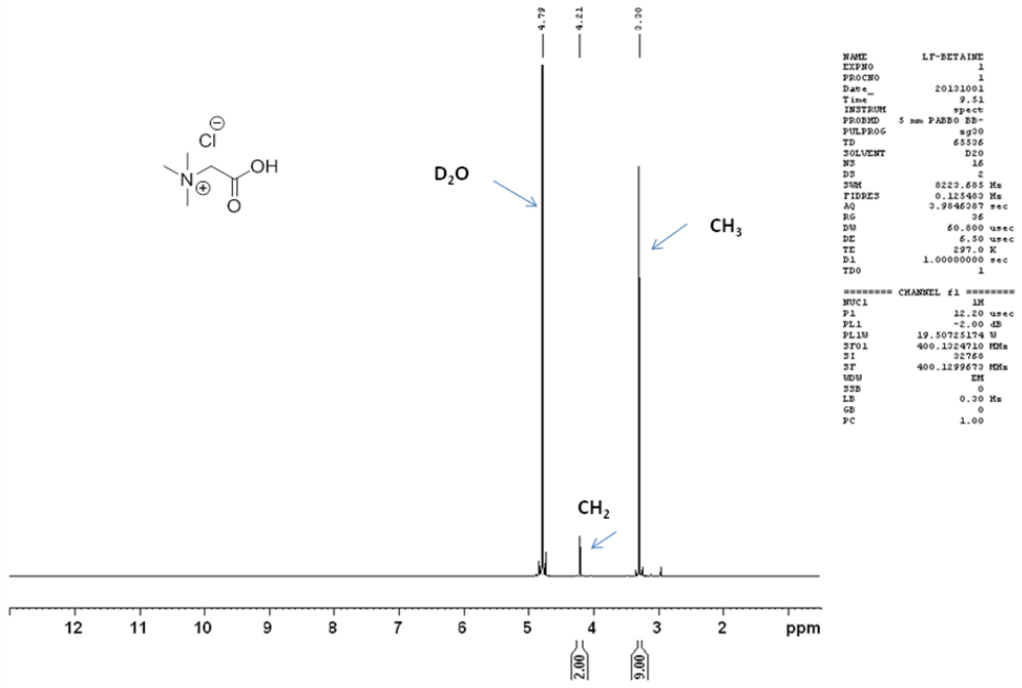


Fig. S7: ^{1}H NMR spectra of BHC recovered after conversion of wheat straw

Table S1 Hydrolysis of microcrystalline cellulose (AVICEL PH 200) in the presence of BHC[a]

Cellulose (wt%)	Yield to levulinic acid(%)
1,3	64
1,3[b]	65
1,3[c]	75
5	60
10	60
20	53
30	48

[a] BHC, 40wt%, MW 250 W, 10 mL of water, 200°C, t = 1 h, [b] T = 180°C t = 2 h, [c] cotton, T= 180°C, t= 2h

Fig. S8: Picture of the reactor used