Electronic Supplementary Material (ESI) for Green Chemistry

Supporting Information

Microwave Assisted Conversion of Carbohydrates and Biopolymers to 5-Hydroxymethylfurfural with Aluminum Chloride Catalyst in Water

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Table S1 Optimization of reaction time for dehydration of 30 wt% of fructose catalyzed by $AlCl_3$ in biphasic water-MIBK (1/4 volume ratio).

Entry	*t* (min)	HMF Yield (%)
1	30	27.7
2	60	32.8
3	75	33.3
4	90	33.9

Table S2 Results of fructose dehydration to HMF catalyzed by $AlCl_3$ in different reaction time.

Entry	Fructose (wt%)	$AlCl_3$ (mol%)	*t* (min)	HMF Yield (%)
1.	5	50	0.5	47.8
2.	5	50	2	52.3
3.	5	50	5	53.9
4.	5	50	10	54.6
5.	5	50	15	55.1
6.	5	50	20	55.7

Solvent = water (2 mL), *T* = 120 °C, MW

Figure S1 Representative ^{1}H NMR spectrum of HMF product obtained from dehydration reaction of fructose with $AlCl_3$ in water. This spectrum recorded in $CDCl_3$ at room temperature for determining HMF yield using mesitylene as an internal standard.

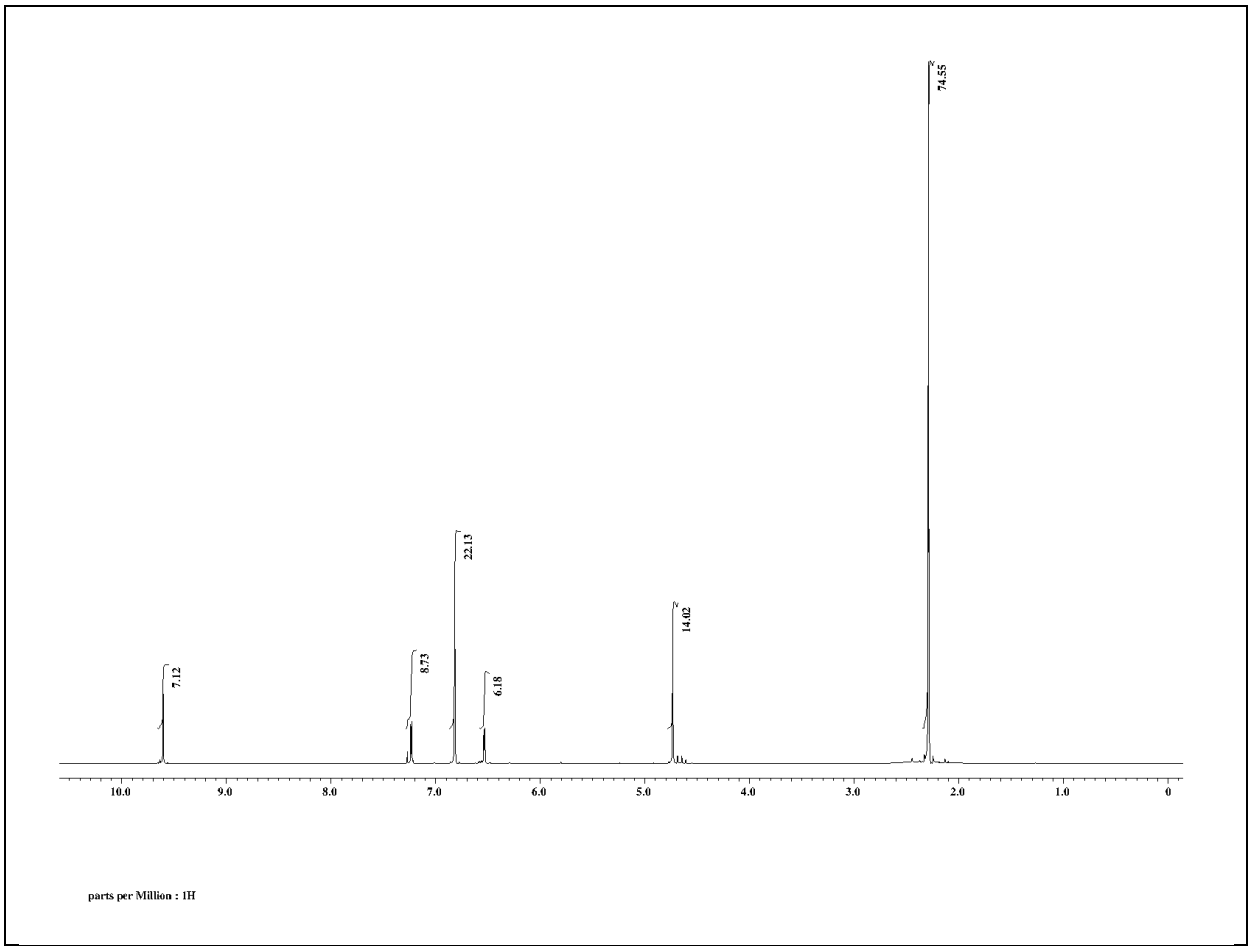

Figure S2 Representative UV-Vis spectrum of HMF product solution obtained from dehydration reaction of fructose with $AlCl_3$ in water.

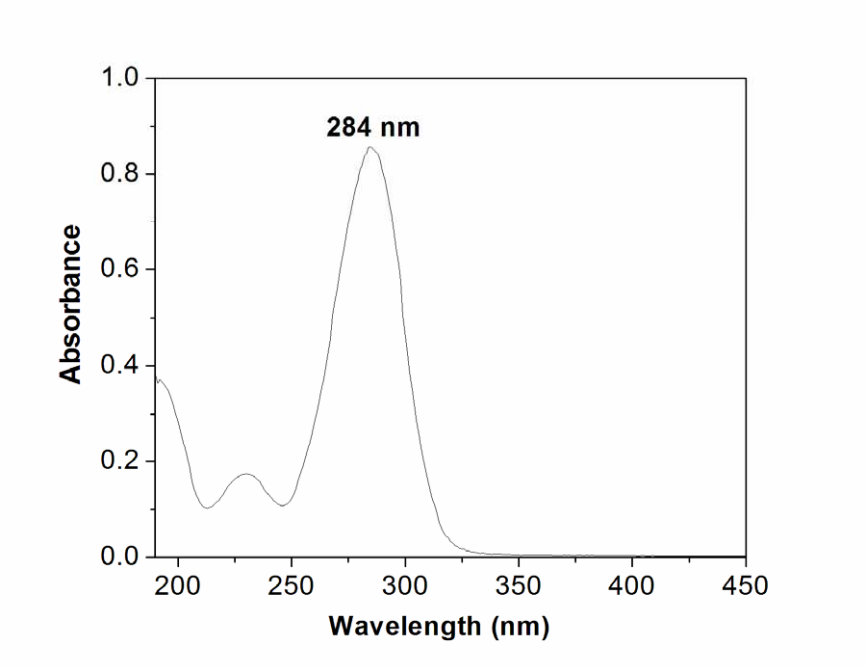

Figure S3 Representative ^{1}H NMR (in $CDCl_3$) spectrum of the reaction product collected from MIBK phase of fructose (30 wt%) dehydration reaction with 50 mol% $AlCl_3$ catalyst in biphasic water-MIBK solvent under microwave heating at T = 120 °C for 5 min.

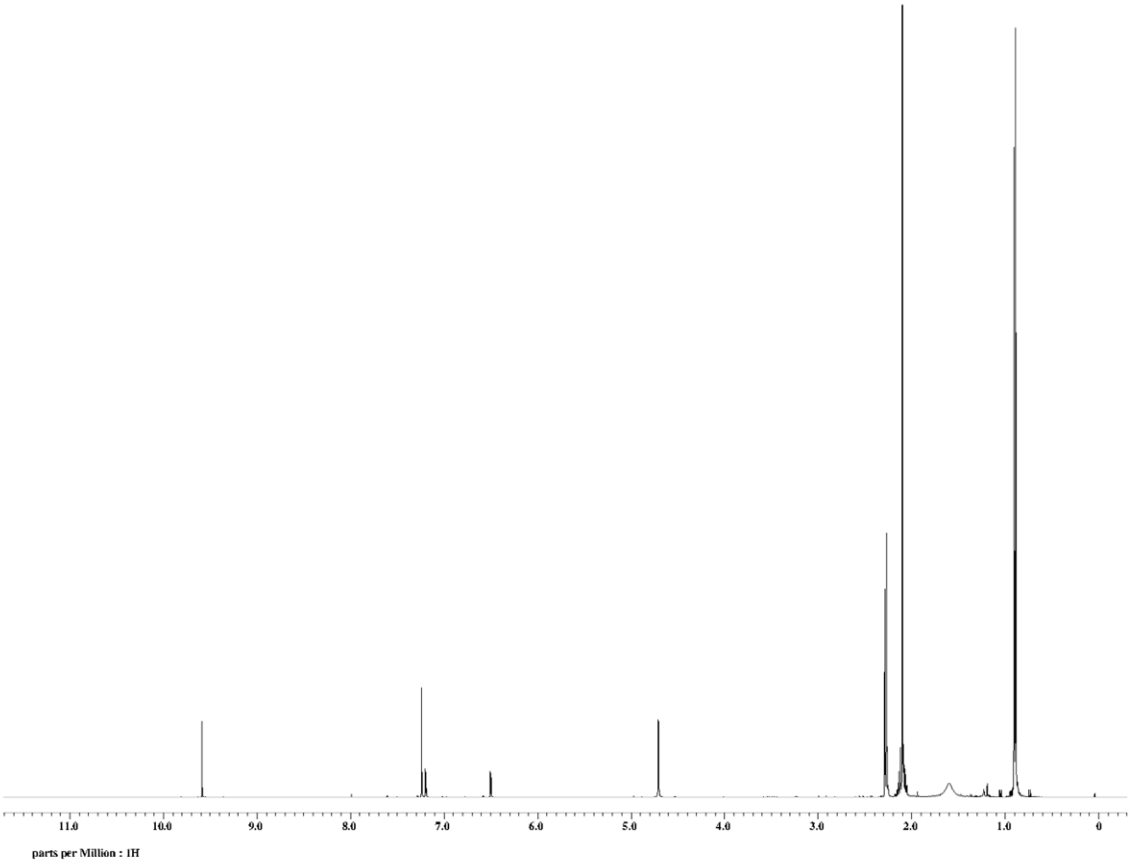

Figure S4 Representative ^{1}H NMR (DMSO-d_6) spectrum of the insoluble brown material obtained in fructose (30 wt%) dehydration with $AlCl_3$ in water under microwave heating at 120 °C for 5 min.

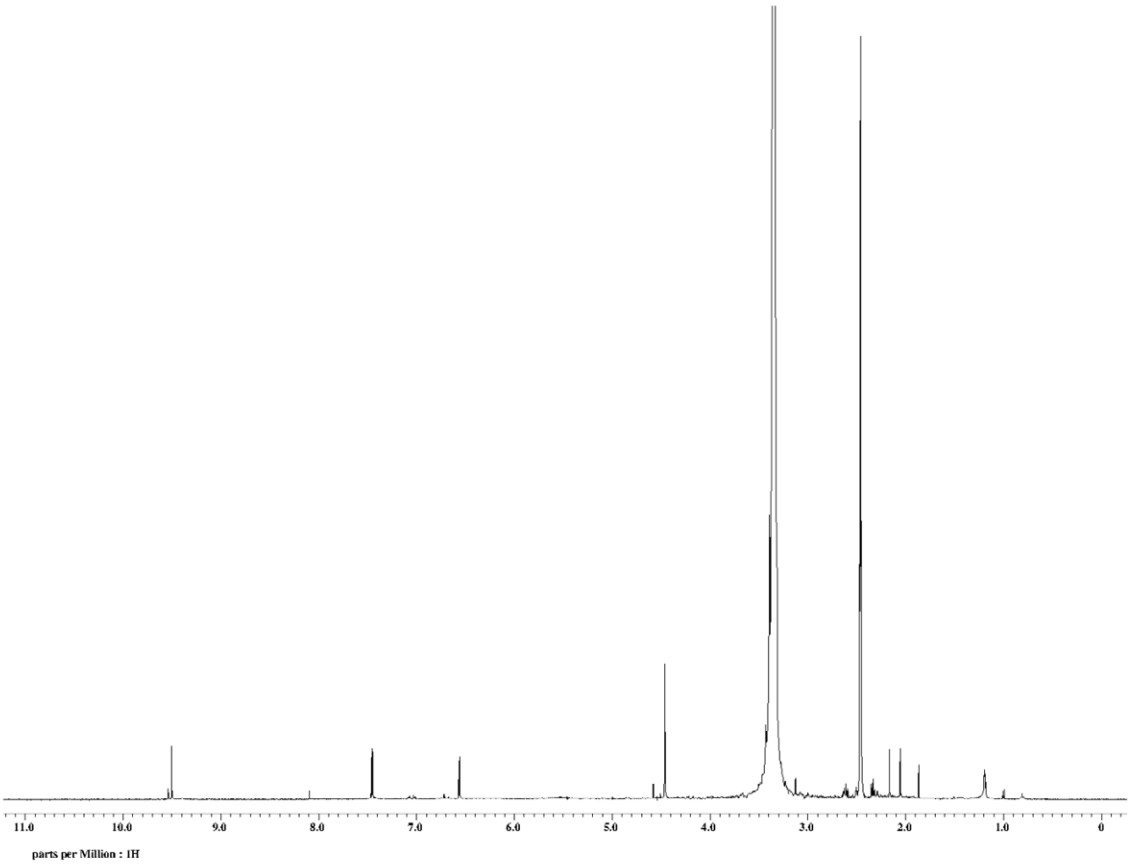

Figure S5 Representative ^{1}H NMR spectrum for a mixture of glucose and $AlCl_3$ in DMSO-d_6 at room temperature to 100 °C; (a) ^{1}H NMR spectrum of glucose (25 mg) at room temperature in DMSO-d_6. (b) ^{1}H NMR spectrum of glucose (25 mg) + $AlCl_3$ (9.3 mg) (2:1 molar ratio) at room temperature displaying broad $H_{O–H}$ signals of glucose due to the interaction of Cl of $AlCl_3$ with OH of glucose in DMSO-d_6. (c) ^{1}H NMR spectrum of glucose at 100 °C heated for 40 min. (d) ^{1}H NMR spectrum of glucose + $AlCl_3$ (2:1 molar ratio) at 100 °C heated for 40 min. Data presented for δ4 to 5 ppm with full spectrums (δ1 to 10 ppm) as inset.

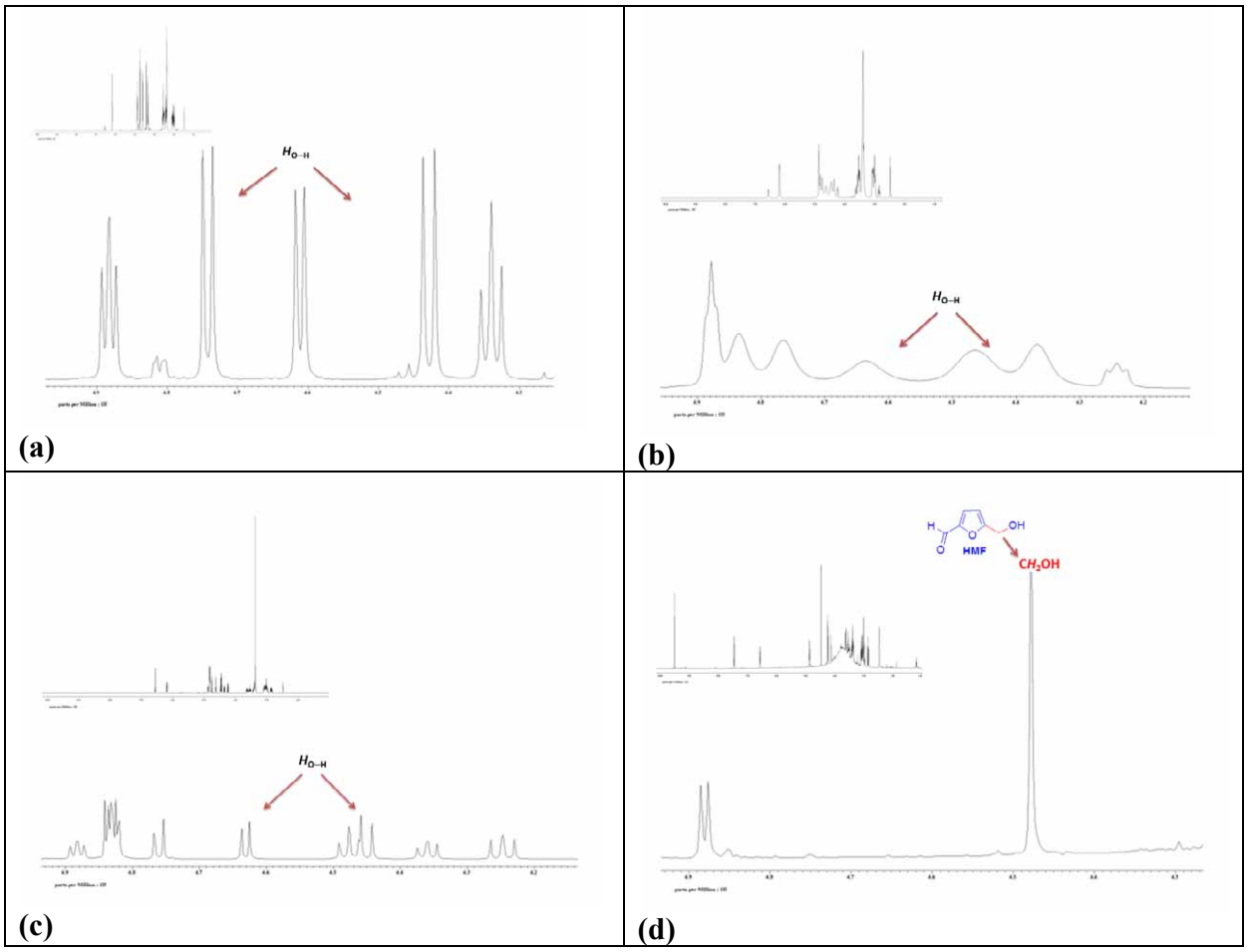

Figure S6 Representative ^{1}H NMR spectrum for a mixture of glucose and $SnCl_4$ in DMSO-d_6 at room temperature to 120 °C; (a) ^{1}H NMR spectrum of glucose (25 mg) at room temperature in DMSO-d_6. (b) ^{1}H NMR spectrum of glucose (25 mg) + $SnCl_4$ (9 mg) (4:1 molar ratio) at room temperature displaying disappearance of H_{O-H} signals of glucose due to the interaction of $SnCl_4$ with OH of glucose in DMSO-d_6. (c) ^{1}H NMR spectrum of glucose + $SnCl_4$ (2:1 molar ratio) at 80 °C heated for 40 min. (d) ^{1}H NMR spectrum of glucose + $SnCl_4$ (2:1 molar ratio) at 120 °C heated for 30 min. Full spectrums (δ0 to 11 ppm) for each steps presented as inset.

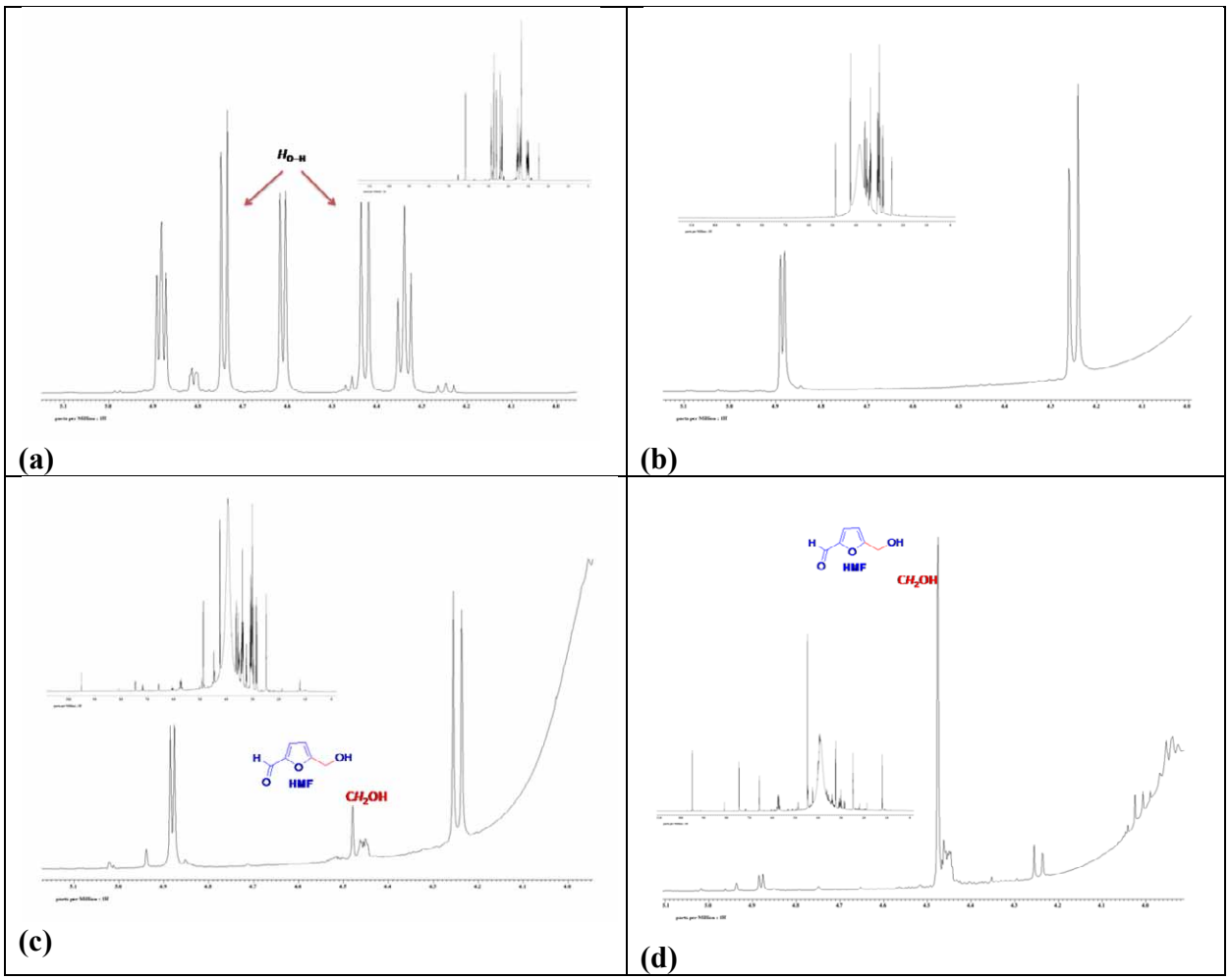

Figure S7 Representative ^{1}H NMR spectrum for a mixture of glucose and $Yb(OTf)_3$ in DMSO-d_6 at room temperature to 120 °C; (a) ^{1}H NMR spectrum of glucose (25 mg) at room temperature in DMSO-d_6. (b) ^{1}H NMR spectrum of glucose (25 mg) + $Yb(OTf)_3$ (9.3 mg) (2:1 molar ratio) at 120 °C heated for 40 min in DMSO-d_6. Full spectrums (δ 0 to 11 ppm) for each steps presented as inset.