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Supporting Information

Inulin Dehydration to 5-HMF in Deep Eutectic Solvents Catalyzed by Acidic Ionic Liquids Under Mild Conditions

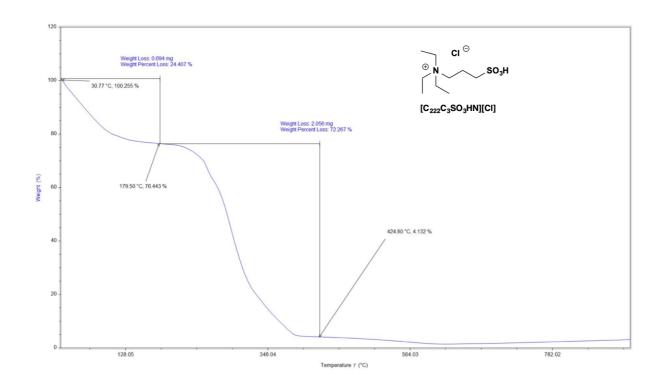
Salvatore Marullo, Giovanna Raia, Josh J. Bailey, H. Q. Nimal Gunaratne,* and Francesca D'Anna*

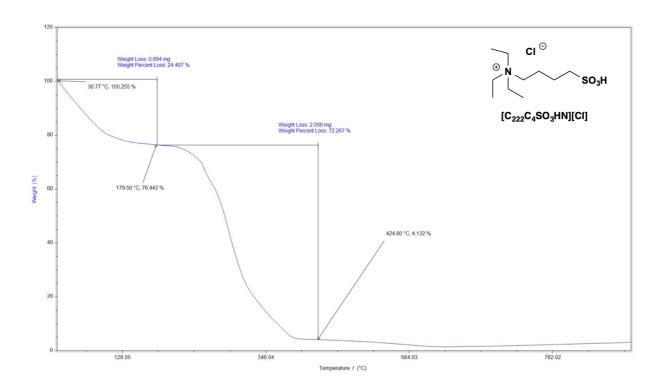
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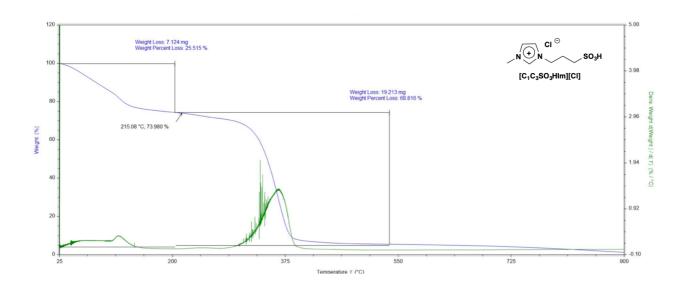
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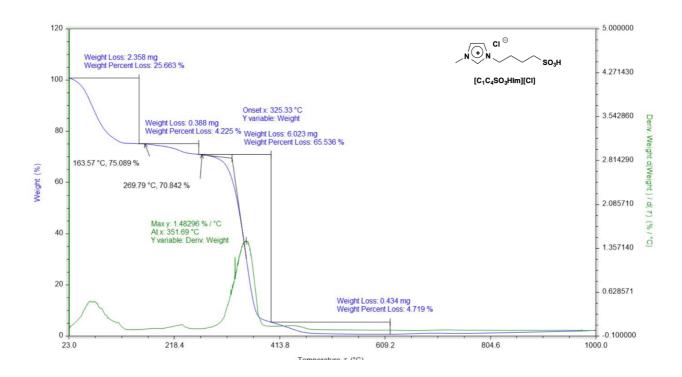
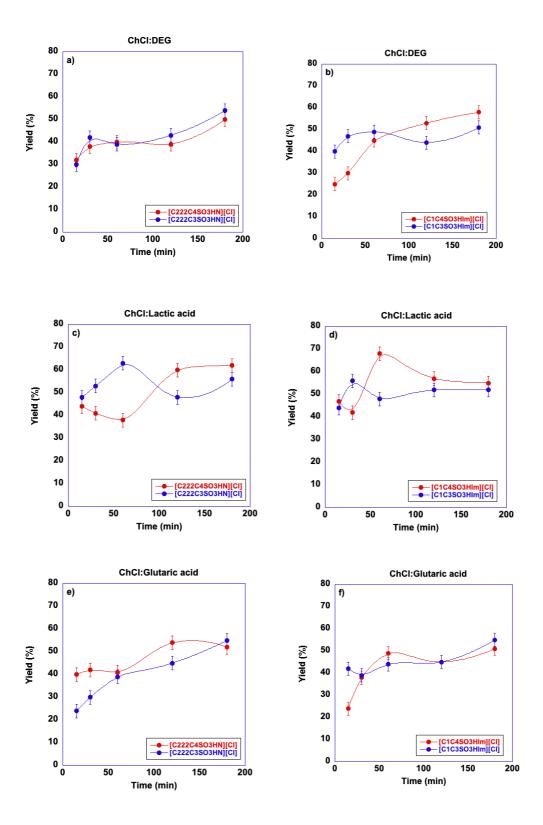
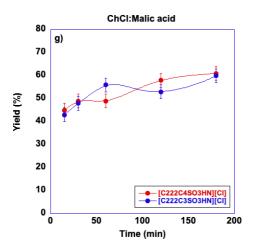


Figure S1. TGA traces for the catalysts empolyed.





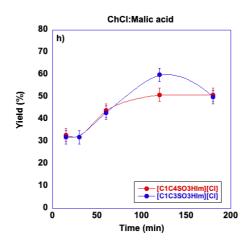
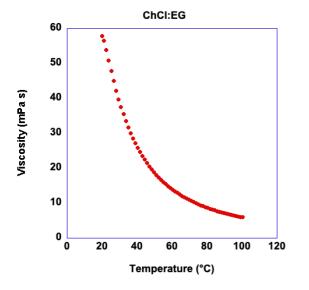
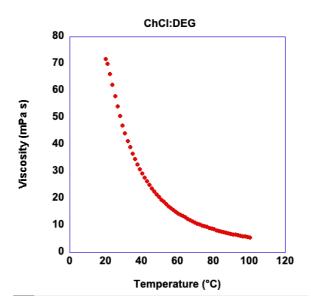
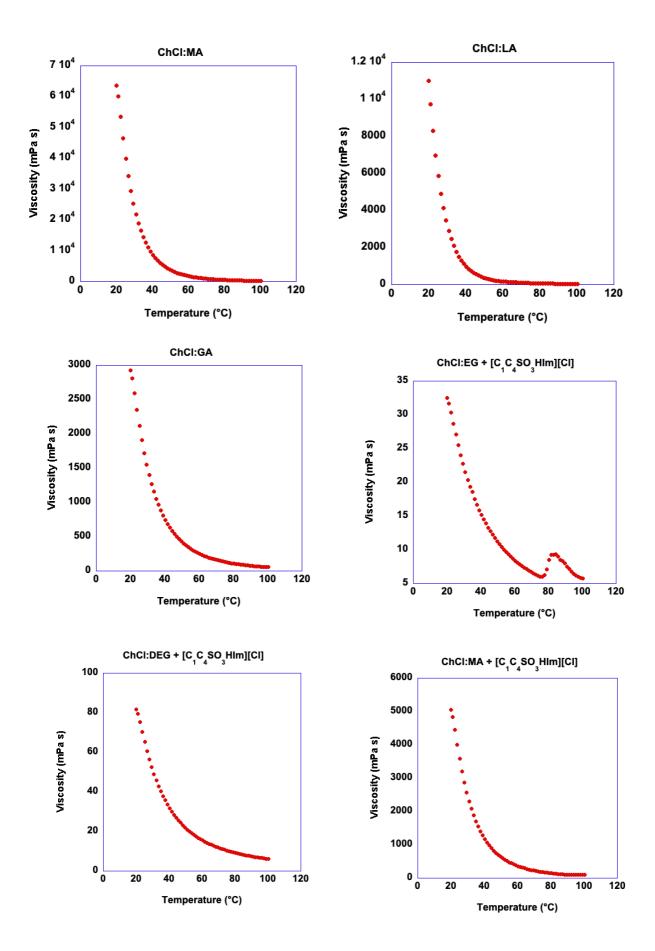


Figure S2. Plots of yield in 5-HMF from inulin at 80 °C, in different DES and in the presence of different acidic TSILs as catalysts.







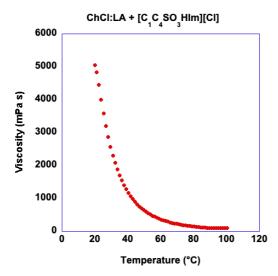


Figure S3. Plots temperature-dependent viscosity measurements for pure DES and solutions of the catalyst in solution of the DES considered.

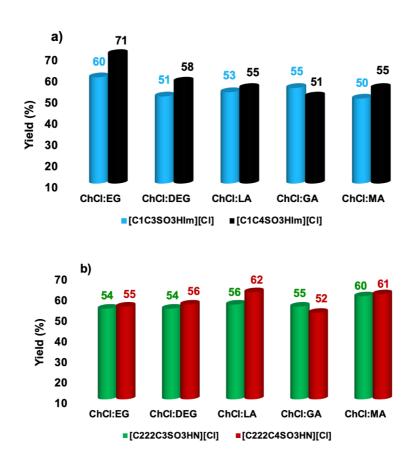
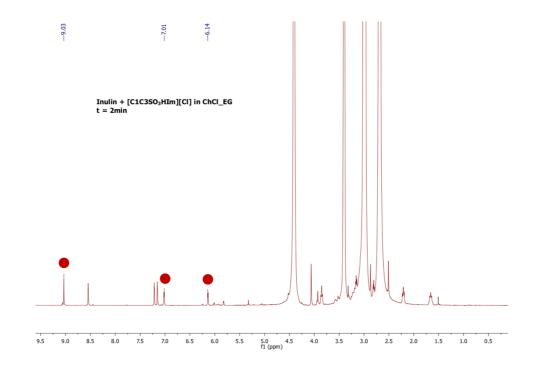
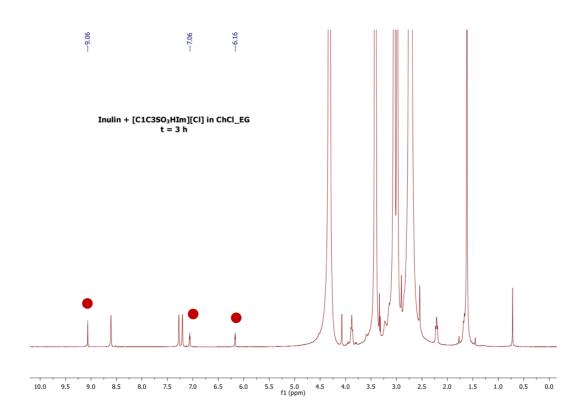
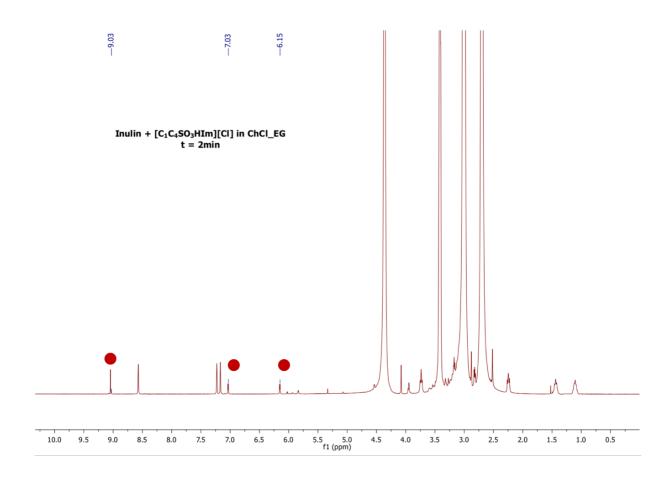
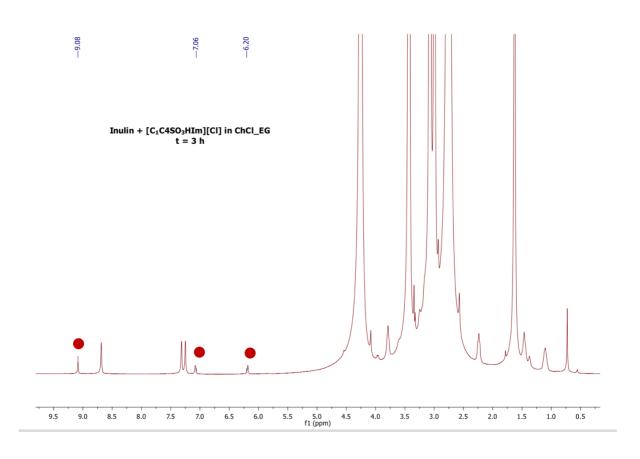


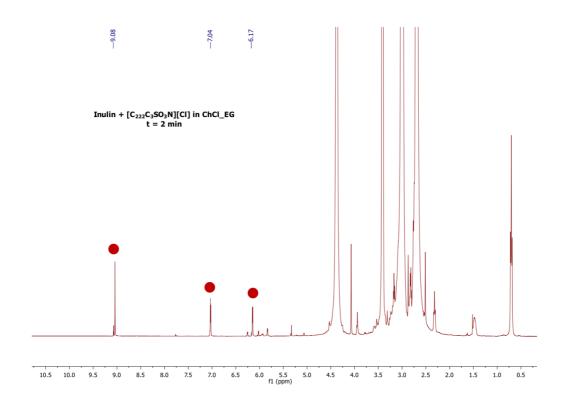
Figure S4. Plots of yields in 5-HMF obtained at 80 °C, 3h, in the presence of a) imidazolium- and b) ammonium-based TSILs, differing for the spacer length.

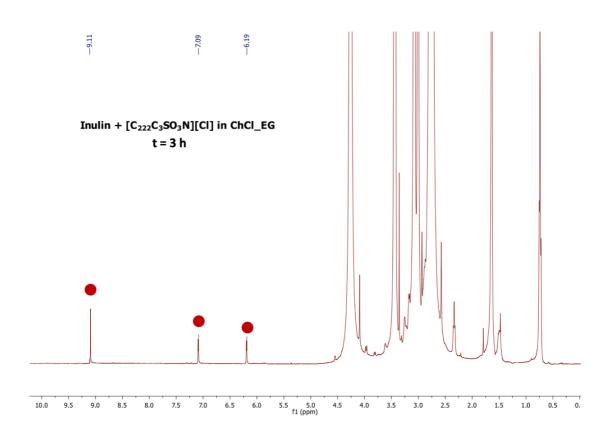












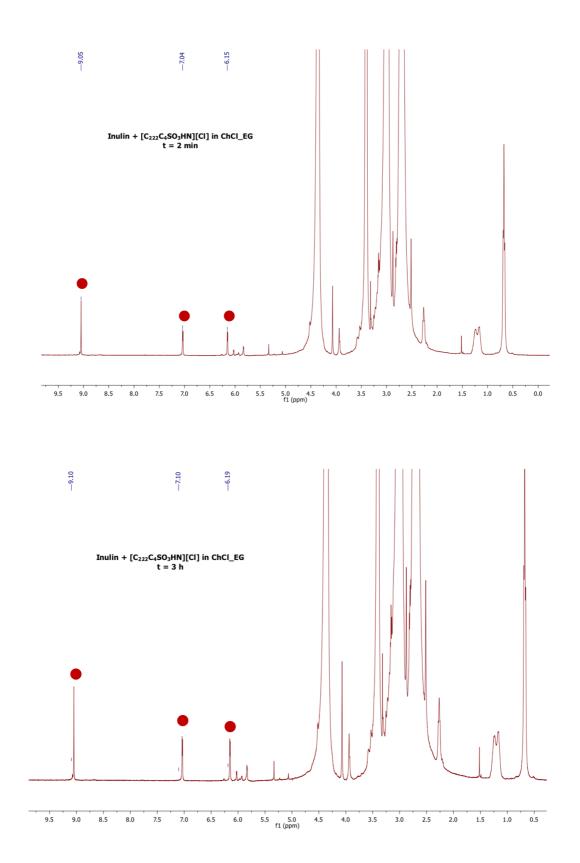


Figure S5. 1 H NMR spectra in D6-DMSO of the reaction mixtures after 2 min and 3 h in ChCl:EG. Marked signals refer to protons of 5-HMF

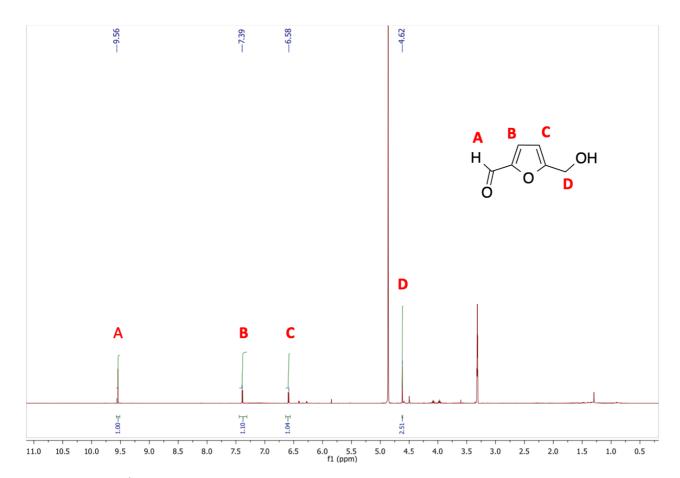


Figure S6. ¹H NMR spectrum in CD₃OD of 5-HMF isolated.

Table S1. Yields in 5-HMF for the dehydration of inulin at 80 °C, in ChCl:EG in the presence of acidic TSILs.^a

in ChCl:EG in the presence of	of actuic 151Ls.		
[C ₂₂₂ C ₃ SO ₃ HN][Cl]			
Time (min)	Yield in 5-HMF (%)		
15	37		
30	43		
60	47		
120	44		
180	54		
[C ₂₂₂ C ₄ SO ₃ HN][Cl]			
15	41		
30	39		
60	50		
120	54		
180	55		
[C ₁ C ₃ SO ₃ HIm][Cl]			
15	49		
30	44		
60	57		
120	60		
180	60		
[C ₁ C ₄ SO ₃ HIm][Cl]			
15	37		
30	43		
60	47		
120	50		
180	71		

[a] Yields are reproducible within \pm 4%.

Table S2. Yields in 5-HMF for the dehydration of inulin at 80 °C, in ChCl:DEG in the presence of acidic TSILs.^a

in Chel.DEG in the pr	esence of acidic 151Ls."		
	$[C_{222}C_3SO_3HN][Cl]$		
Time (min)	Yield in 5-HMF (%)		
15	30		
30	42		
60	30		
120	43		
180	54		
	$[C_{222}C_4SO_3HN][Cl]$		
15	32		
30	38		
60	40		
120	39		
180	50		
	IC.C.SO.HImIICII		
15	[C ₁ C ₃ SO ₃ HIm][Cl] 40		
30	47		
	49		
60	49		
120	51		
180	21		
	[C ₁ C ₄ SO ₃ HIm][Cl]		
15	25		
30	30		
60	45		
120	53		
180	58		

[a] Yields are reproducible within $\pm 4\%$.

Table S3. Yields in 5-HMF for the dehydration of inulin at 80 °C, in ChCl:LA in the presence of acidic TSILs.^a

in chei.En in the pres	selice of acture 151Ls.		
[C ₂₂₂ C ₃ SO ₃ HN][Cl]			
Time (min)	Yield in 5-HMF (%)		
15	48		
30	53		
60	63		
120	48		
180	56		
[C ₂₂₂ C ₄ SO ₃ HN][Cl]			
15	44		
30	41		
60	38		
120	60		
180	62		
	$[C_1C_3SO_3HIm][Cl]$		
15	44		
30	56		
60	48		
120	52		
180	52		
[C ₁ C ₄ SO ₃ HIm][Cl]			
15	47		
30	42		
60	68		
120	57		
180	55		

[[]a] Yields are reproducible within \pm 4%.

Table S4. Yields in 5-HMF for the dehydration of inulin at 80 °C, in ChCl:GA in the presence of acidic TSILs.^a

-	in ChCi.GA in the presence of acidic TSILs.		
	C ₃ SO ₃ HN][Cl]		
Time (min)	Yield in 5-HMF (%)		
15	24		
30	30		
60	39		
120	45		
180	55		
[C222C	C4SO3HN [Cl]		
15	40		
30	42		
60	41		
120	54		
180	52		
IC C.	SO HIMICH		
	SO ₃ HIm][Cl]		
15 30	42		
60	39 44		
120	45 55		
180	55		
[C ₁ C ₄ SO ₃ HIm][Cl]			
15	24		
30	38		
60	49		
120	45		
180	51		

[[]a] Yields are reproducible within \pm 4%.

Table S5. Yields in 5-HMF for the dehydration of inulin at 80 °C, in ChCl:MA in the presence of acidic TSILs.^a

in ChCi:MA in the presence of acidic 151Ls."			
$[C_{222}C_3SO_3HN][Cl]$			
Time (min)	Yield in 5-HMF (%)		
15	43		
30	48		
60	56		
120	53		
180	60		
[C ₂₂₂ C ₄ SO ₃ HN][Cl]			
15	45		
30	49		
60	49		
120	58		
180	61		
	$[C_1C_3SO_3HIm][Cl]$		
15	32		
30	32		
60	43		
120	60		
180	50		
[C ₁ C ₄ SO ₃ HIm][Cl]			
15	36		
30	34		
60	50		
120	56		
180	55		

[a] Yields are reproducible within \pm 4%.