

Supporting Information

Microwave assisted organocatalytic synthesis of 5-hydroxymethyl furfural in a monophasic green solvent system

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1 Purification procedure

1.1 Purification of HMF by silica column

Solvent distilled crude HMF was purified before structural characterization by silica column chromatography. Silica gel (60-120 mesh), in about three times weight of HMF, was poured in the solution of crude HMF in 5 volumes of ethyl acetate, and ethyl acetate removed by vacuum distillation. The silica gel slurry was dried at 50°C in a vacuum oven. The silica adsorbed HMF solid was loaded on top of a 30cm long 10mm diameter silica gel column. The column was eluted by 50:50 hexane-ethyl acetate mixture and the eluted fractions analyzed by TLC. Fractions containing HMF were pooled and distilled under vacuum. The resulting faint to dark yellow color oil (0.625 gm, 89.29 % yield) was used for structural characterization as HMF.

Material Balance

	Fructose	HMF	Molar Step Yield, %	Overall Molar Yield, %	Purity, w/w%
Initial	1.0g	--	--		--
Crude reaction Mixture	0.0g	0.64g	91.0		
Ethyl Acetate Extract	0.0g	0.63g	98.0		
Column Eluate	0.0g	0.625g	98.0		
Product after drying	0.0g	0.625g	100.0%	88%	>99%

2 HPLC analysis of HMF

2.2 HPLC chromatogram of reaction mass

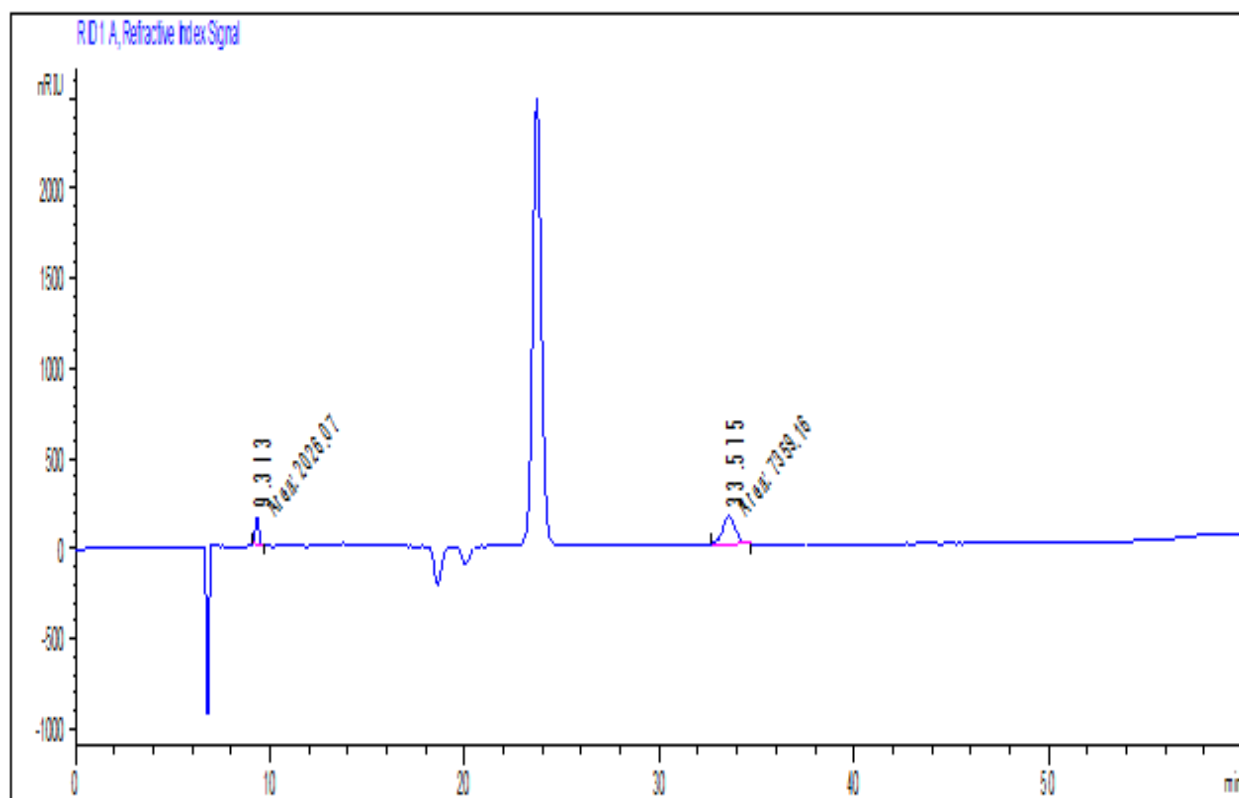


Fig S1: HPLC chromatogram of the IPA reaction mass after 90s.

2.1 HPLC chromatogram of HMF

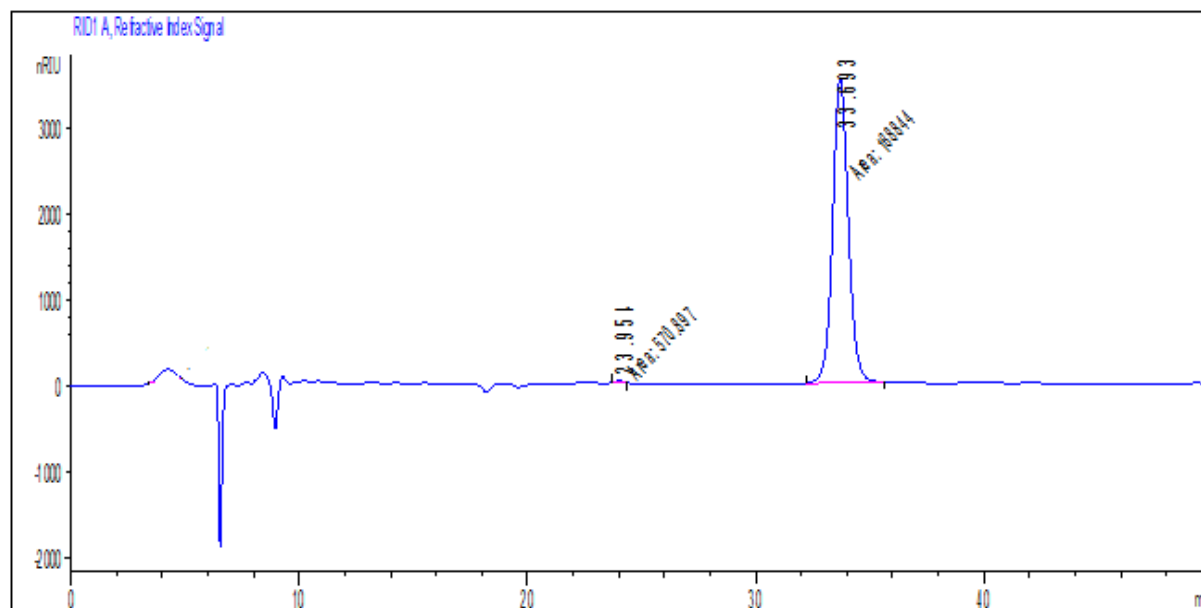


Fig S2.HPLC chromatogram of HMF before purification (after distillation of solvent)

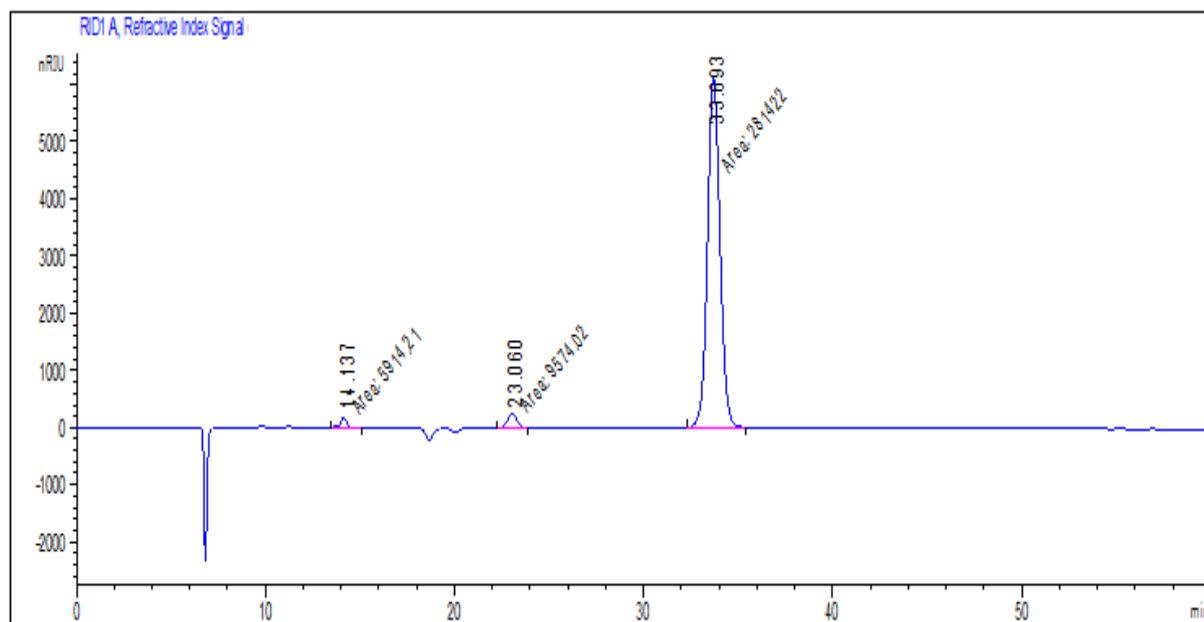


Fig S3.HPLC chromatogram of HMF after purification

3. Characterization of HMF

3.1. ATR- FTIR analysis of HMF

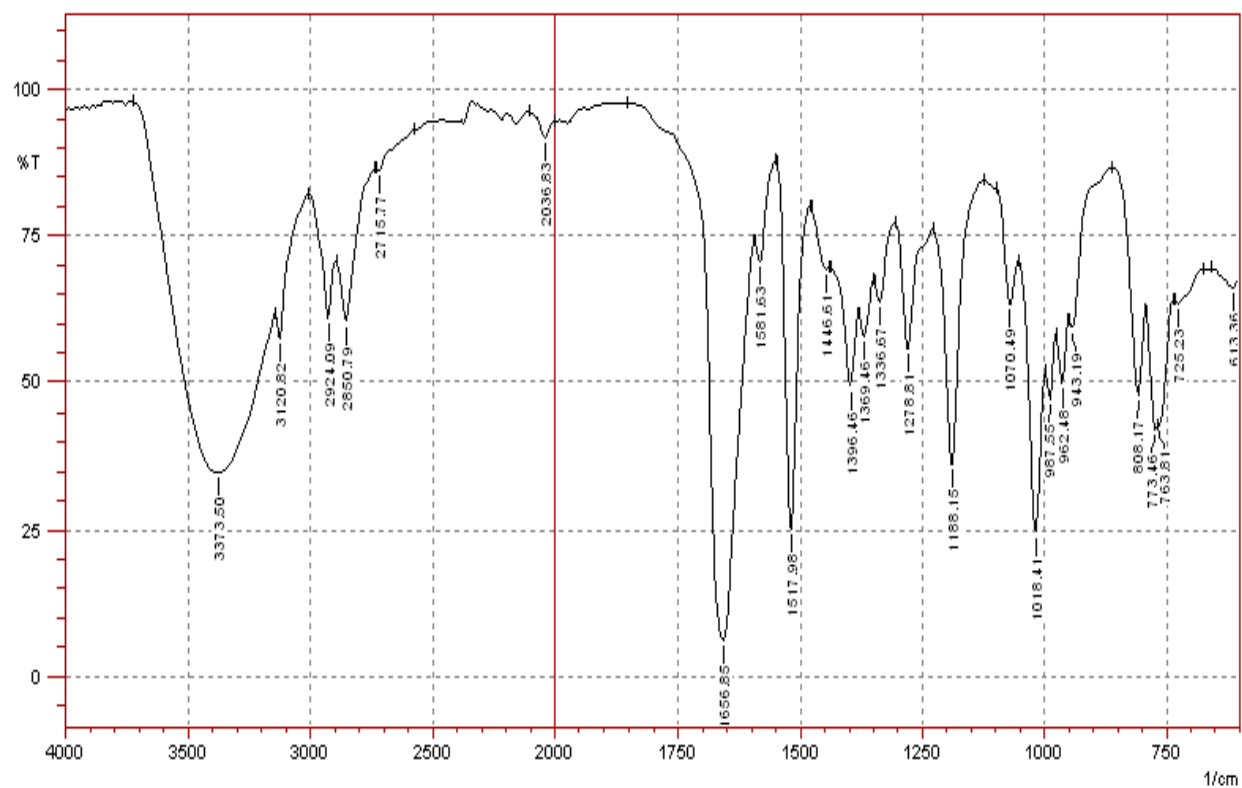


Fig S4: ATR –FTIR of HMF

3.2. GC-MS analysis of HMF

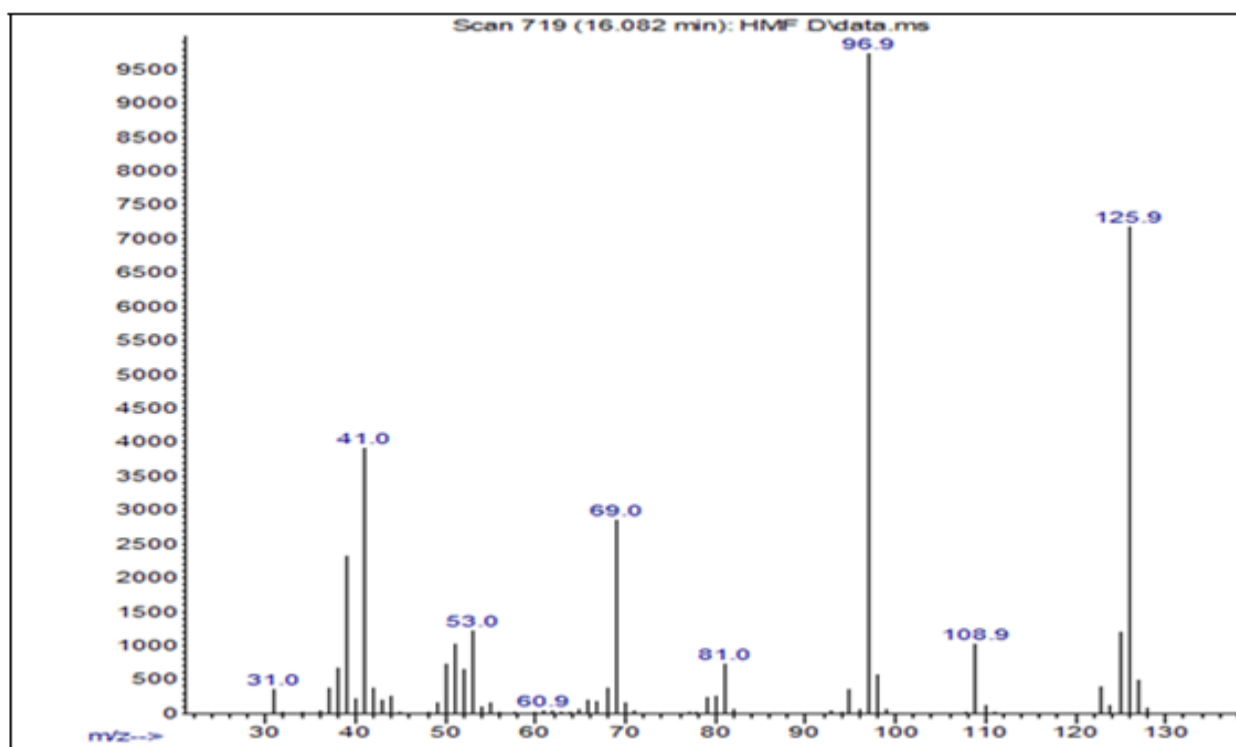


Fig S5: GC-MS mass chromatogram of purified HMF

3.3. LC-MS and MS-MS analysis of HMF

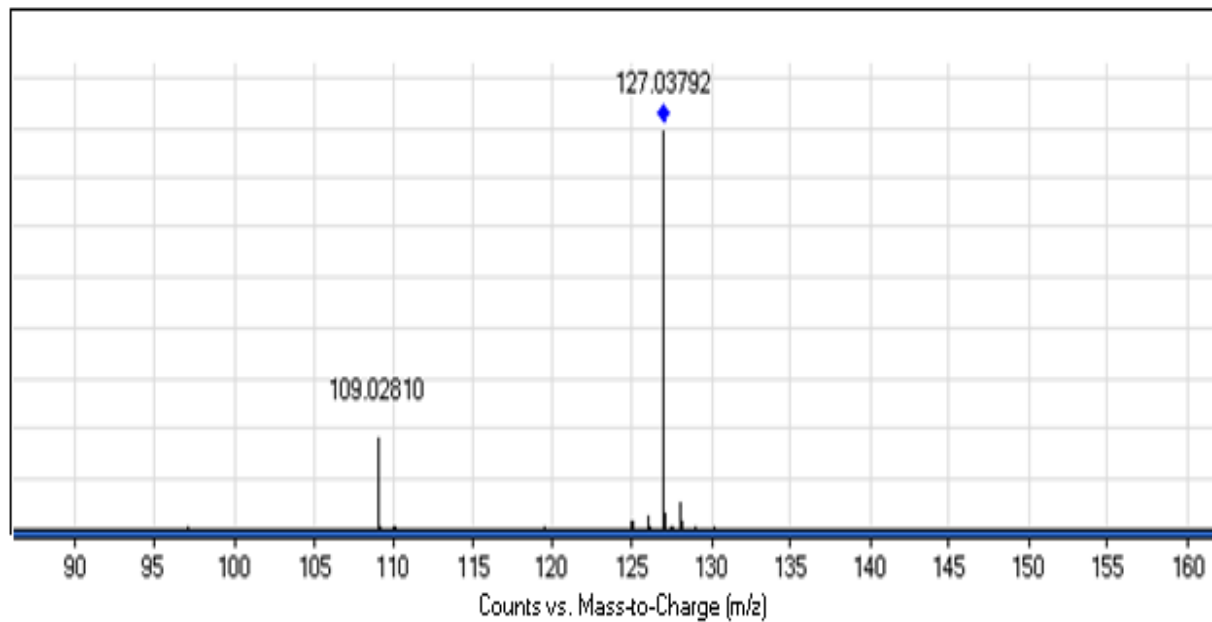


Fig S6: LC-MS mass chromatogram of purified HMF

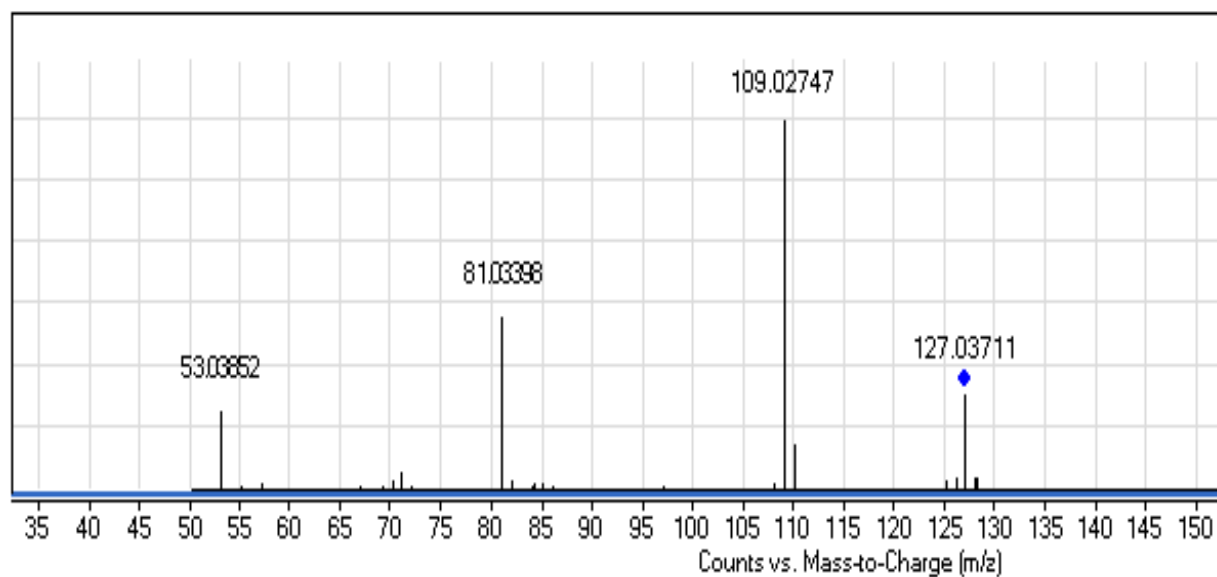


Fig S7: MS-MS mass chromatogram of purified HMF

3.4. ^1H NMR analysis of HMF

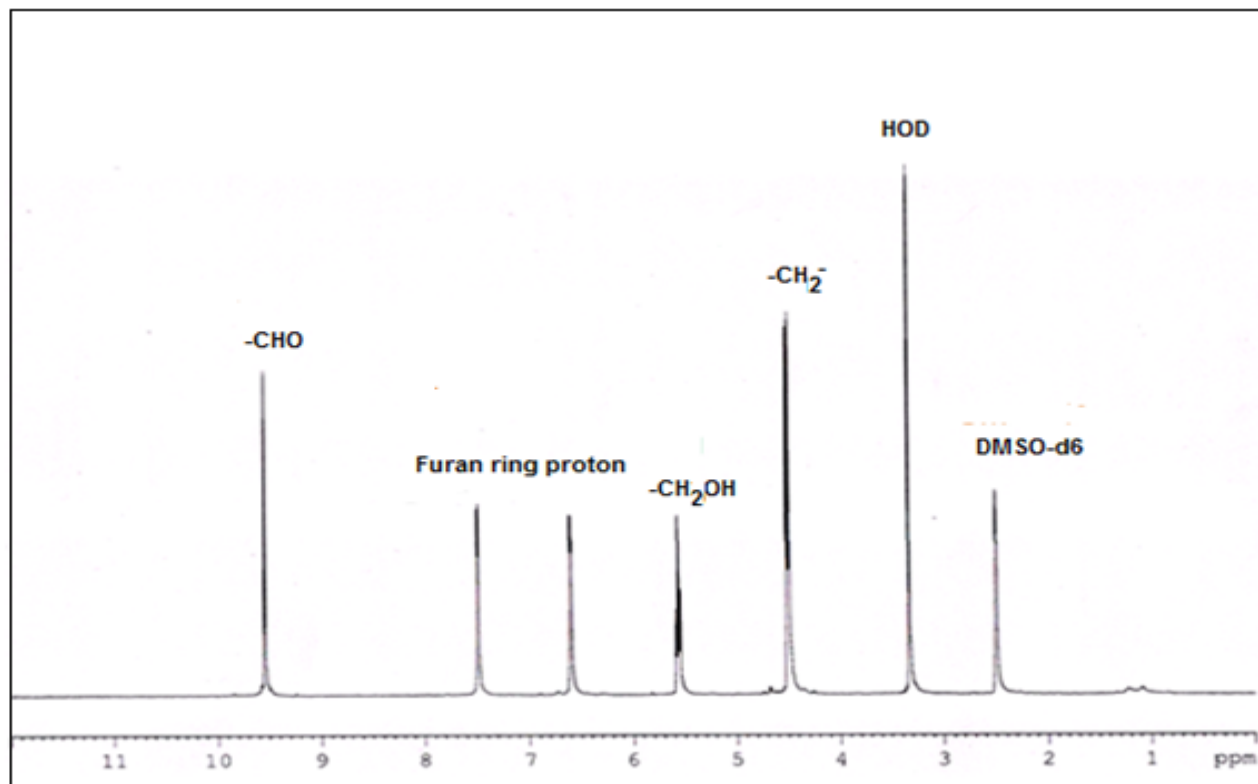


Fig S8: ^1H NMR of purified HMF