Conversion of glucose into 5-hydroxymethylfurfural catalyzed by chromium(III) schiff base complexes and acidic ionic liquids immobilized on mesoporous silica

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Supplementary Information

Ligand Analysis

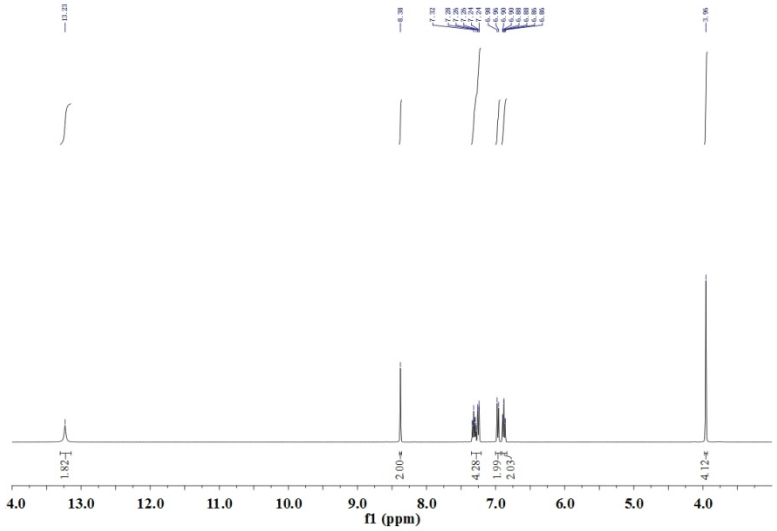

Figure S1: ^{1}H NMR spectrum of Salen.

^{1}H NMR ($CDC1_3$, 400 MHz): δ ppm 13.23 (s, 2H), 8.38 (s, 2H), 7.35-7.21 (m, 5H), 6.97 (d, 2H, J = 8.3 Hz), 6.88 (td, 2H, J=7.6, 0.9 Hz), 3.96 (s, 4H).

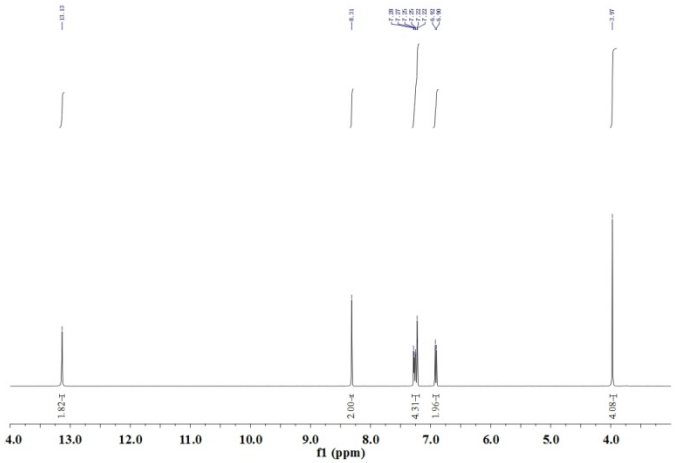

Figure S2: ^{1}H NMR spectrum of Salen-Cl.

^{1}H NMR ($CDC1_3$, 400 MHz): δ ppm 13.13 (s, 2H), 8.31 (s, 2H), 7.31-7.19 (m, 5H), 6.91 (d, 2H, *J*=8.7 Hz), 3.97 (s, 4H).

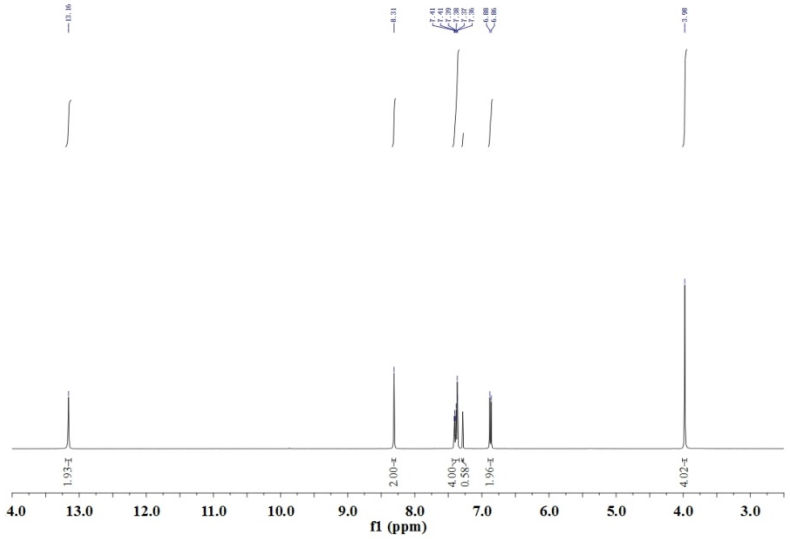

Figure S3: ^{1}H NMR spectrum of Salen-Br.

^{1}H NMR ($CDC1_3$, 400 MHz): δ ppm 13.16 (s, 2H), 8.31 (s, 2H), 7.44-7.34 (m, 5H), 6.87 (d, 2H, J=8.7 Hz), 3.98 (s, 4H).

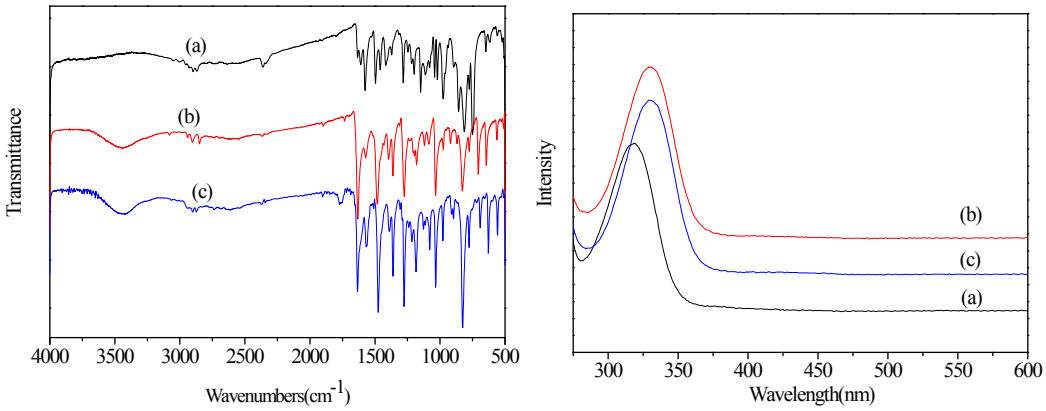

Figure S4: FT-IR (KBr disc) and UV-Vis spectrum of Salen(a), Salen-Cl(b), Salen-Br(c).

Salen(a), FT-IR(KBr, cm^{-1}): 1633, 1606, 1575, 1460, 1416, 1282, 1020, 813, 749. UV-Vis spectrum in CH_2Cl_2 (λ_{max}, nm): 317.

Salen-Cl(b), FT-IR(KBr, cm^{-1}): 3441, 1632, 1482, 1274, 1034, 828, 705, 645. UV-Vis spectrum in CH_2Cl_2 (λ_{max}, nm): 330.

Salen-Br(c), FT-IR(KBr, cm^{-1}): 3438, 1634, 1474, 1276, 1033, 826, 776, 691. UV-Vis spectrum in CH_2Cl_2 (λ_{max}, nm): 332.

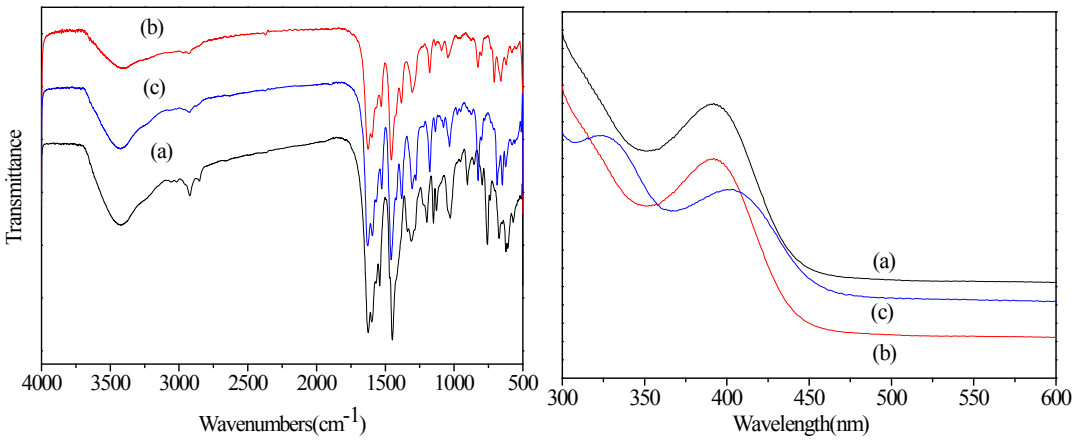

Figure S4: FT-IR (KBr disc) and UV-Vis spectrum of Cr-Salen(a), Cr(Salen-Cl)(b), Cr(Salen-Br)(c).

Cr(Salen), FT-IR(KBr, cm^{-1}): 3418, 2921, 1623, 1594, 1449, 1311, 1029, 757, 672, 620, 569. UV-Vis spectrum in CH_2Cl_2 (λ_{max}, nm): 391.

Cr(Salen-Cl), FT-IR(KBr, cm^{-1}): 3402, 2929, 1625, 1602, 1456, 1383, 1304, 1177, 826, 708, 660. UV-Vis spectrum in CH_2Cl_2 (λ_{max}, nm): 401.

Cr(Salen-Br), FT-IR(KBr, cm^{-1}): 3426, 2921, 1628, 1594, 1457, 1380,1304, 1177, 1033, 825, 687, 649. UV-Vis spectrum in CH_2Cl_2 (λ_{max}, nm): 402.

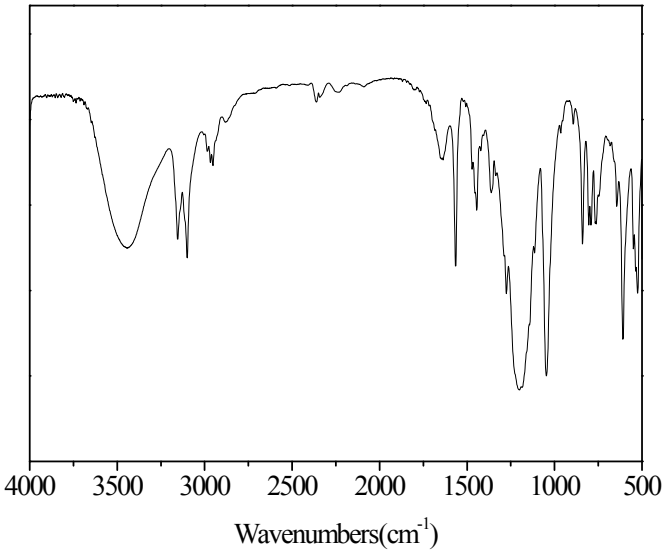

Figure S5: FT-IR (KBr disc) spectrum of [CPTES-IM-SO_3H][HSO_4]

FT-IR(KBr, cm^{-1}): 3443, 3154, 3100, 2952, 1565, 1444, 1361, 1275, 1184, 1046, 840, 608.

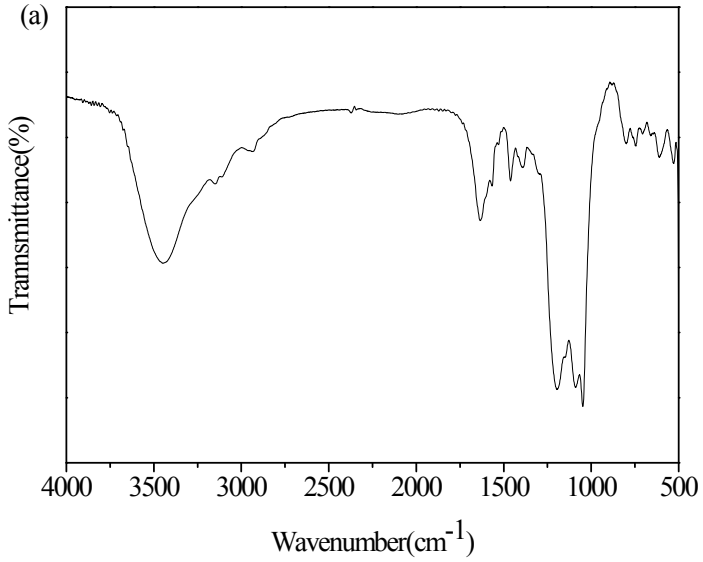

Figure S6 (a)FT-IR spectra of Cr(Salen)-IM-Cl-MCM-41

FT-IR(KBr, cm^{-1}): 3441, 3155, 2931, 1625, 1545, 1460, 1388, 1192, 1089, 1048, 803, 740, 601, 524.

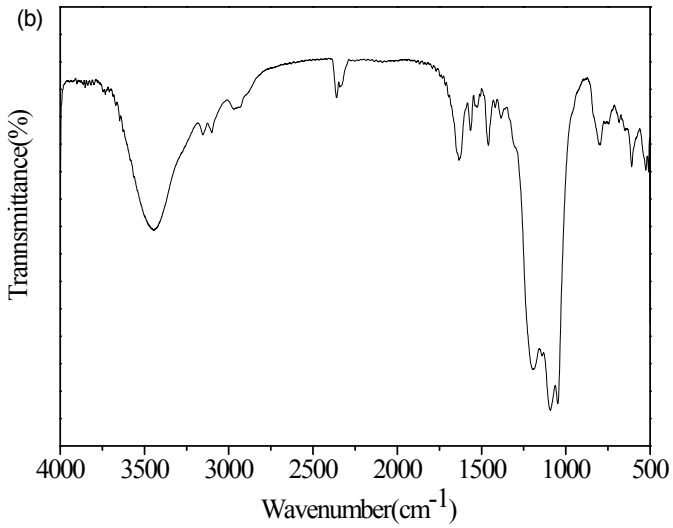

Figure S6 (b)FT-IR spectra of Cr(Salen-Cl)-IM- HSO_4-MCM-41.

FT-IR(KBr, cm^{-1}): 3445, 3159, 3103, 2363, 1626, 1564,1460, 1190, 1089, 1048, 783, 605, 507.

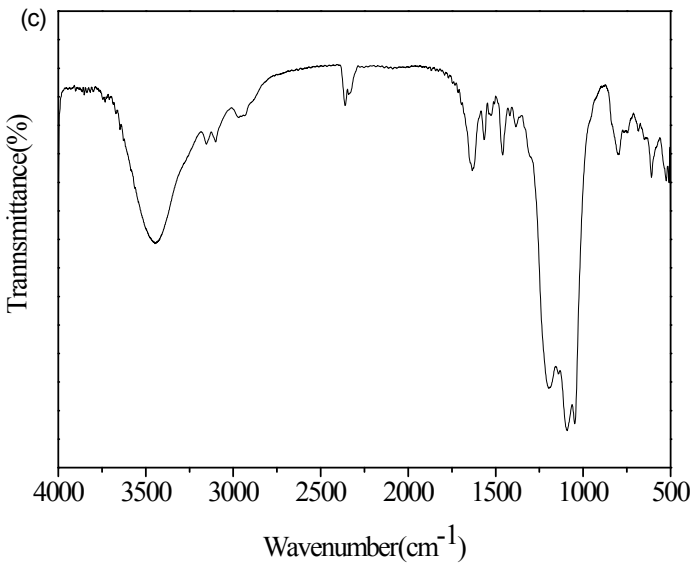

Figure S6 (c)FT-IR spectra of Cr(Salen-Cl)-IM- HSO_4-MCM-41.

FT-IR(KBr, cm^{-1}): 3441, 3160, 3105, 2361, 1627, 1565,1459, 1191, 1091, 1048, 785, 607, 509.

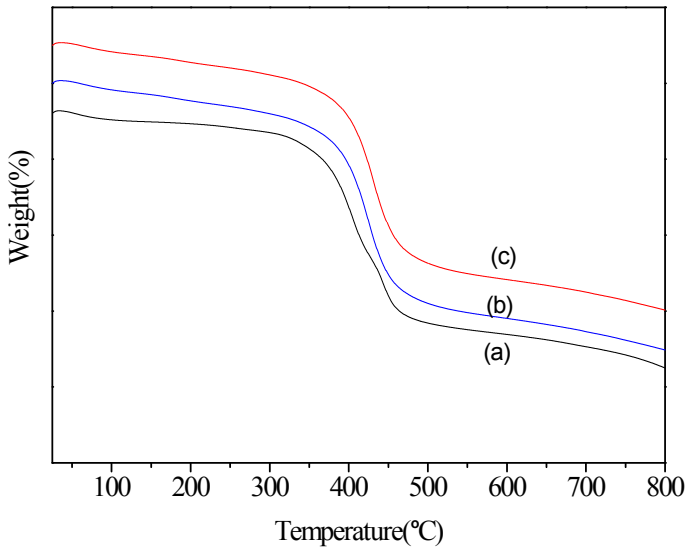

Figure S7 TG analysis for the Cr(Salen)-IM-Cl-MCM-41(a), Cr(Salen-Cl)-IM-HSO_4-MCM-41 (b), and Cr(Salen-Br)-IM-HSO_4-MCM-41(c).

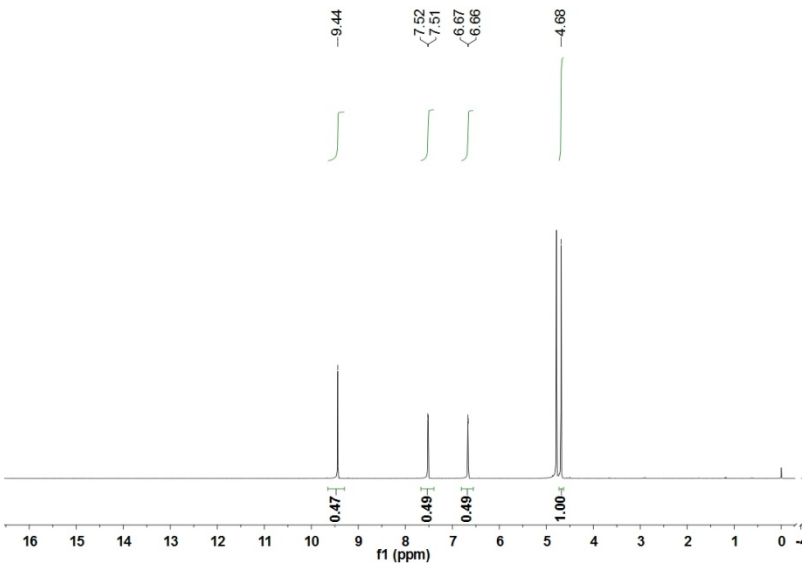

Figure S8 (a): ^{1}H NMR spectrum of HMF.

^{1}H NMR (D_2O, 400 MHz): δ 4.68 (s, 2H), 6.66 (d, *J* = 4 Hz, 1H), 7.51 (d, *J* = 4 Hz, 1H), 9.44 (s, 1H).

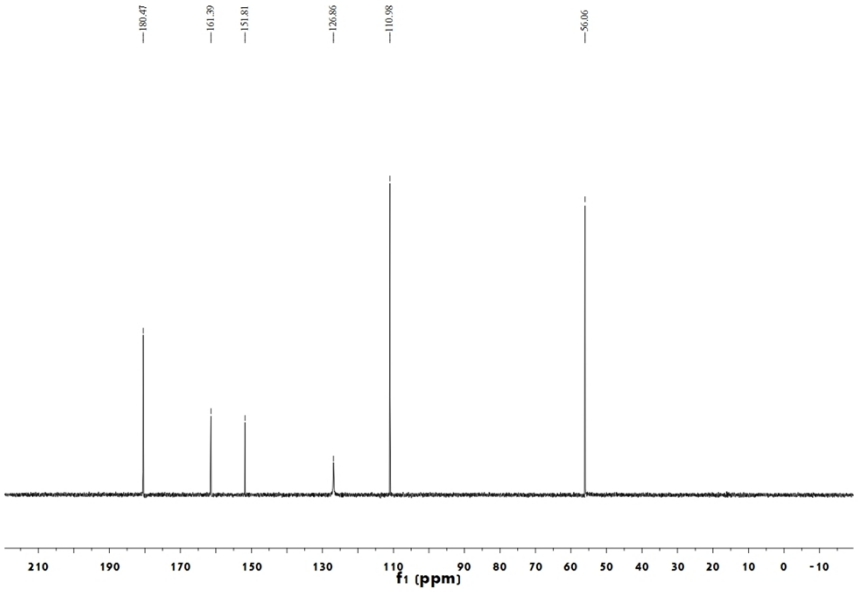

Figure S8 (b): ^{13}C NMR spectrum of HMF.

^{13}C NMR (CD_3Cl, 100 Hz): δ(ppm) 56.06, 110.98, 126.86, 151.81, 161.39, 180.47.

Table S1 Dehydration of fructose to HMF using different catalysts

Entry	Catalyst	Time(min)	Temperature(°C)	Fructose conversion (%)	HMF Yield (%)
1	IM-HSO_4-MCM-41	60	130	99.9	87.5
2	IM-HSO4-MCM-41	60	140	99.9	82.3
3	IM-Cl-MCM-41	60	130	99.9	76.5
4	IM-Cl-MCM-41	60	140	99.9	74.2

Reaction Conditions: Fructose (100mg), catalyst (40 mg), solvent (2mL DMSO).