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Highly efficient and recyclable copper based ionic liquid catalysts for amide synthesis

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Supporting Information

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NMR investigation for [Bmim][OAc]-[$Cu(OAc)_2$] and its precursor compound

^1H NMR spectrum of [Bmim][OAc] (Fig. S1a) shows that all the protons (labeled a-g) associated with N-methyl imidazole ring, butyl and acetate groups are present except the $(N)_2$-C-H imidazole ring proton. This shows that $(N)_2$-C-H proton is in dynamic equilibrium between N-methyl imidazole ring and acetate anion. ^{13}C NMR spectrum of [Bmim][OAc] (Fig. S1a) shows that all the carbons (labeled a-i) associated with N-methyl imidazole, butyl and acetate groups are present. ^1H-^1H correlation (COSY) spectrum (Fig. S1b) shows various H-H interactions present in the sample. ^{13}C-^1H correlation spectrum (HETCOR) (Fig. S1b) further confirms the formation of [Bmim][OAc]. HETCOR spectrum confirms that no proton is correlated with $(N)_2$-C carbon present in the ^{13}C spectrum of [Bmim][OAc], which clearly illustrates that $(N)_2$-C-H proton is in dynamic equilibrium between N-methyl imidazole ring and acetate anion. ^1H NMR spectrum of [Bmim][OAc]-[$Cu(OAc)_2$] (Fig. S2a) shows that protons (labeled a-h) associated with N-methyl imidazole ring and butyl groups are present but acetate protons are absent (Fig. S2). Due to the presence of paramagnetic Cu(II), ^1H spectrum of [Bmim][OAc]-[$Cu(OAc)_2$] is broad when compared with [Bmim][OAc]. ^{13}C NMR spectrum of [Bmim][OAc] (Fig. S2a) confirms that all the carbons (labeled a-i) associated with N-methyl imidazole ring and butyl group are present but the acetate carbon does not exhibit any signal in ^{13}C spectrum. ^1H-^1H and ^{13}C-^1H correlation spectra (Fig. S2b) further confirm that acetate ions exhibit no signal in the NMR investigation. ^1H and ^{13}C NMR investigations confirm that all the acetate ions present in the sample are in dynamic equilibrium with those complexed with paramagnetic Cu(II).

Table S1. Elemental analysis of supported catalysts investigated in this study.

Sample	**C (wt %)[a]**	**N (wt %)[a]**	**Cu (wt%)[b]**
Nano-S-1	0.24	0.04	None
Nano-S1-[Bmim][Cl]-[CuCl]	5.56	1.60	3.41
Nano-S1-[Bmim][Cl]-[$CuCl_2$]	5.64	1.63	3.50
Nano-S1-[Bmim][OAc]-[$Cu(Oac)_2$]	8.50	1.42	3.06

[a]Obtained from CHN elemental analyzer
[b]Obtained from AAS analysis

(a)

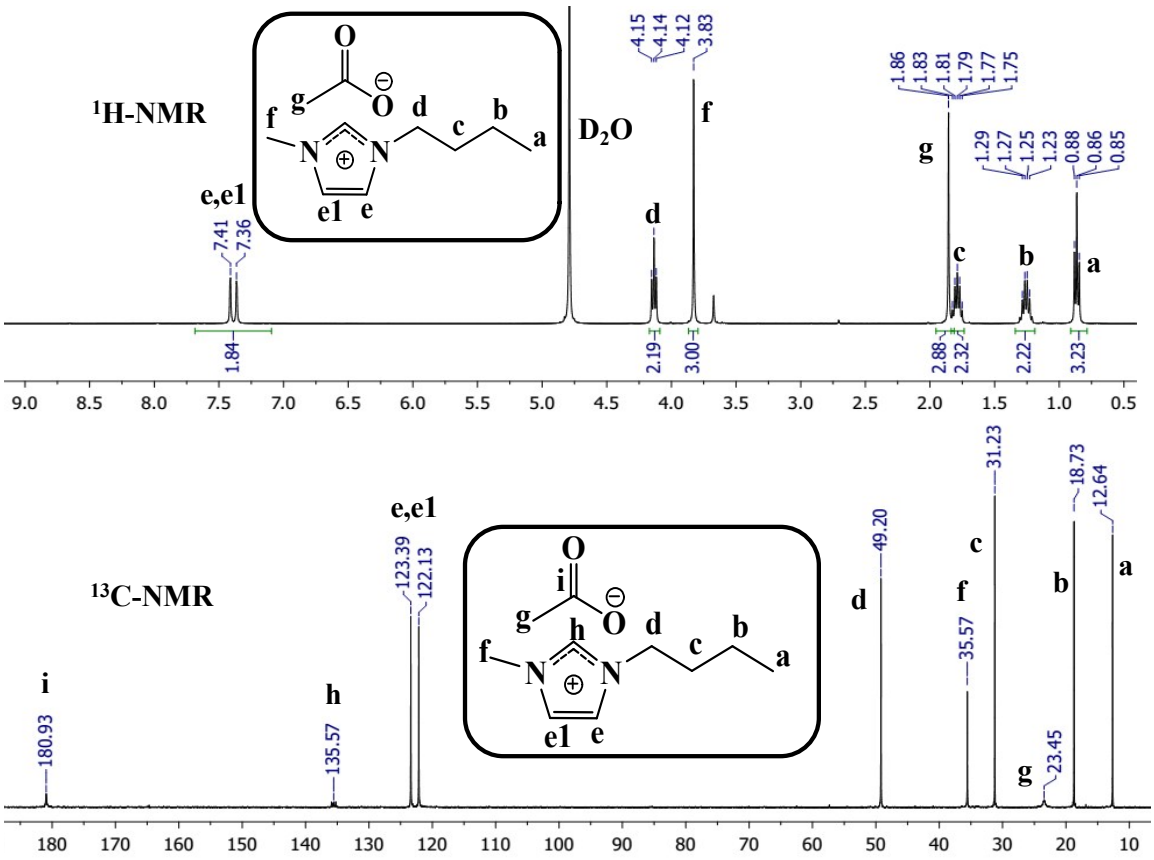


Fig. S1 (Continued)

(b)

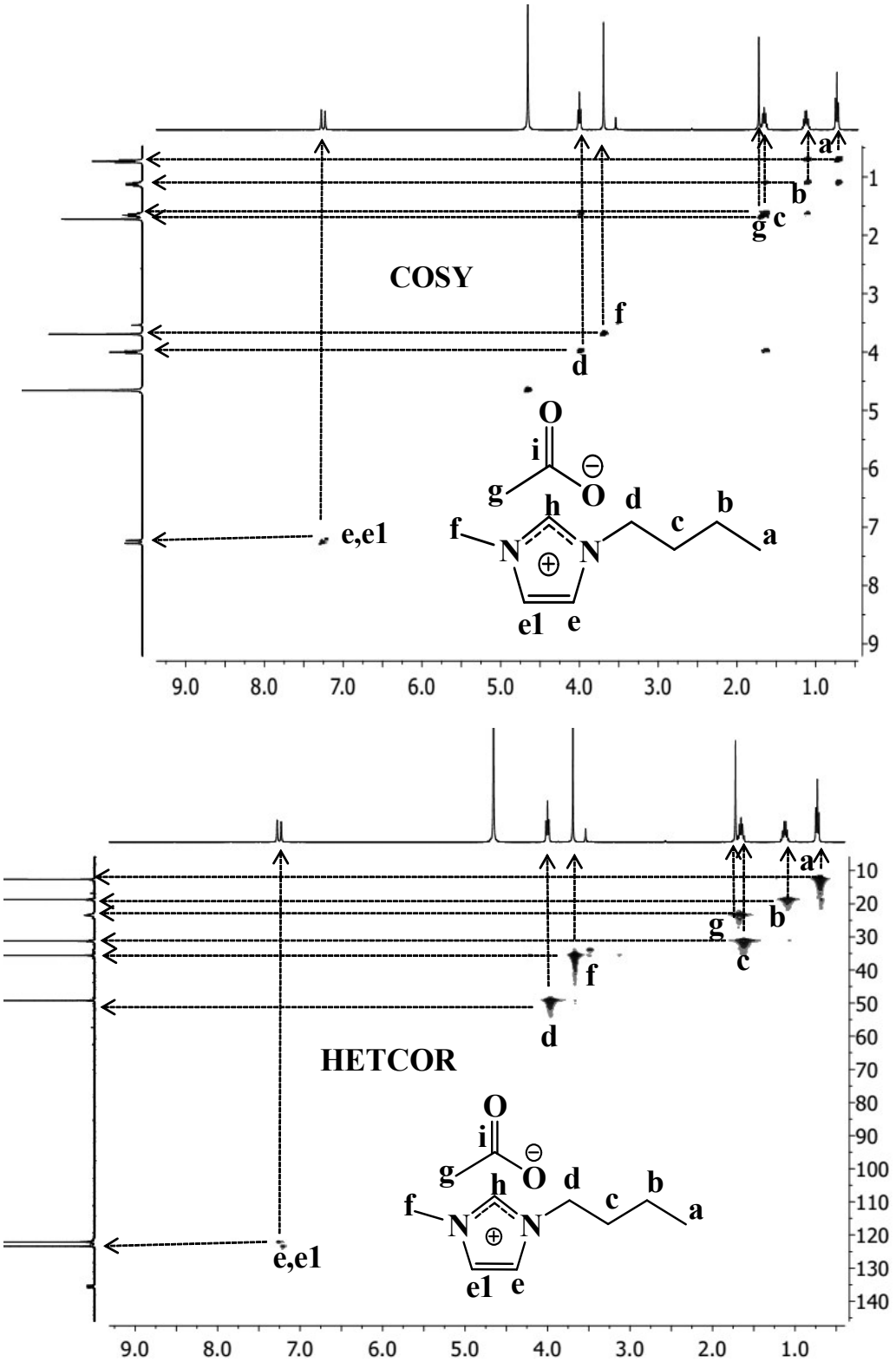


Fig. S1. (a) ^{1}H and ^{13}C, (b) COSY and HETCOR, NMR spectra of [Bmim][OAc].

(a)

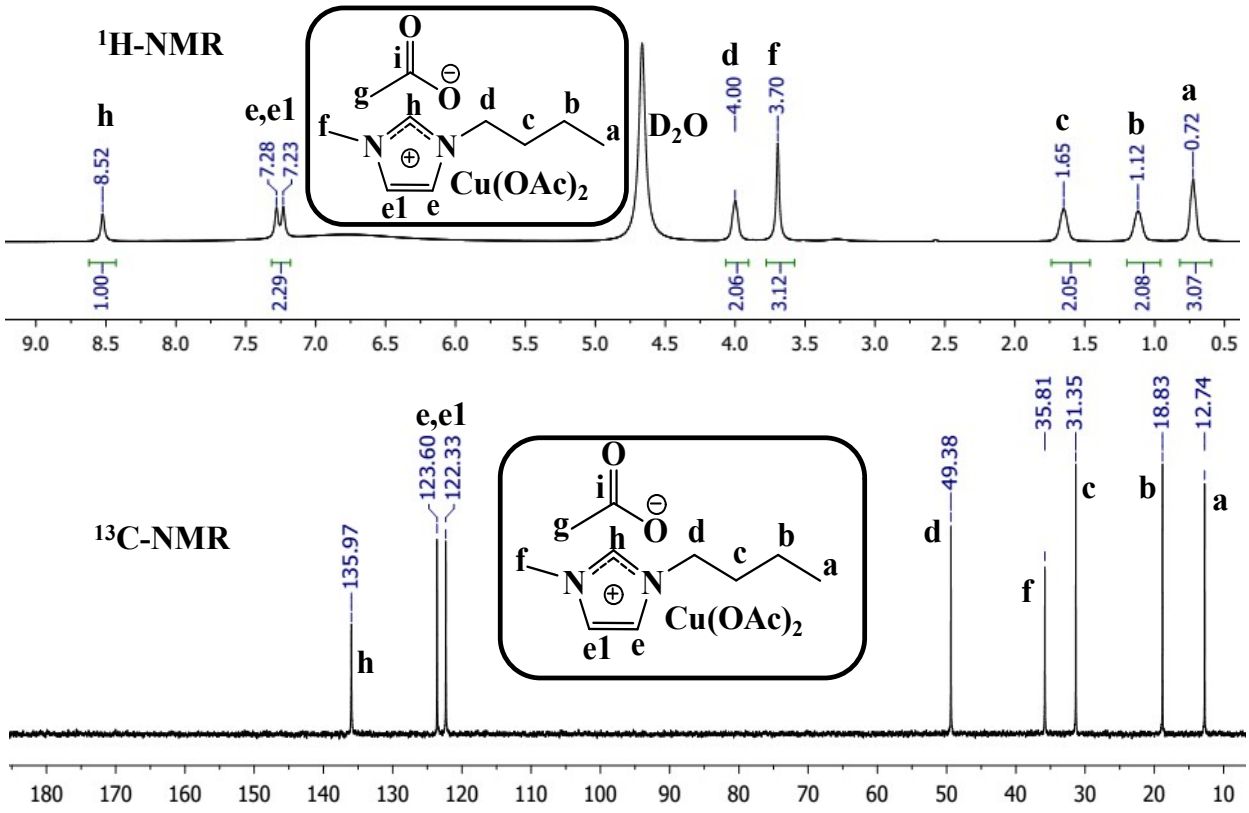


Fig. S2 (Continued)

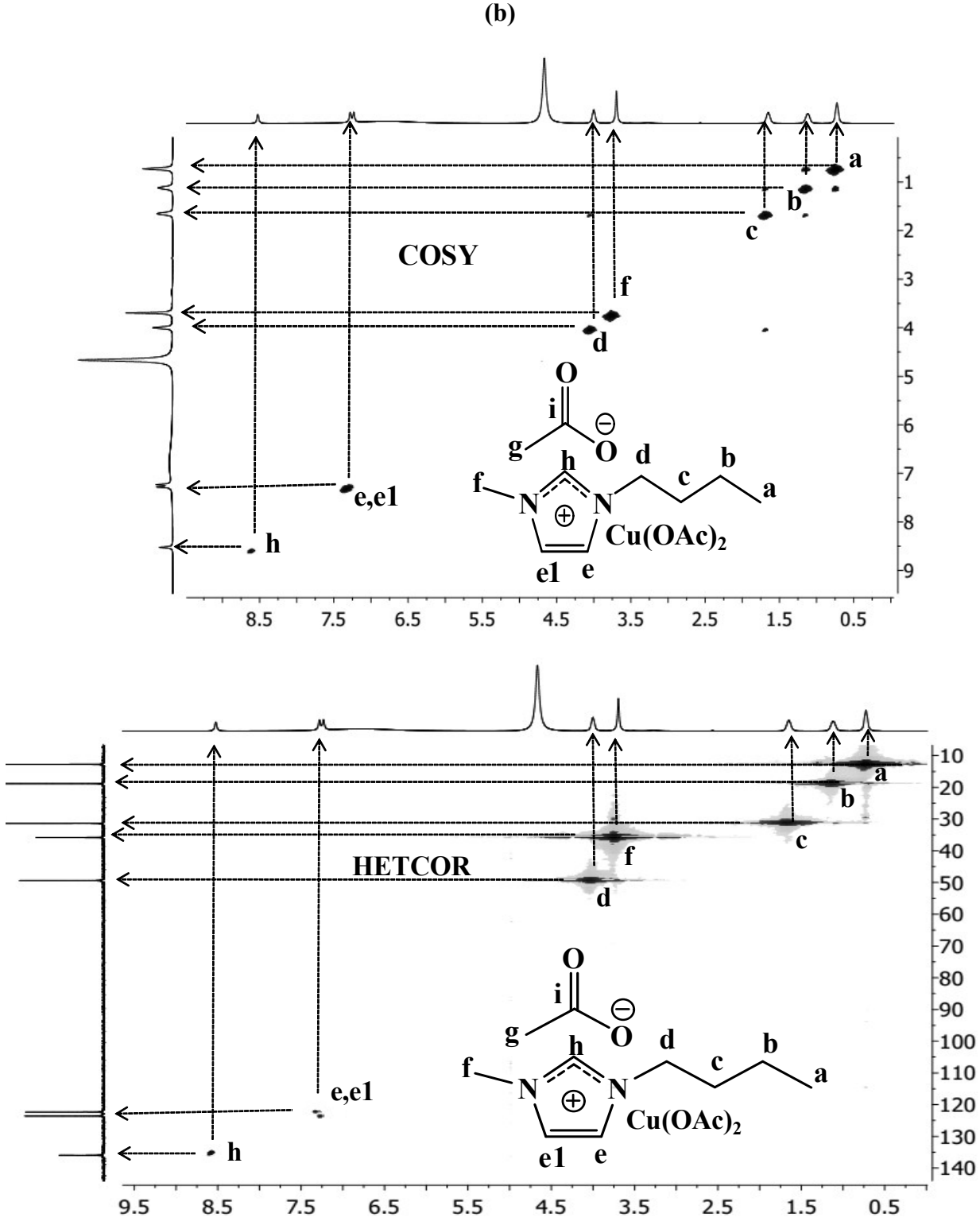


Fig. S2. (a) ^{1}H and ^{13}C (b) COSY and HETCOR, NMR spectra of [Bmim][OAc]-[$Cu(OAc)_2$].

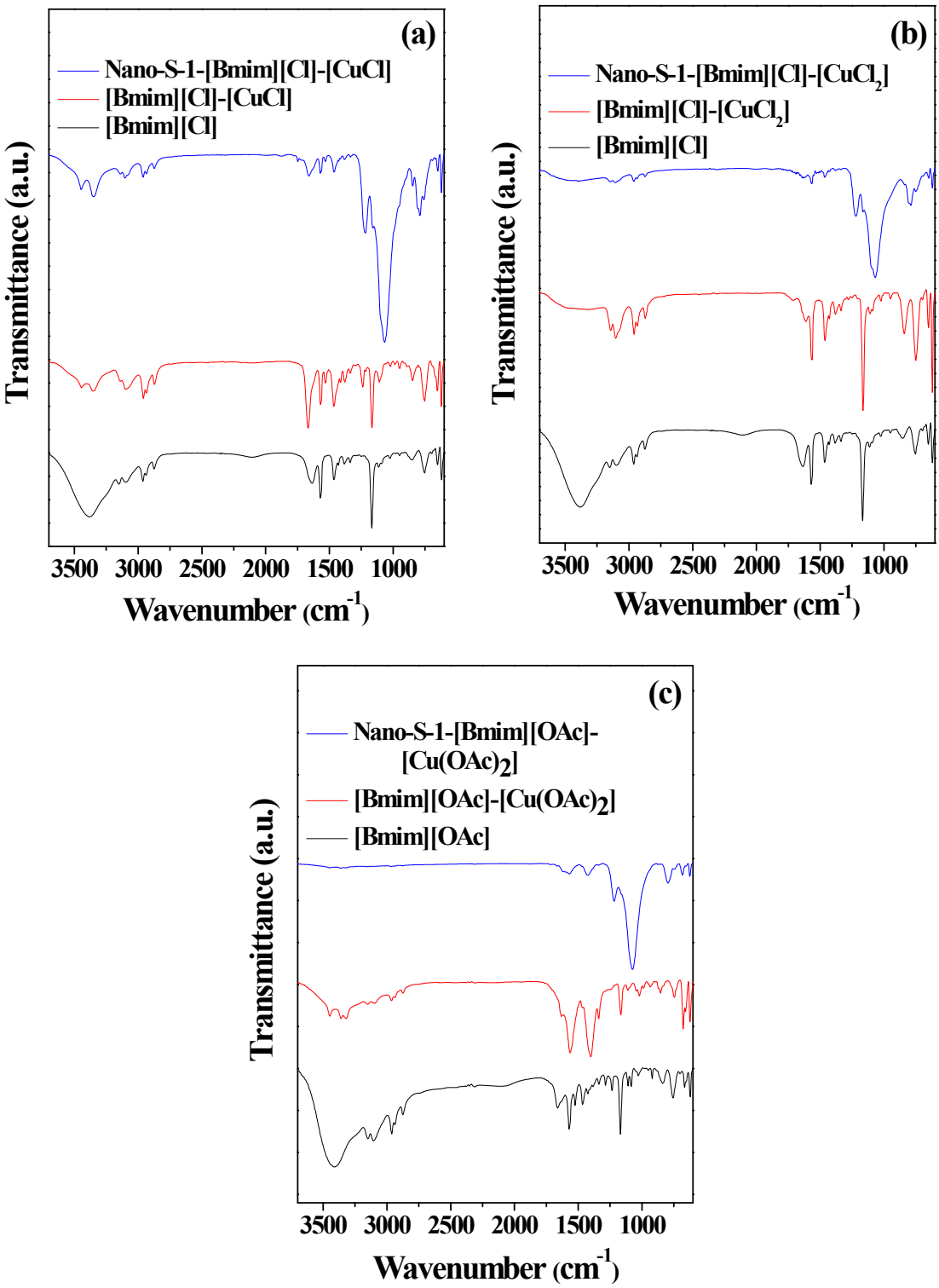


Fig. S3*:* FT-IR spectra of Cu based ILs and functionalized ILs catalysts and their precursor compounds.

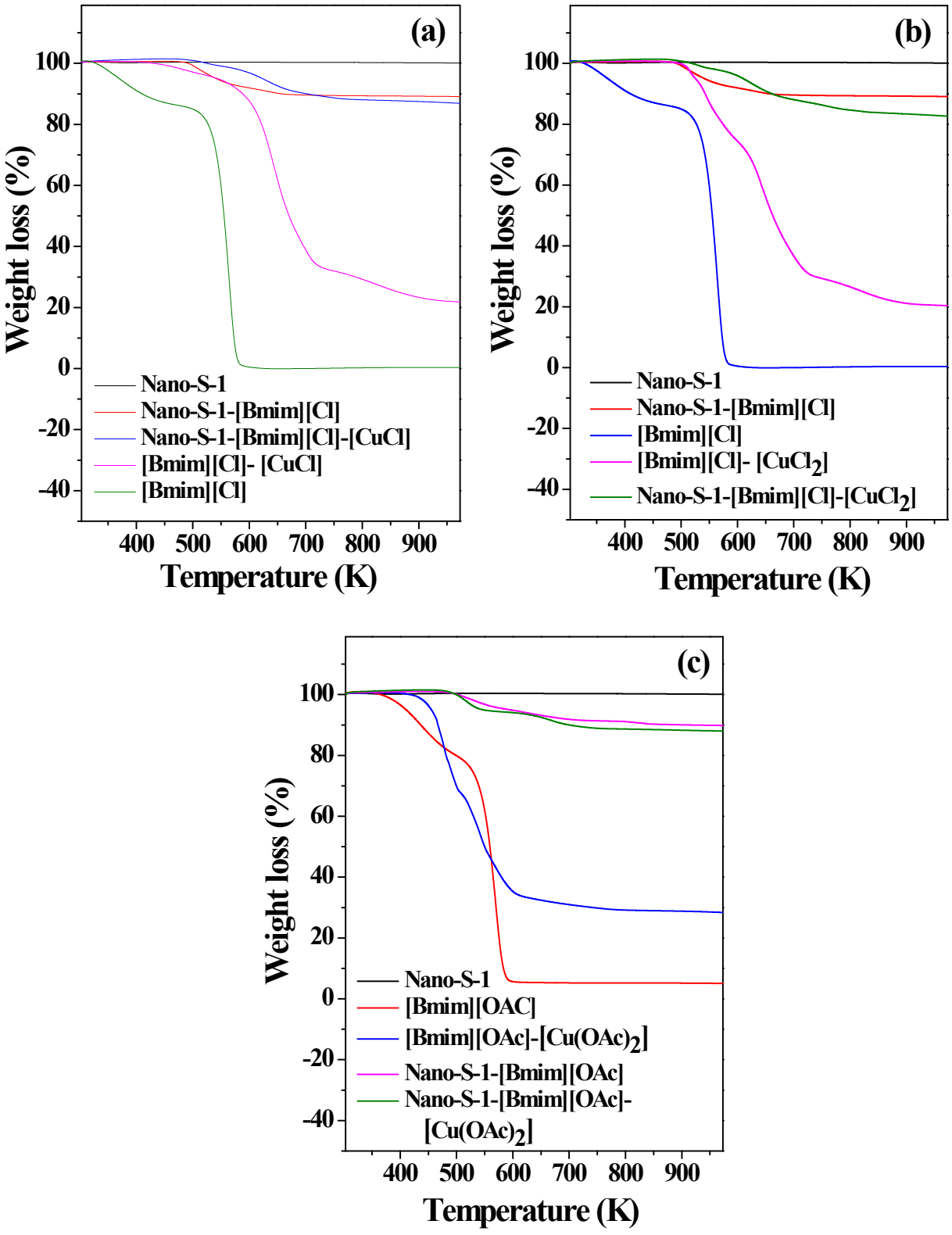


Fig. S4*:* Thermo gravimetric analysis of neat Cu based ILs and Nano-S-1 supported Cu based ILs and their precursor compounds.

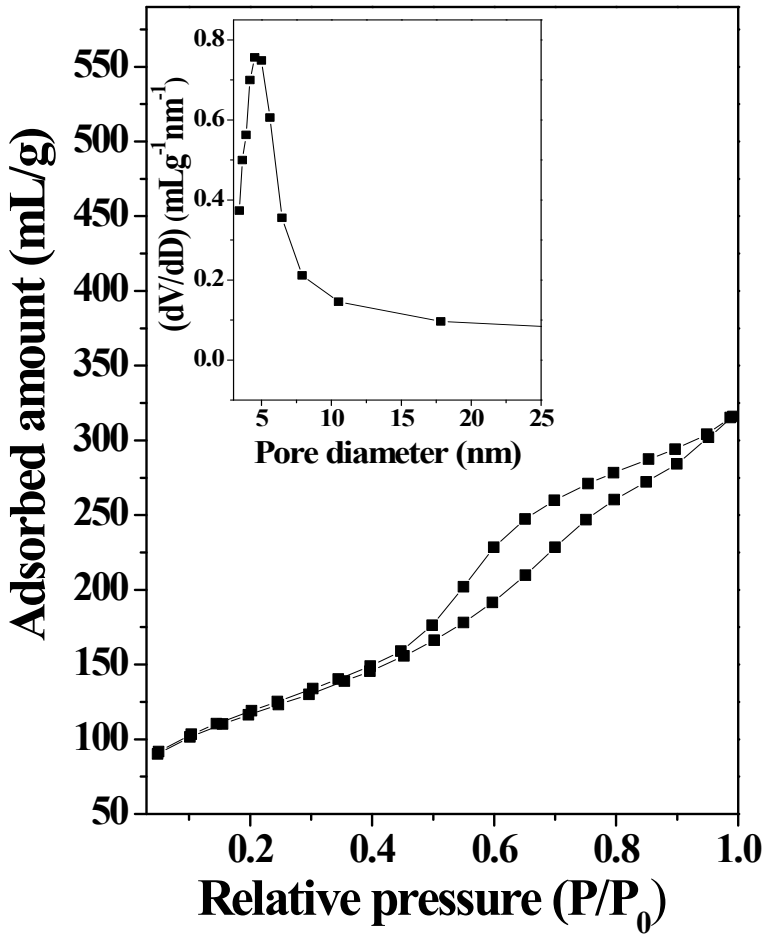


Fig. S5*:* N_2-adsorption isotherm of Nano-S-1 (Inset shows pore size distribution).

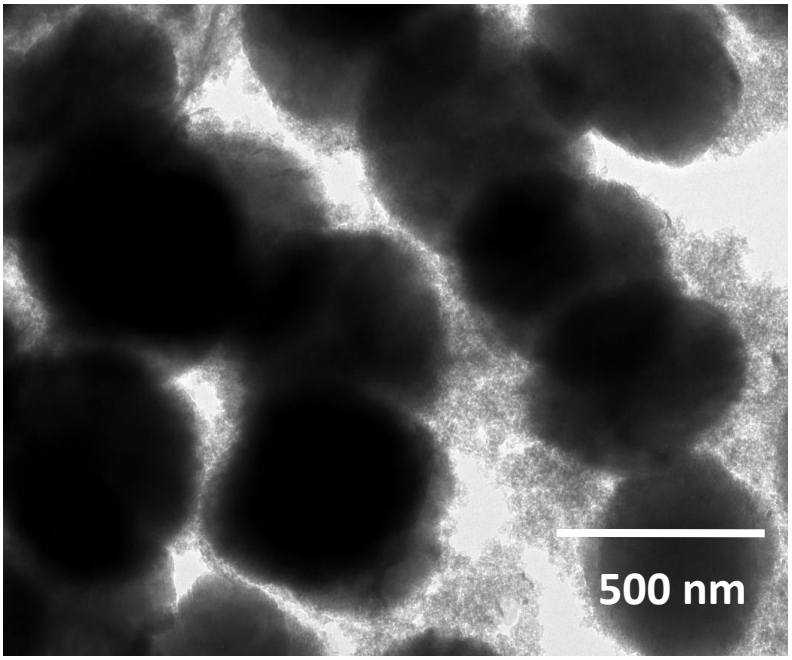


Fig. S6*:* TEM images of Nano-S-1.

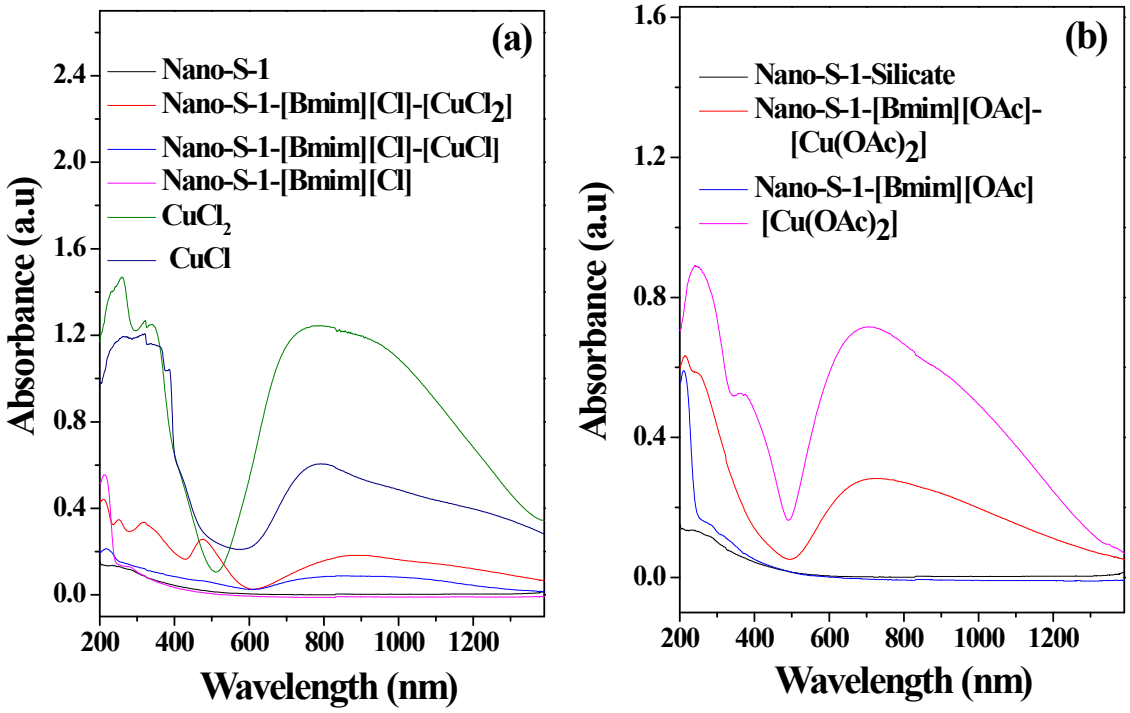
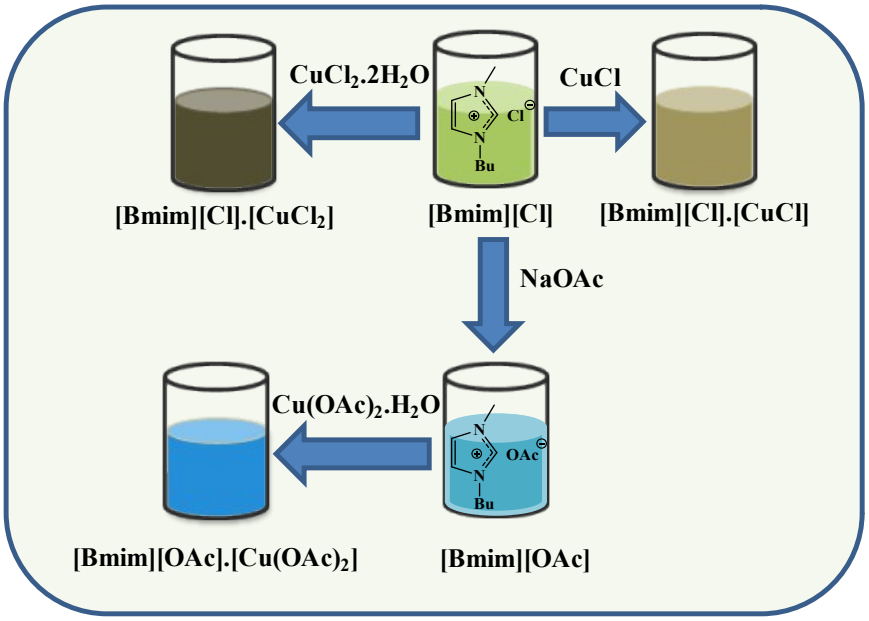


Fig. S7*:* UV-visible spectra of Cu based ILs and functionalized ILs catalysts and their precursor compounds.



Scheme S1: Changes in the color observed during the synthesis of ILs.