

Electronic Supporting Information file

Green carbon-carbon homocoupling of terminal alkynes by silica supported Cu(II)-hydrazone coordination compound

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Materials and Instrumentation

4-Aminobenzoic hydrazide, 2-hydroxy-3-methoxy benzaldehyde, copper(II) nitrate trihydrate, propionyl chloride-functionalized silica gel (≈ 1 mmol/g loading) and solvents were purchased from Sigma-Aldrich and used as received. Elemental analyses (C, H, N) were performed on a Carlo ERBA Model EA 1108 analyzer. A Varian AA-220 atomic absorption spectroscopy instrument was used to measure the copper content of the compounds. The ^1H and ^{13}C NMR spectra were recorded from Bruker DRX-300 spectrometer in DMSO- d_6 solution. FT-IR spectra were taken by using a Nicolet iS10 FT-IR spectrophotometer as KBr disks. The UV-Vis spectra of solutions were recorded on a thermos-spectronic Helios Alpha spectrophotometer between 200-800 nm. Diffuse-reflectance UV-Vis spectroscopy (DRS) were recorded with Sinco S4100 instrument. Energy-dispersive spectroscopy (EDS) and scanning electron microscopy (SEM) were recorded with TESCAN MIRA III instrument. X-ray photoelectron spectroscopy (XPS) analysis was performed by using a Bes Tec 8025 instrument. EPR spectra were recorded on a Bruker ELEXSYS E 500 CW-EPR spectrometer operating at the X-band frequency (≈ 9.5 GHz) and equipped with an ER 036TM NMR Teslameter and E 41 FC frequency counter. The spectra were recorded at room temperature and 77 K by using a standard temperature control system (Bruker) with a nitrogen gas flow. A SETSYS Evolution TGA instrument was used to record the

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thermal gravimetric analysis (TGA) curves. Powdered X-ray diffraction analyses (PXRD) were provided by using a Bruker D8 ADVANCE powder diffractometer with Cu radiation and Vantec detector.

X-ray Crystallography

The crystallographic data and structure refinement parameters are given in Table 5. The data were collected using a Kuma KM-4 diffractometer equipped with graphite-monochromated Mo K α radiation ($\lambda = 0.71073 \text{ \AA}$) and Sapphire a CCD detector. The data collections, unit cell refinements, data reduction were performed with the *CrysAlisPro* software package.ⁱ The intensity data were collected by ω -scan mode within $2.8^\circ < \theta < 28^\circ$ in the triclinic system. A blue crystal of compound **1** was selected and intensity data were measured at 100 K. The structure was solved by direct methods and refined by full-matrix least squares based on F^2 .

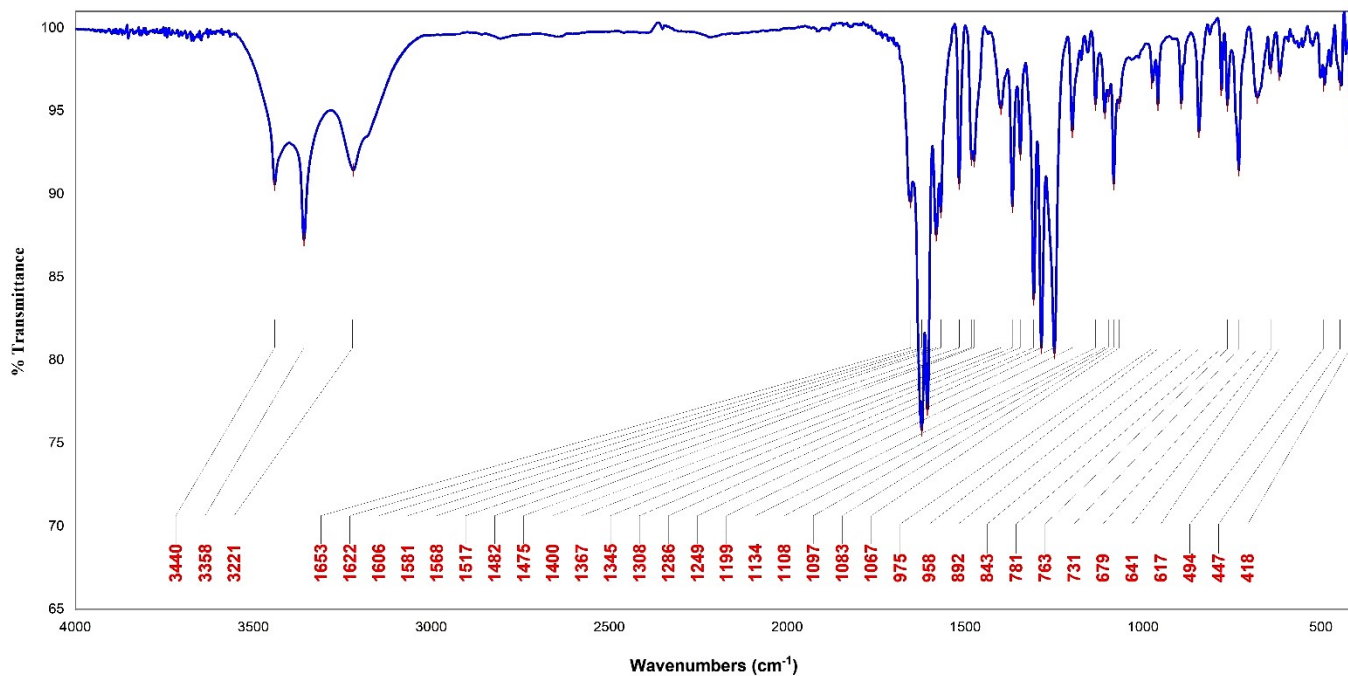


Fig. S1. FT-IR spectrum of H₂L

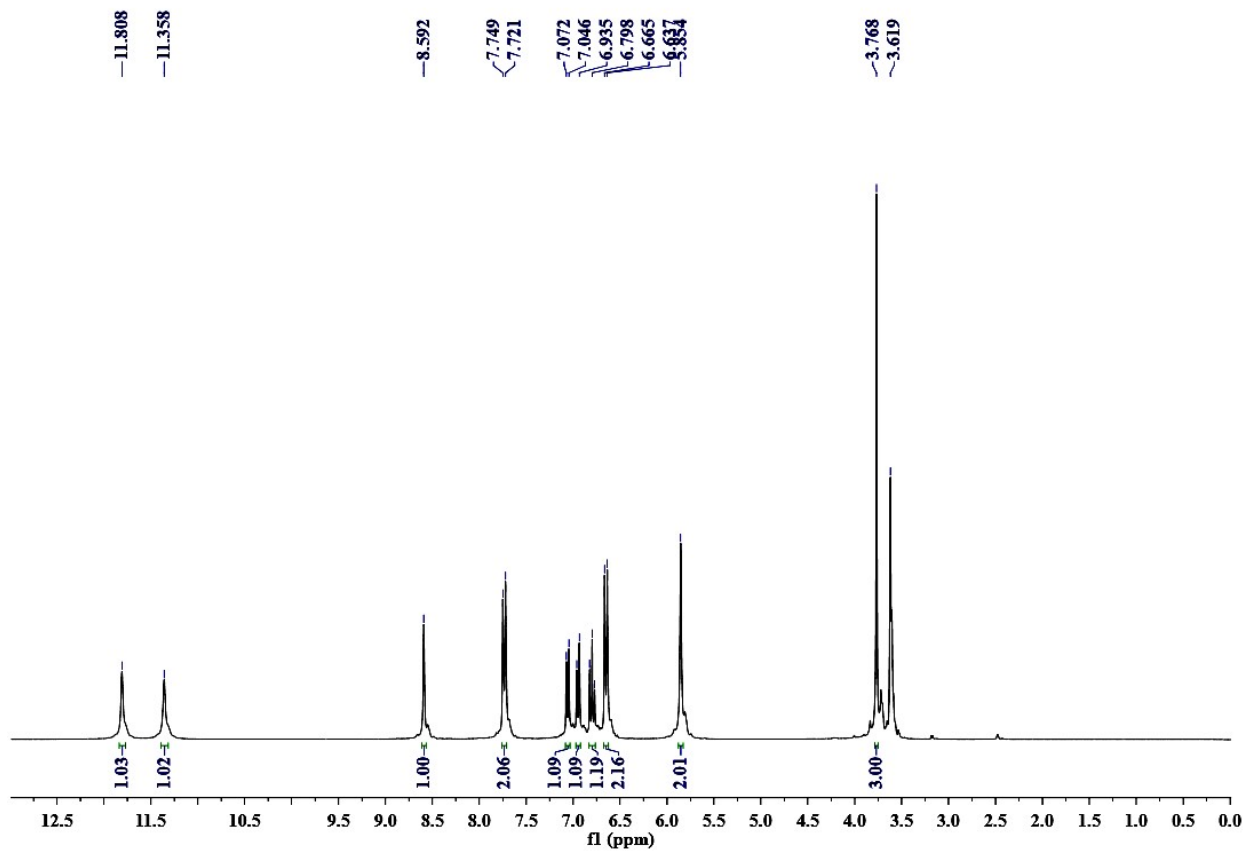


Fig. S2. ^1H NMR spectrum of H_2L in DMSO-d_6

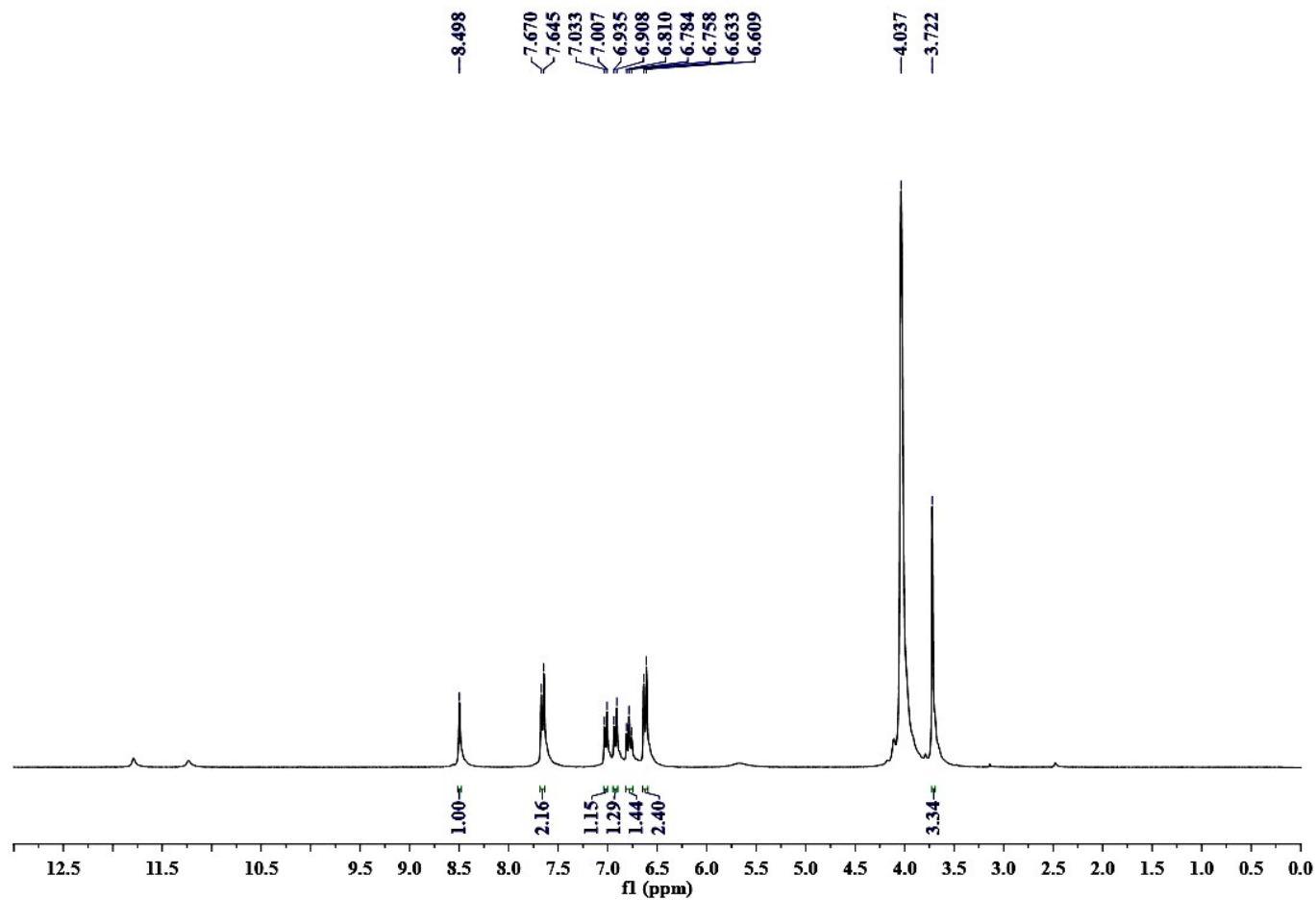


Fig. S3. ^1H NMR spectrum of H_2L in $\text{DMSO-d}_6 + \text{D}_2\text{O}$

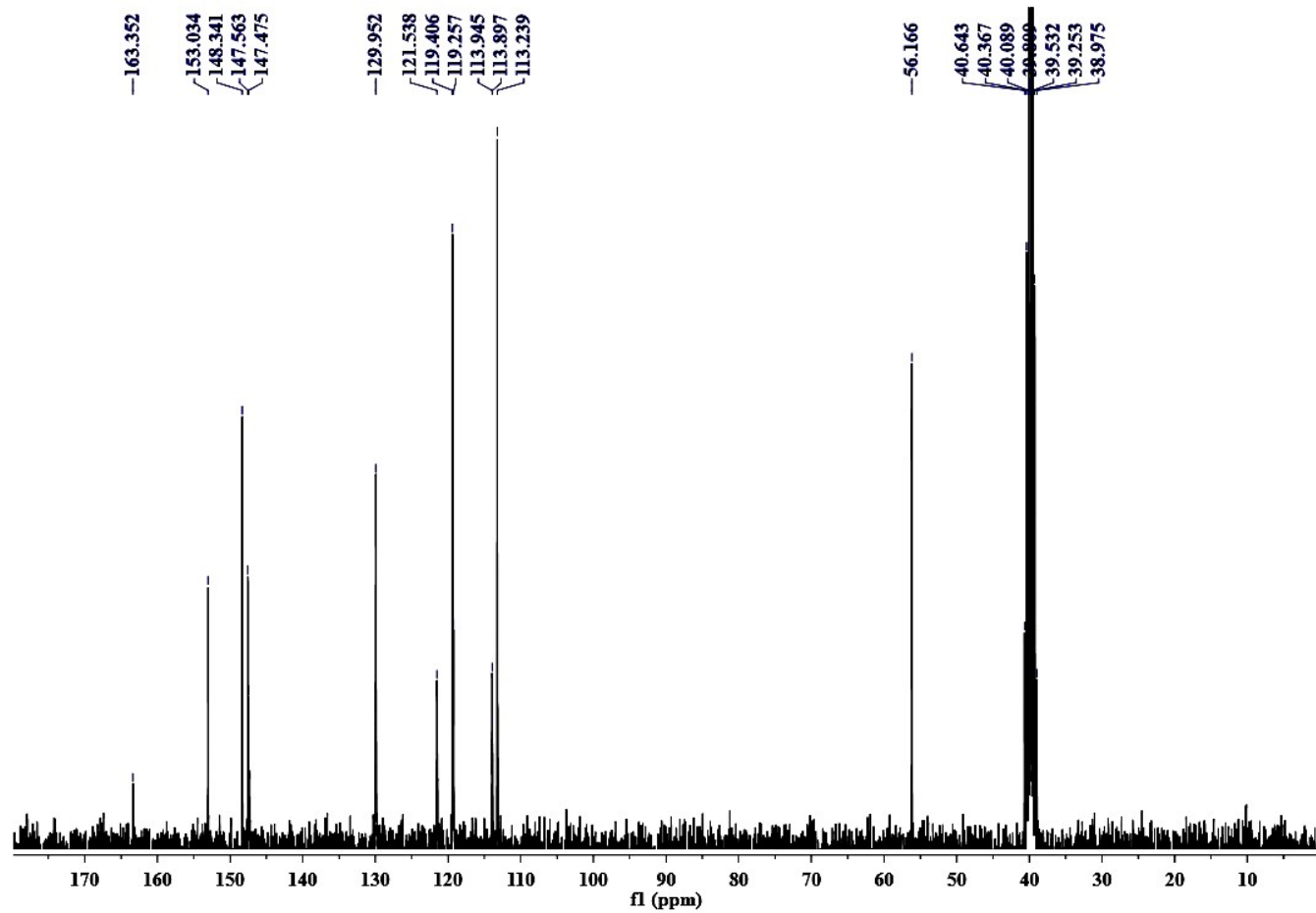


Fig. S4. ^{13}C NMR spectrum of H_2L in DMSO-d_6

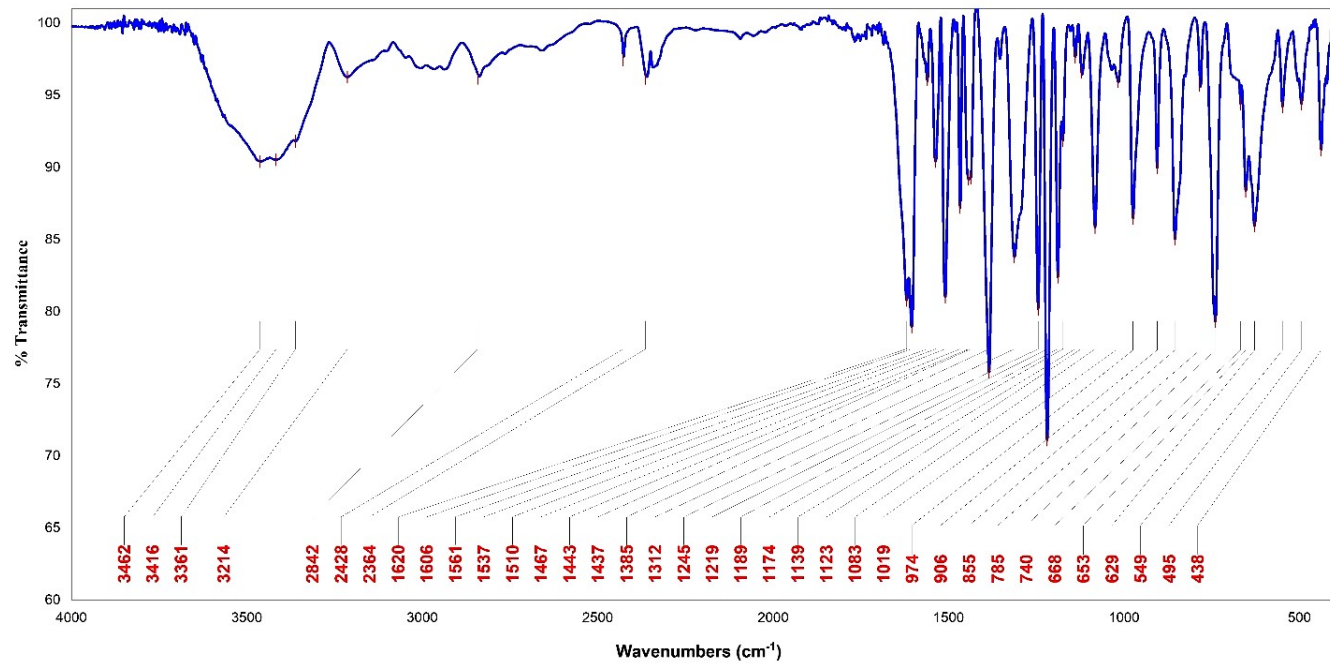


Fig. S5. FT-IR spectrum of compound **1**

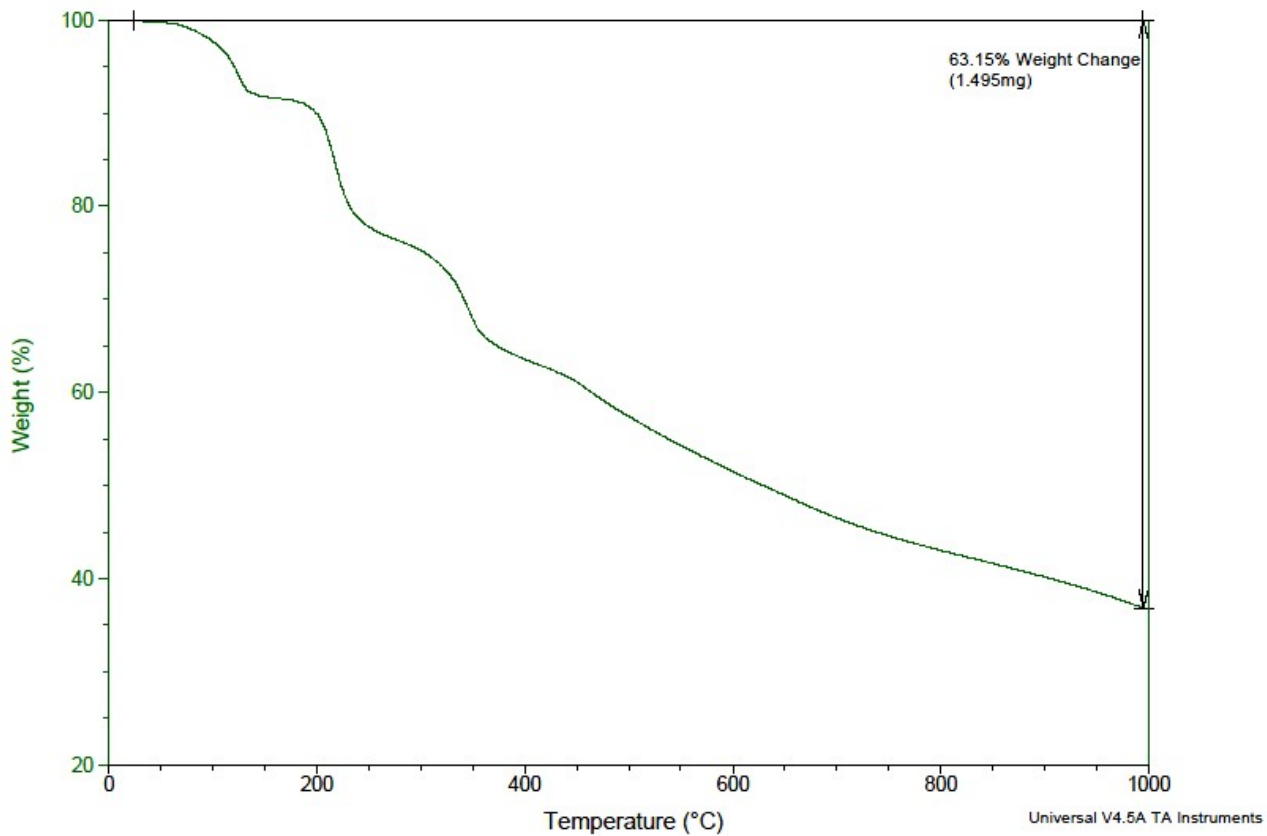


Fig. S6. TGA diagram of compound 1

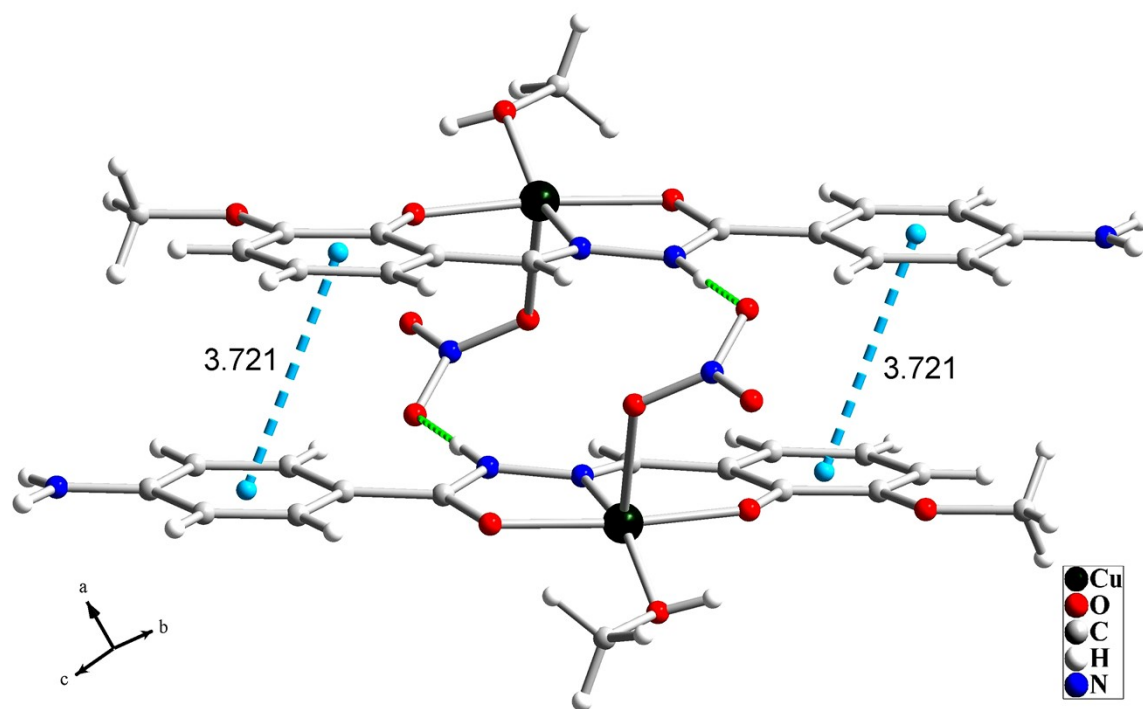


Fig. S7. Intermolecular $\pi \cdots \pi$ stacking (blue dashed line) and hydrogen bond interaction (O-H \cdots N) (green dashed line) in the crystal structure of **1**

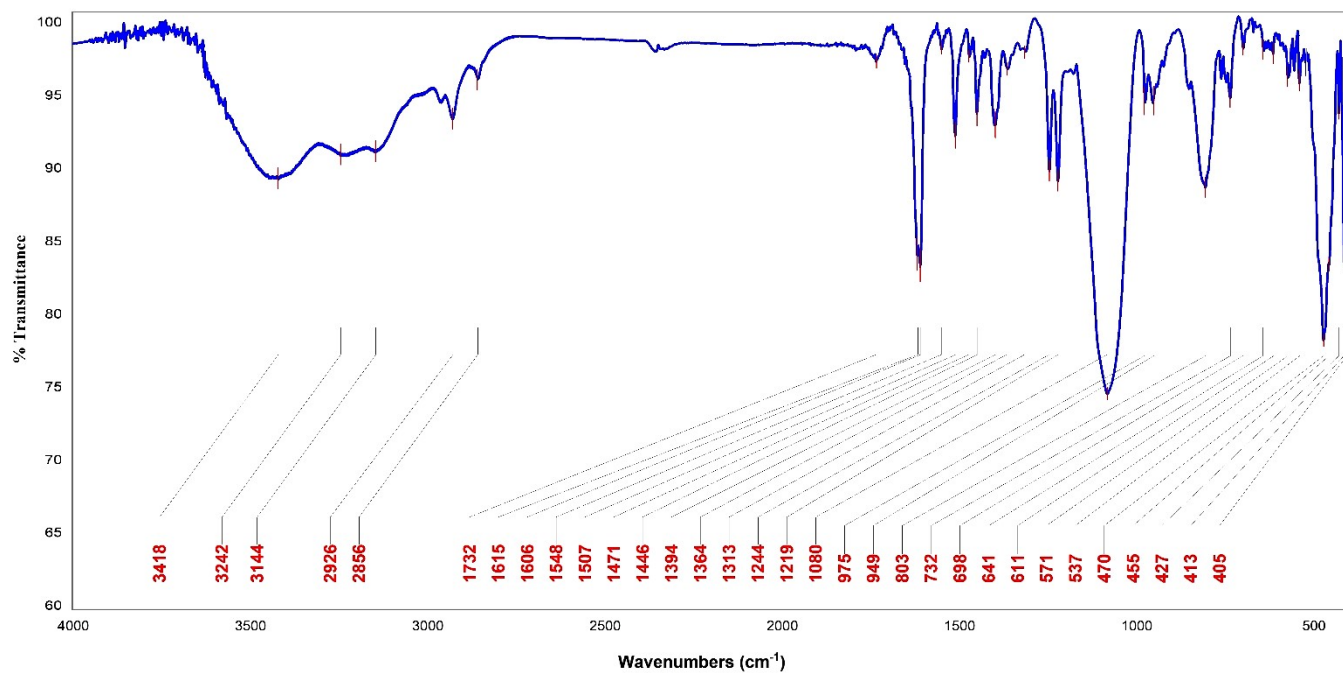


Fig. S8. FT-IR spectrum of supported catalyst

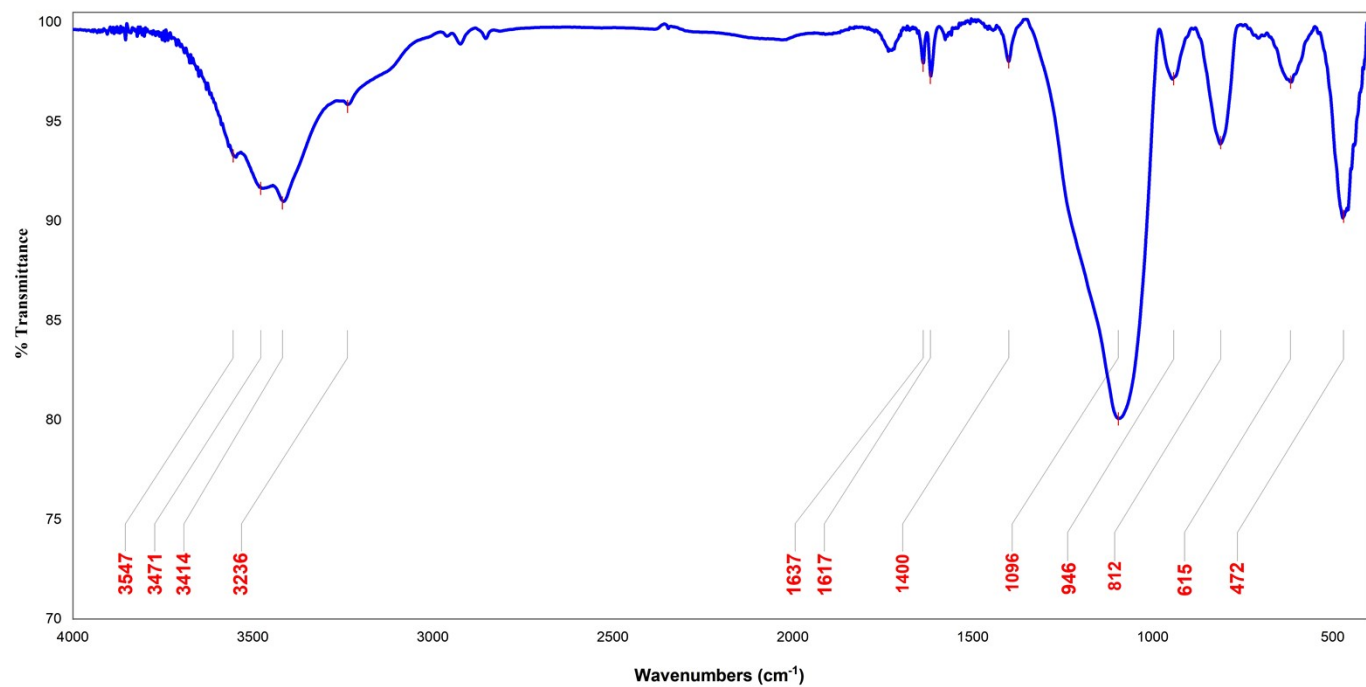


Fig. S9. FT-IR spectrum of propionyl chloride-functionalized silica gel

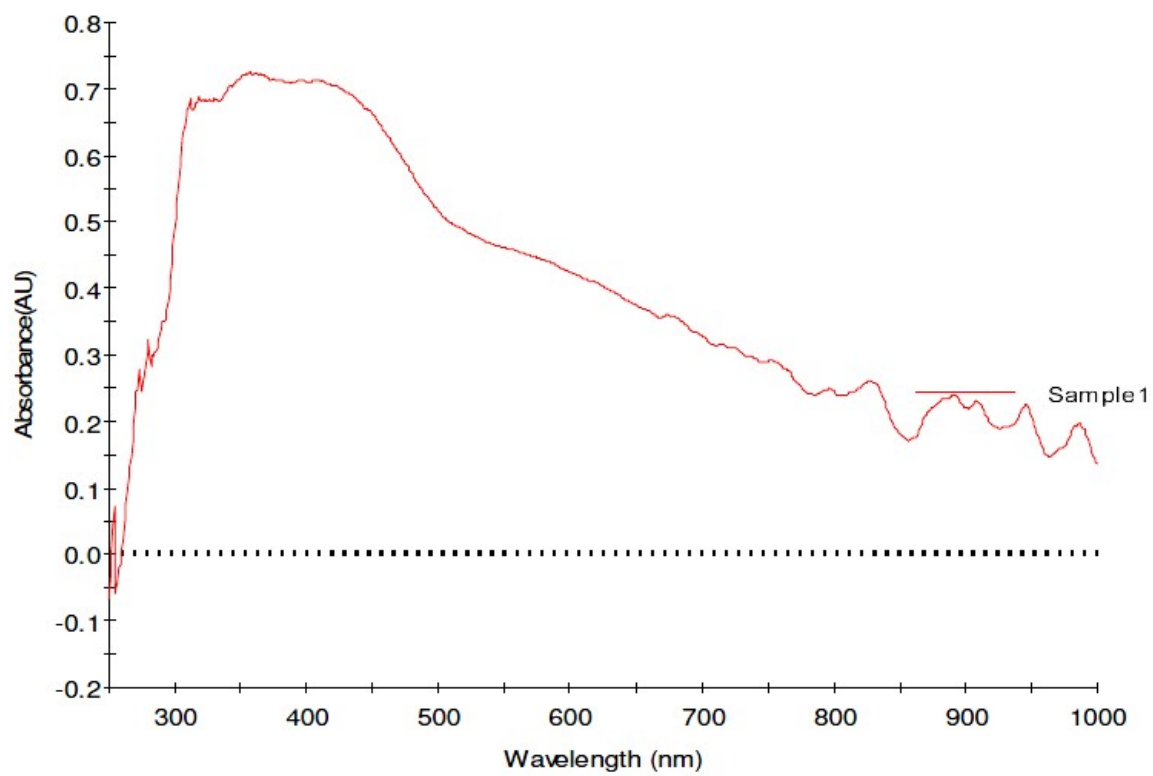


Fig. S10. DRS spectrum of supported catalyst

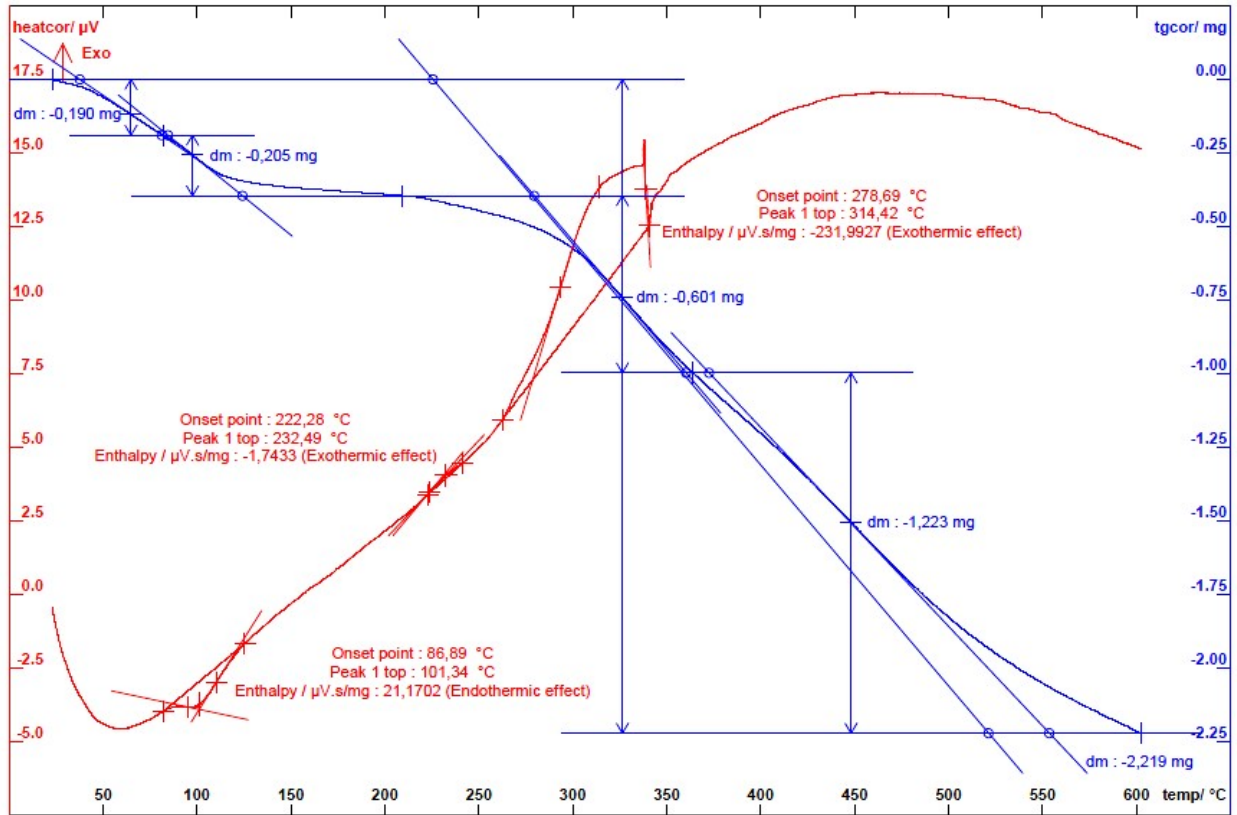
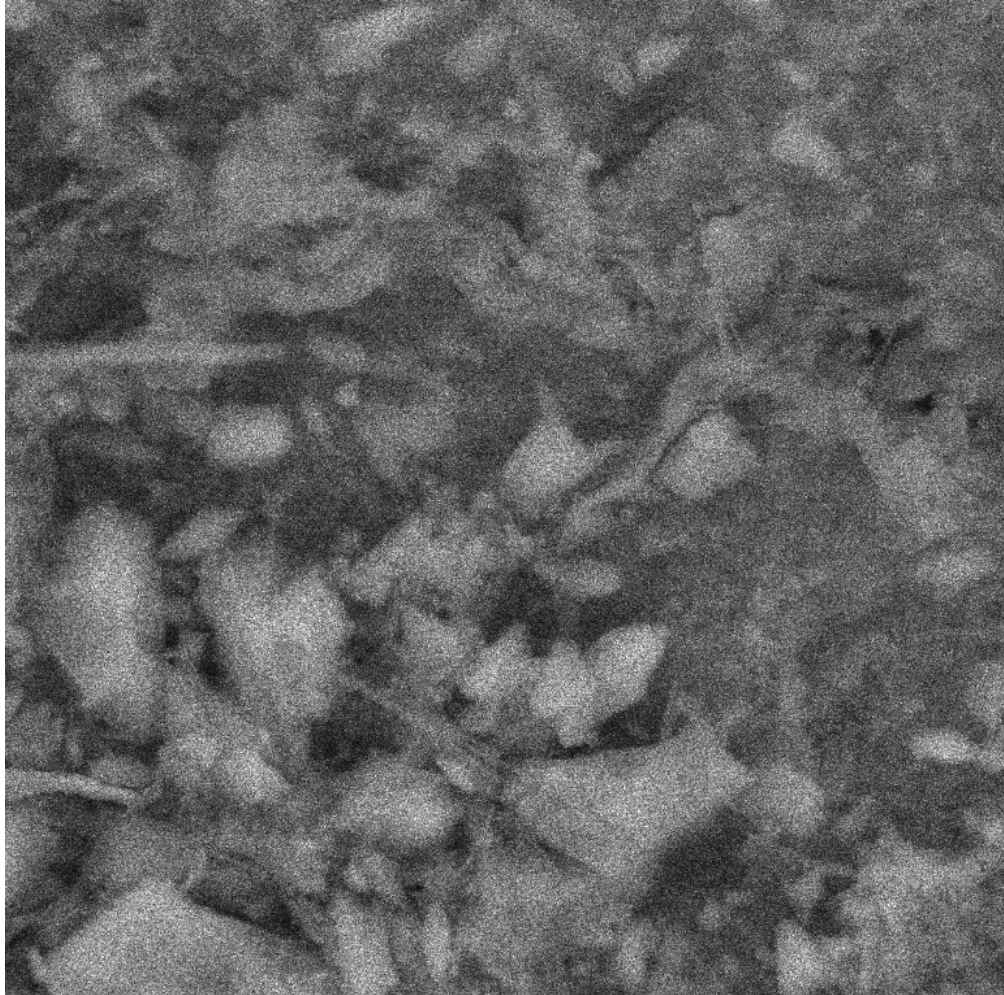


Fig. S11. TGA/DTA diagram of supported catalyst (Si-Cu)



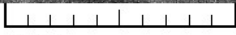

SEM HV: 15.00 kV WD: 15.15 mm  MIRA\\ TESCAN
SEM MAG: 5.00 kx Det: BSE 10 μ m
View field: 43.34 μ m Date(m/d/y): 02/16/21 

Fig. S12. Electron micrograph of Si-Cu

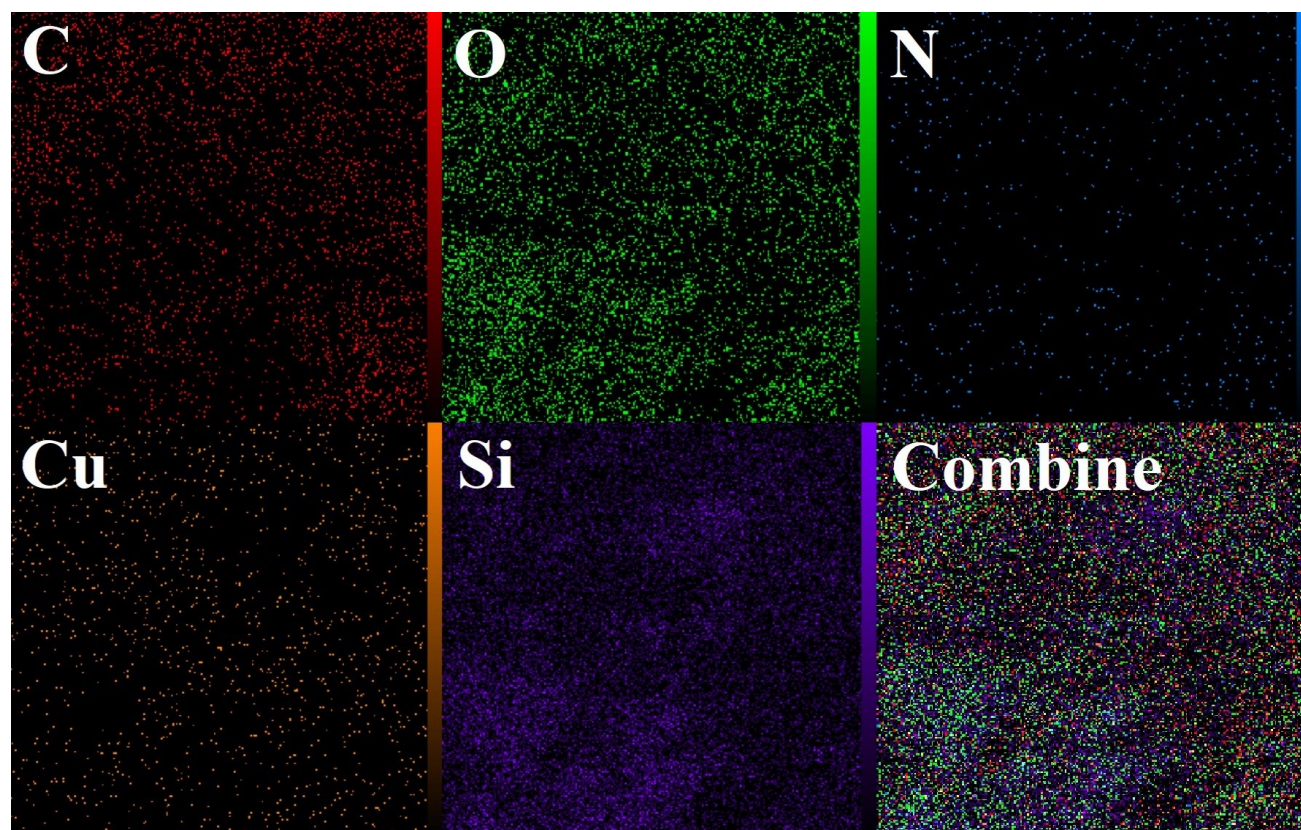


Fig. S13. EDS mapping images of Si-Cu

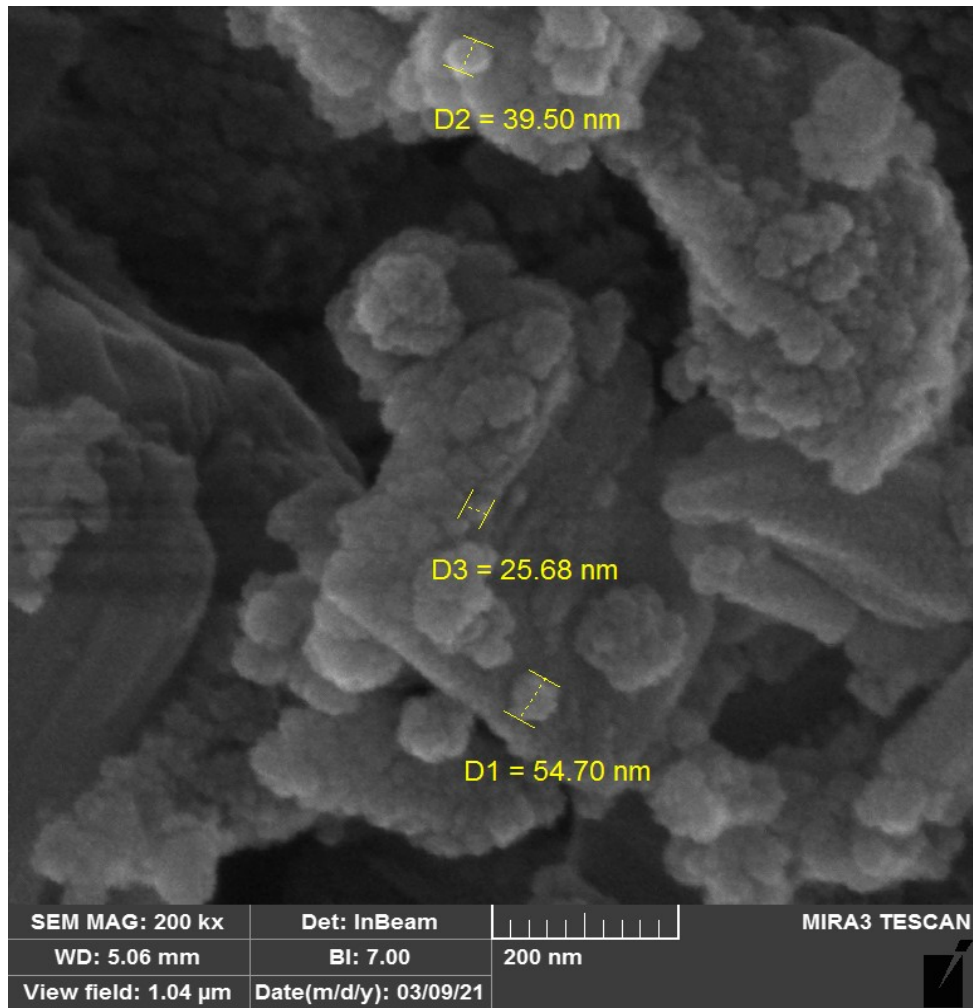


Fig. S14a. SEM images of Si-Cu (MAG: 200.0 kx)

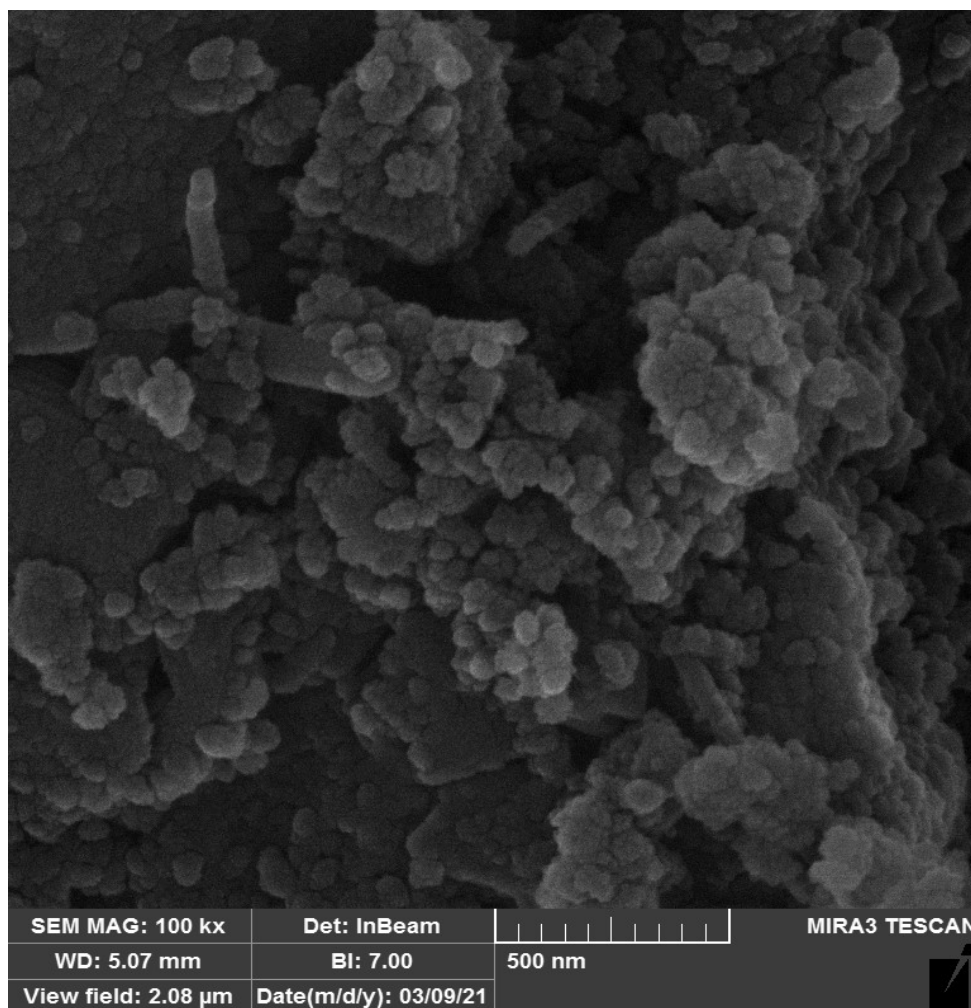


Fig. S14b. SEM image of Si-Cu (MAG: 100.0 kx)

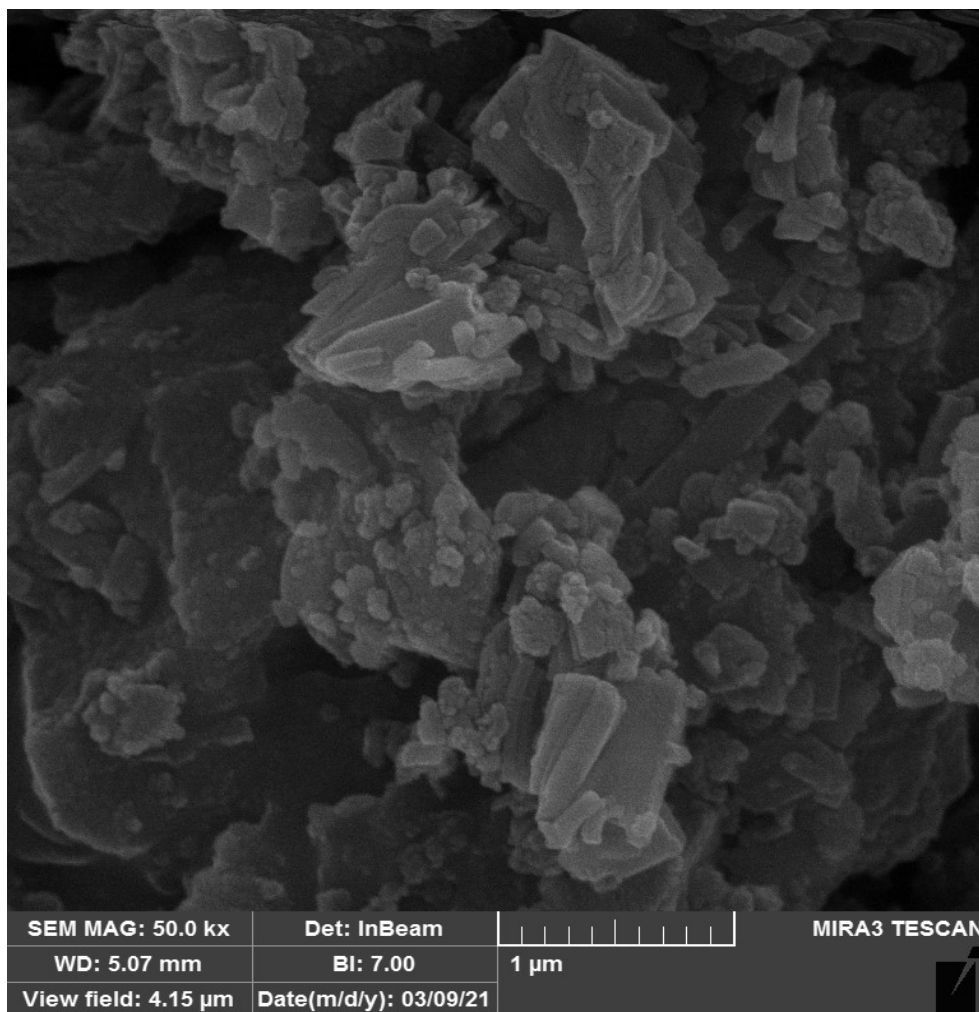


Fig. S14c. SEM image of **Si-Cu** (MAG: 50.0 kx)

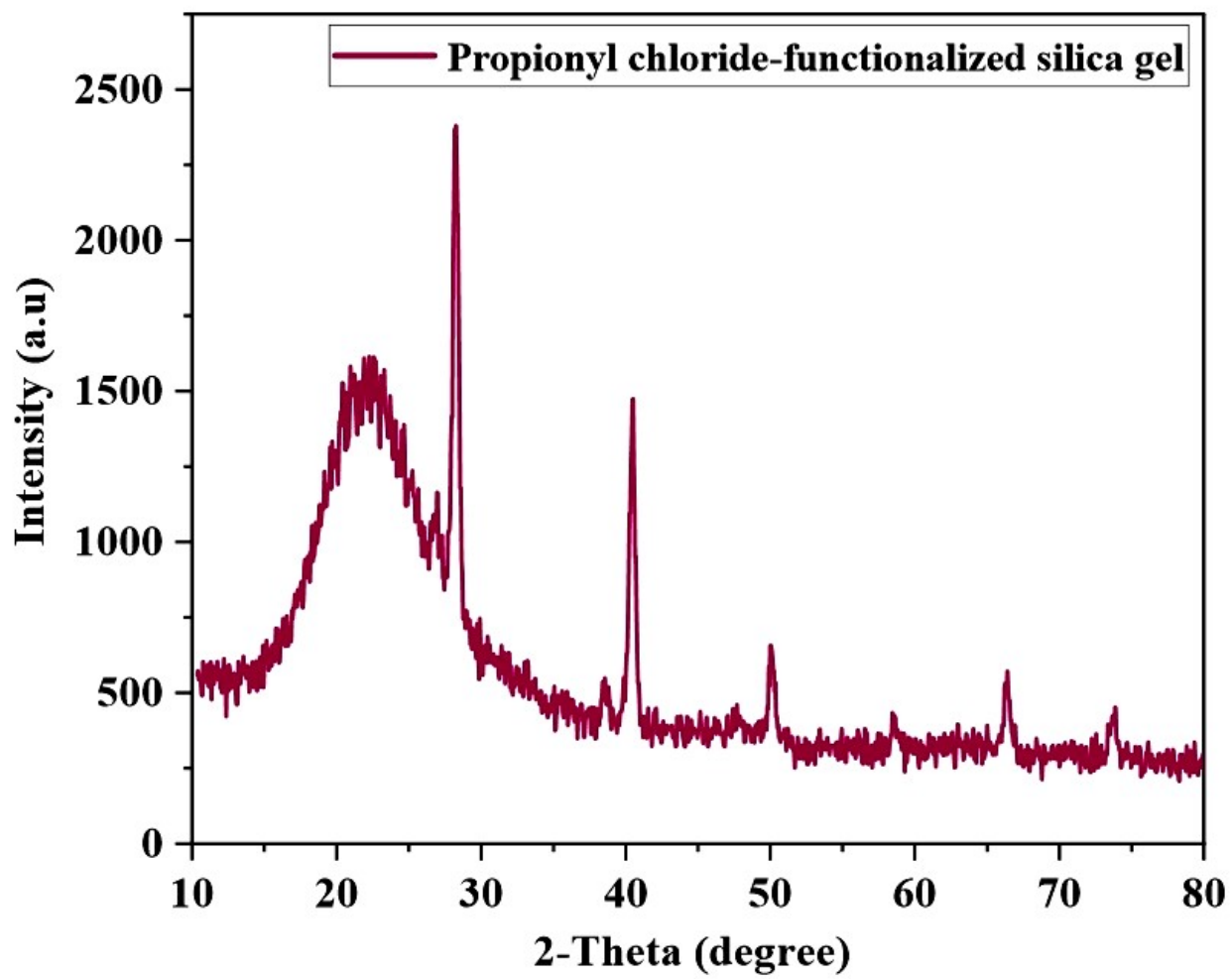


Fig. S15. XRD pattern of propionyl chloride-functionalized silica gel

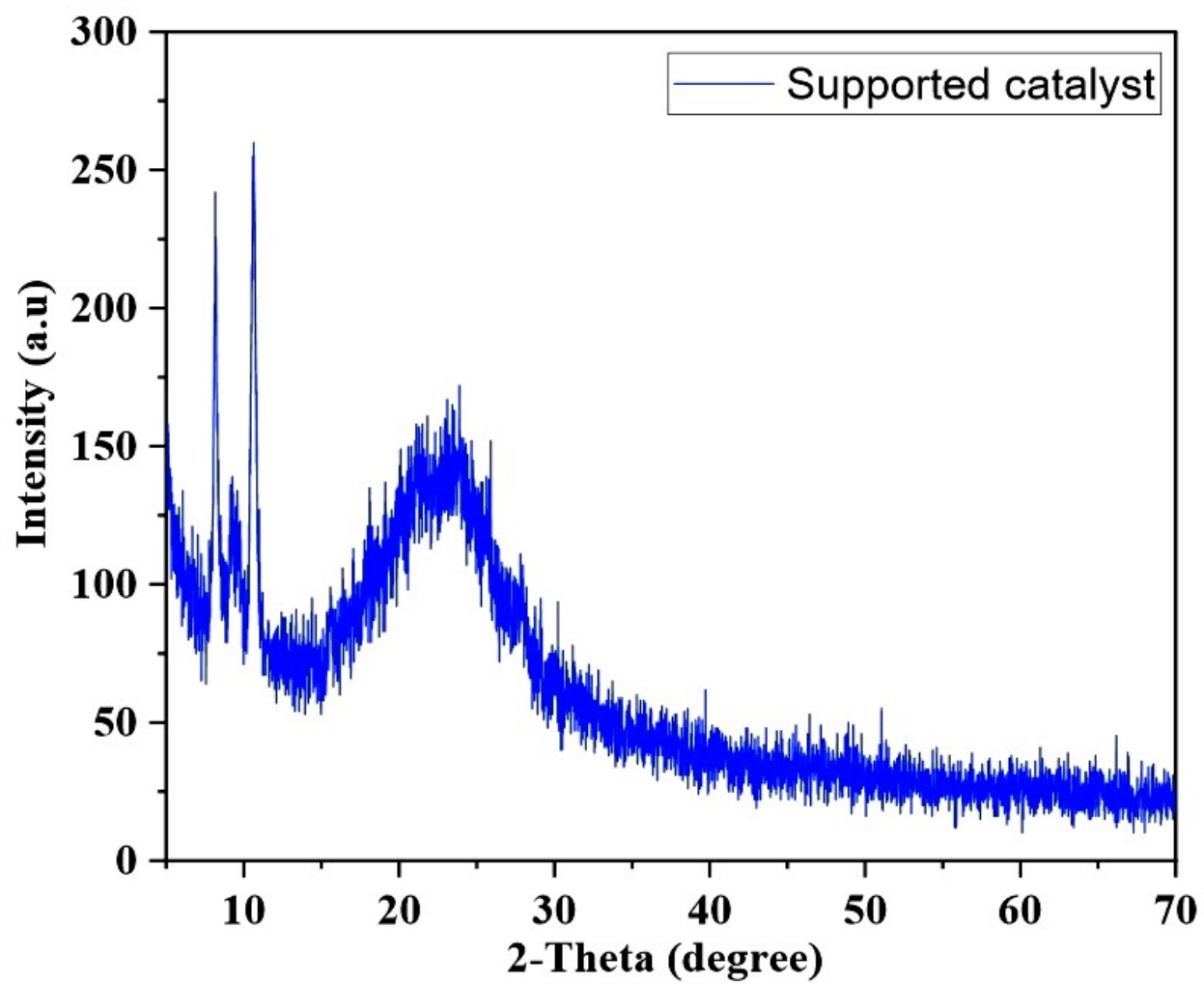


Fig. S16. XRD pattern of supported catalyst

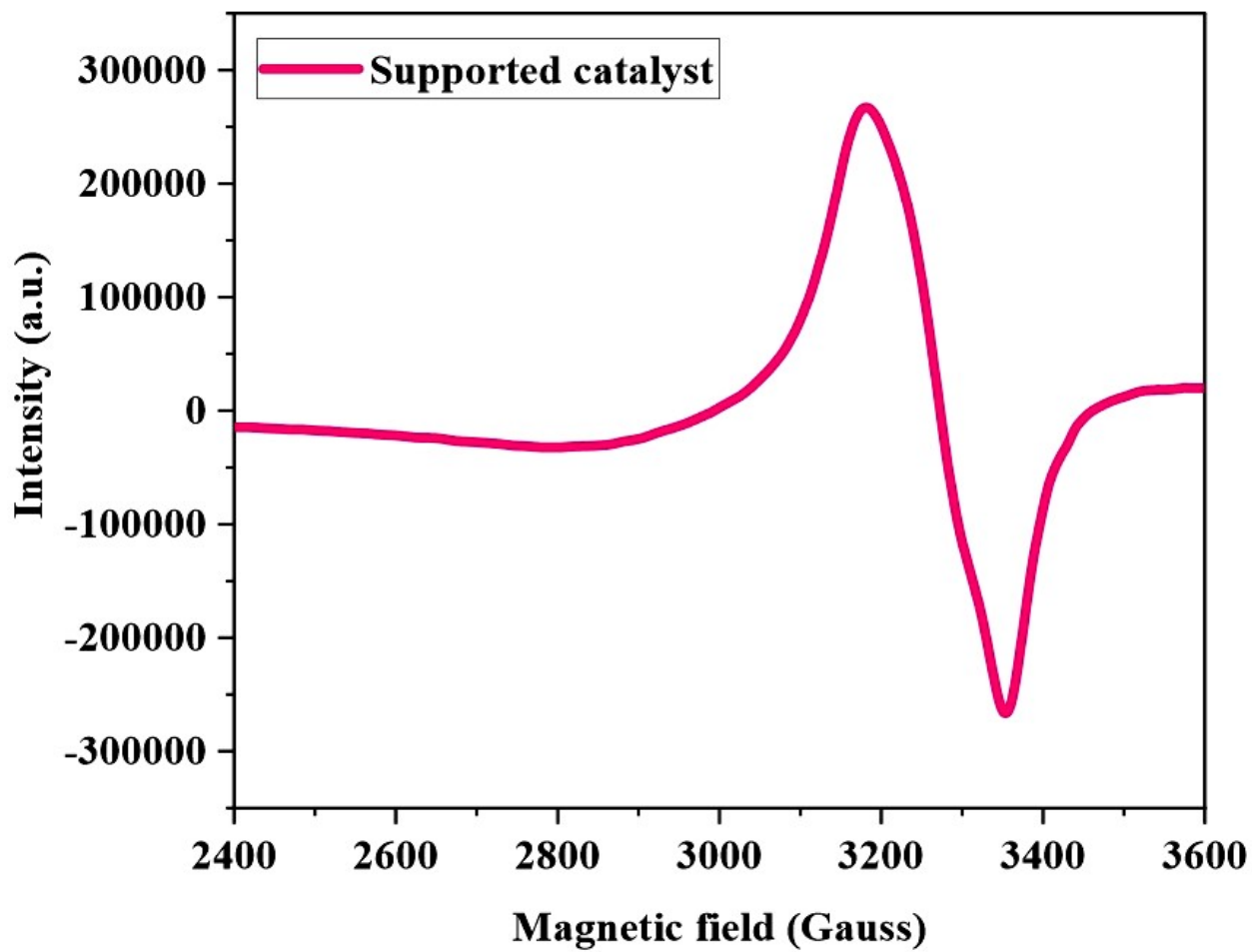


Fig. S17. EPR spectrum of the supported catalyst recorded at 77 K

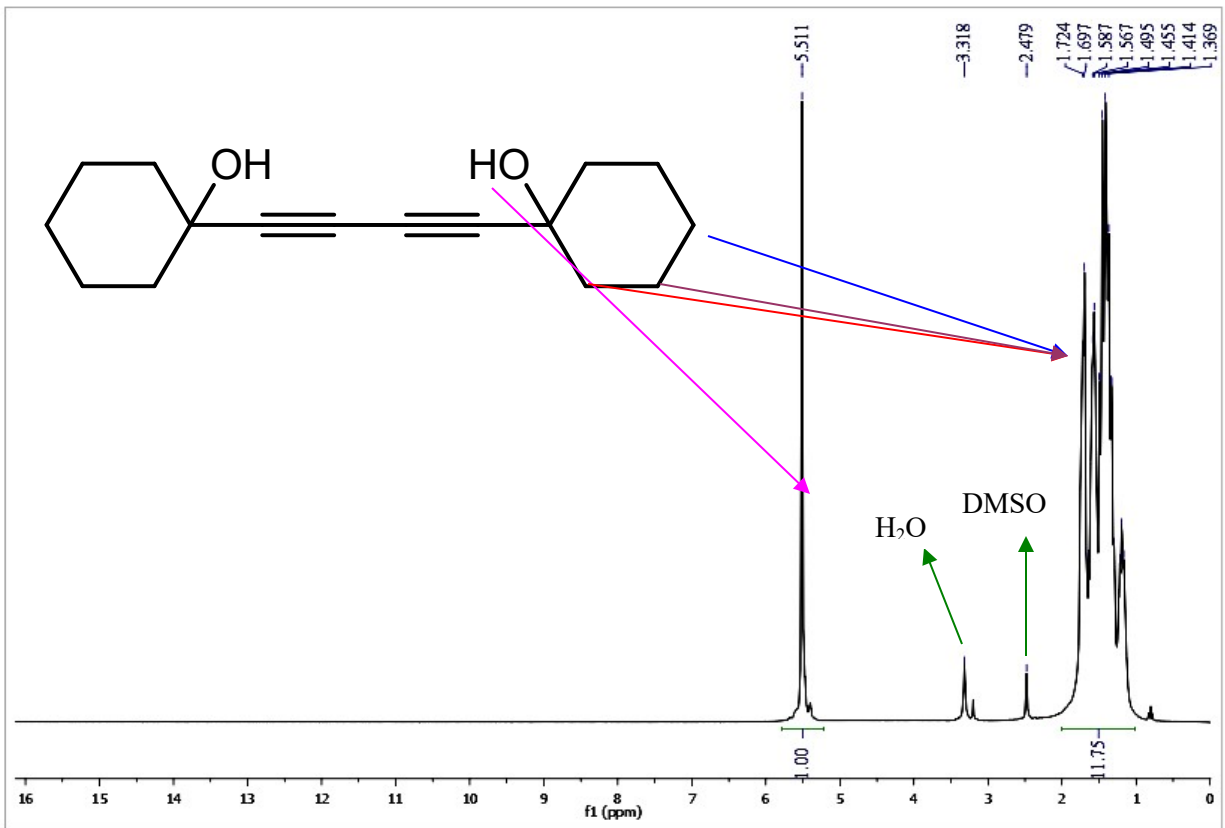


Fig. S2. ¹H NMR spectrum of CP1 in DMSO-d₆

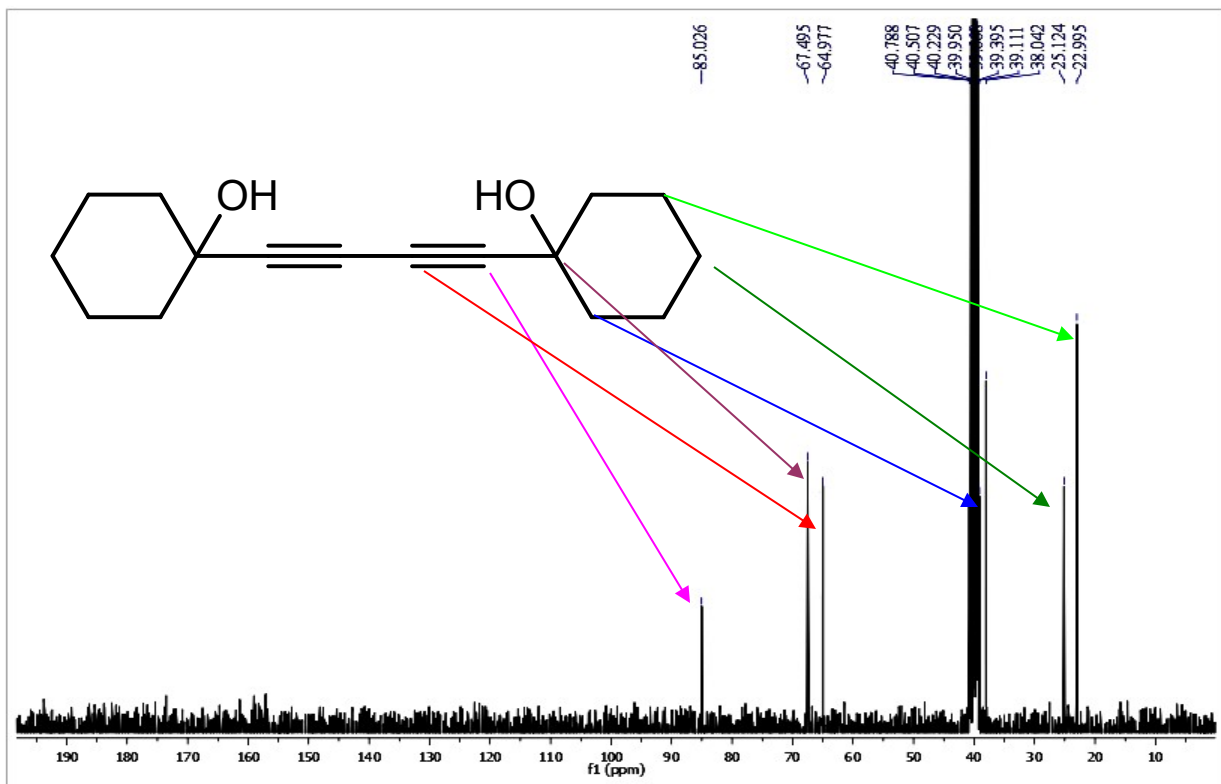


Fig. S2. ¹³C NMR spectrum of CP1 in DMSO-d₆

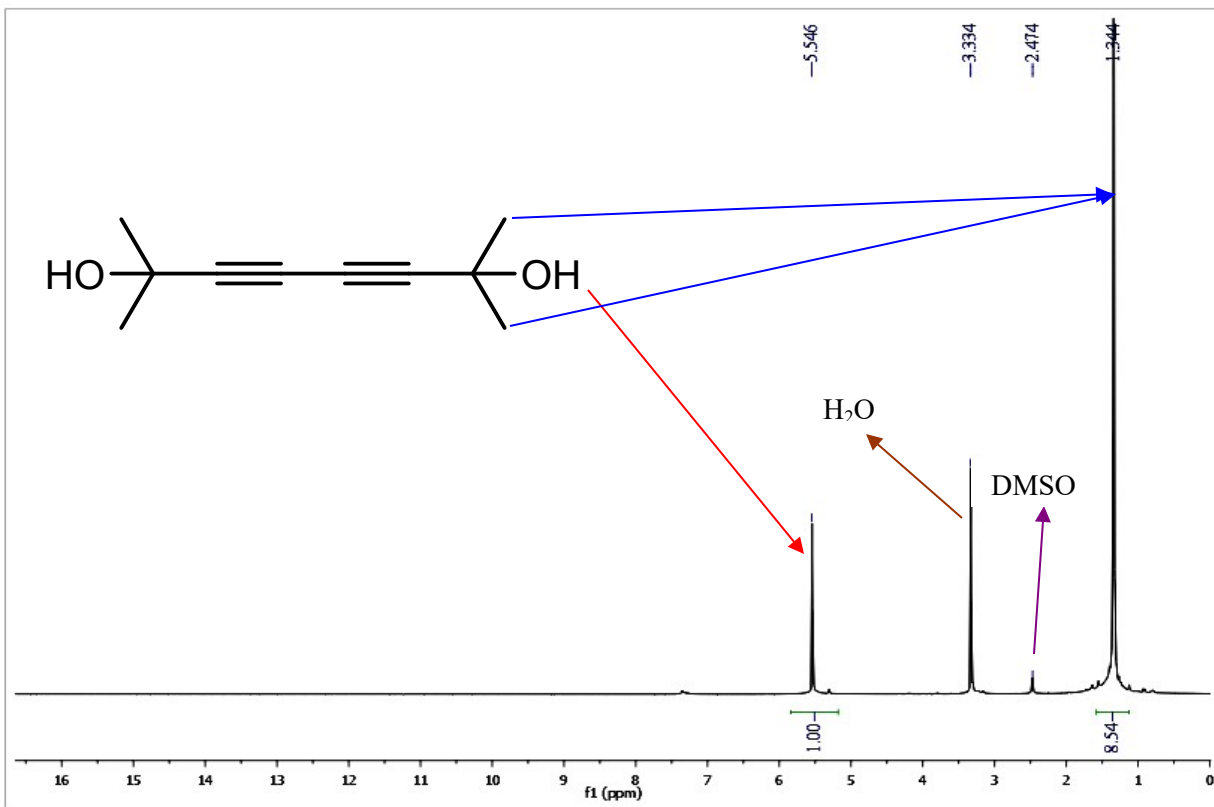


Fig. S2. ¹H NMR spectrum of CP2 in DMSO-d₆

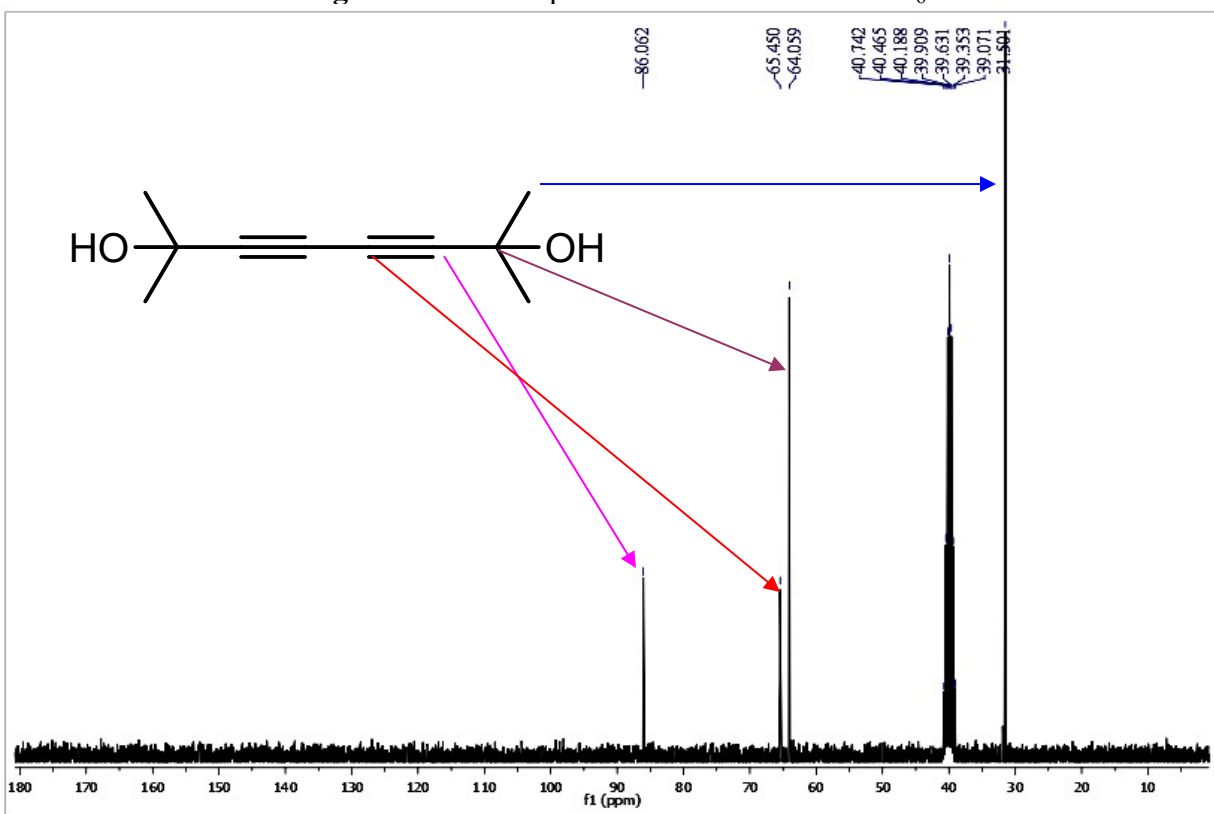


Fig. S2. ¹³C NMR spectrum of CP2 in DMSO-d₆

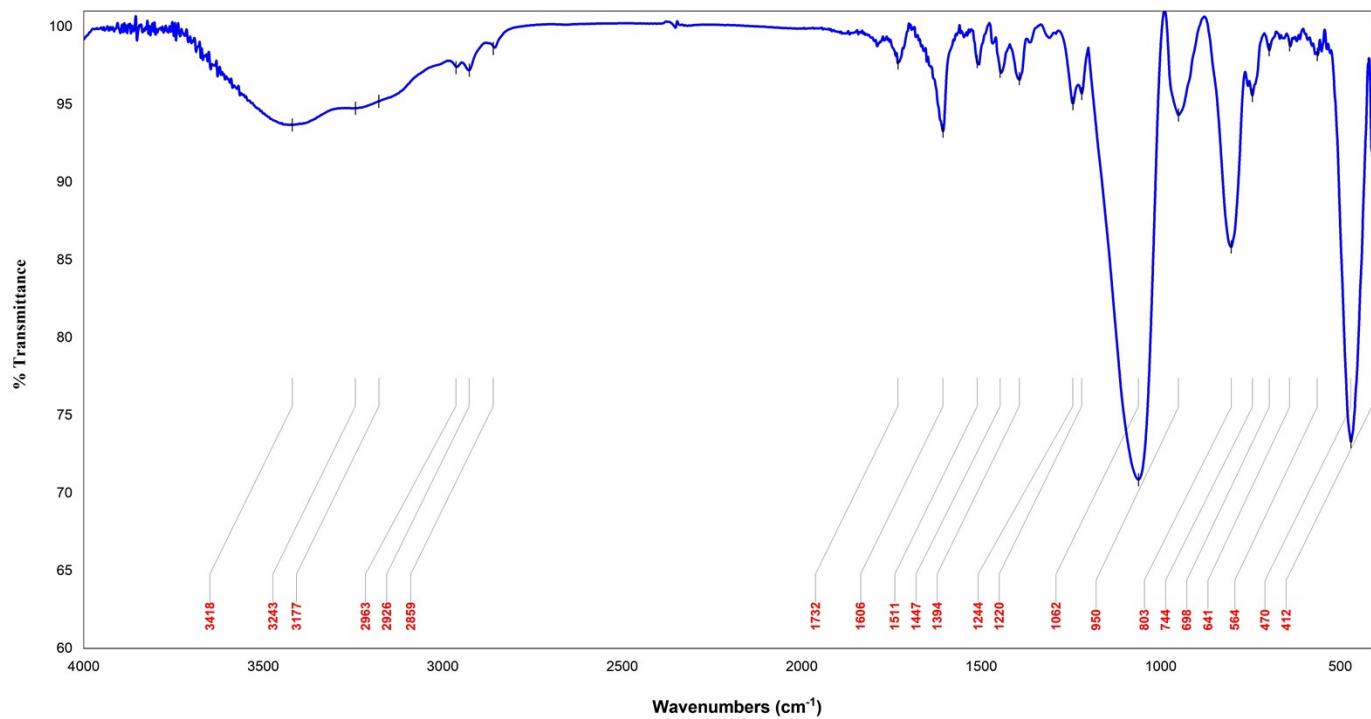


Fig. S22. FT-IR spectrum of the recovered catalyst

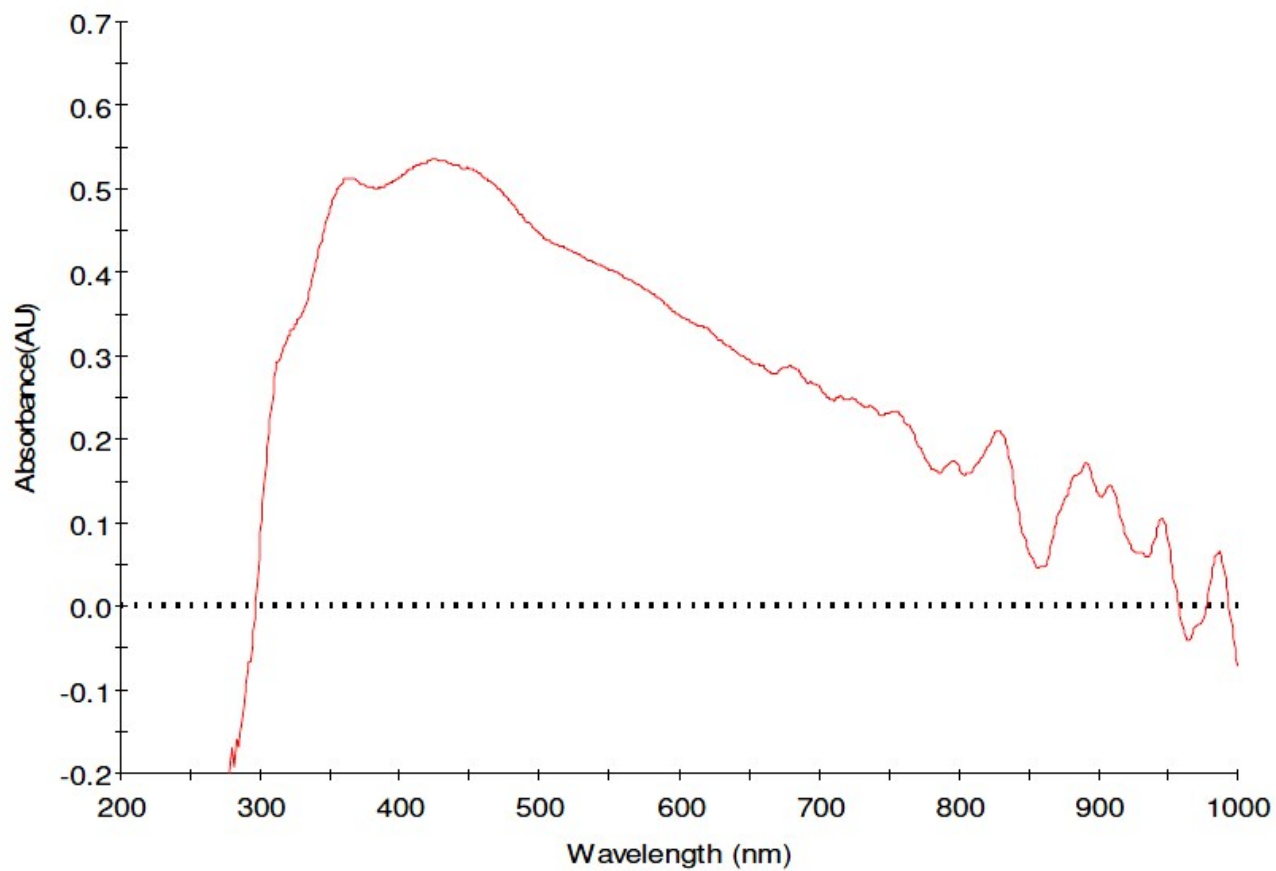


Fig. S23. DRS spectrum of recovered catalyst

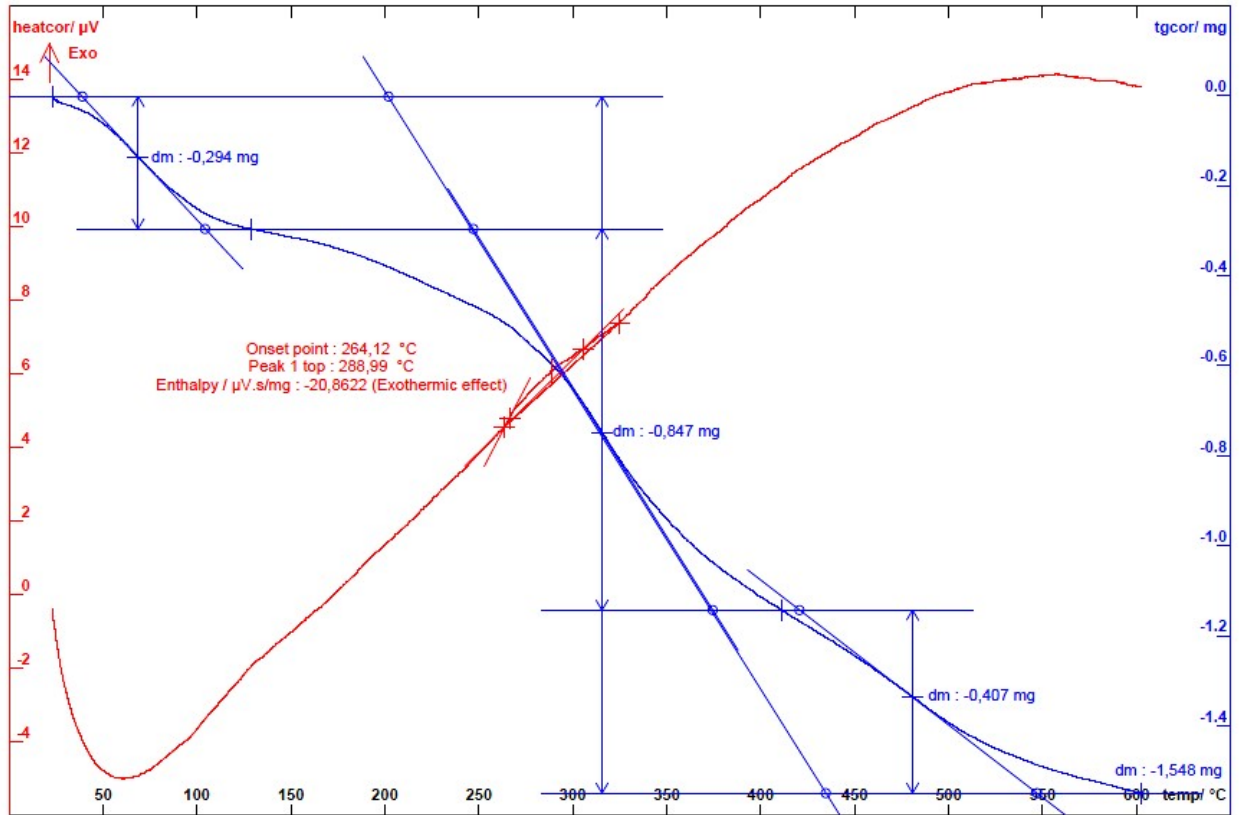


Fig. S24. TGA/DTA diagram of recovered catalyst

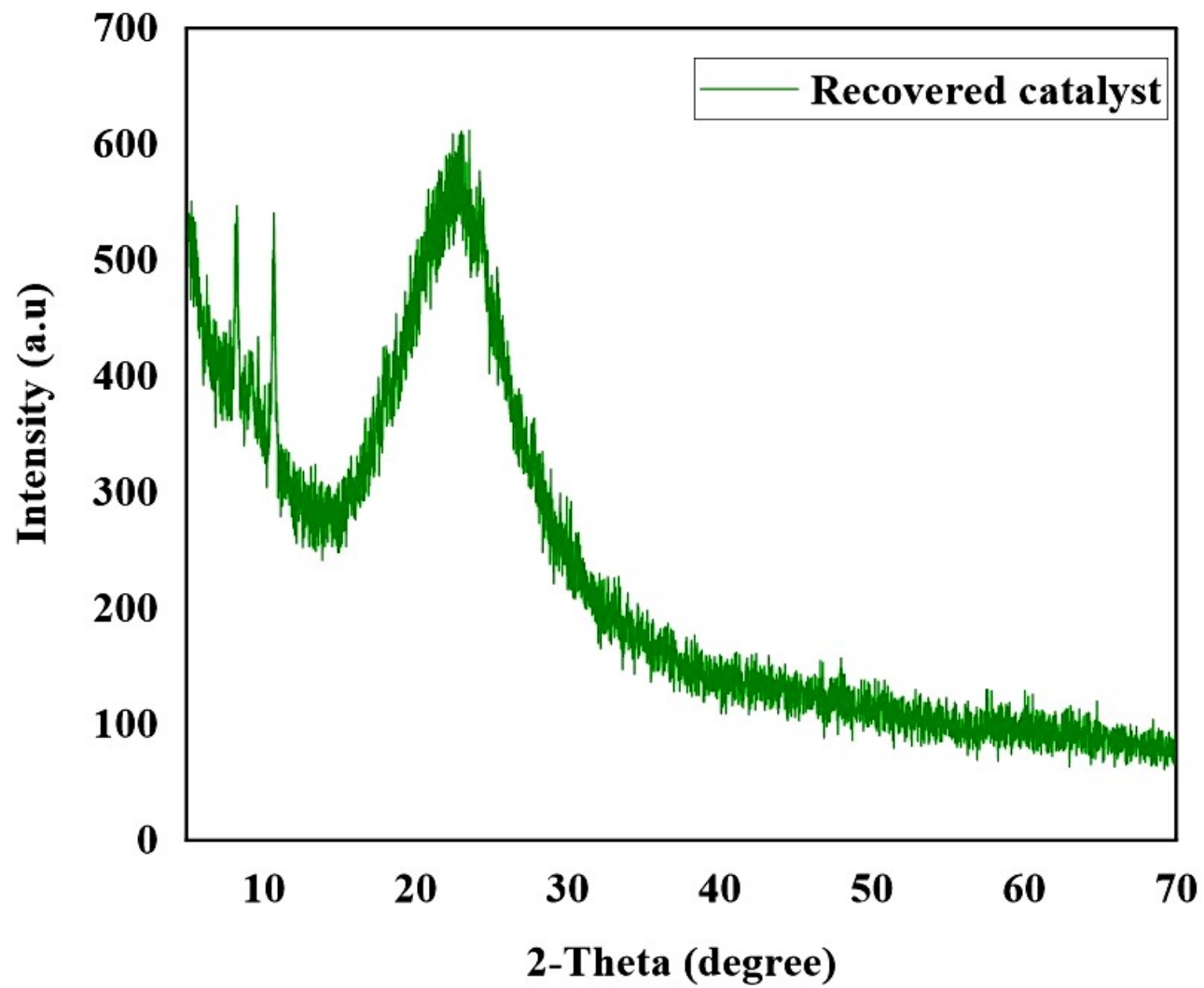


Fig. S25. XRD pattern of recovered catalyst

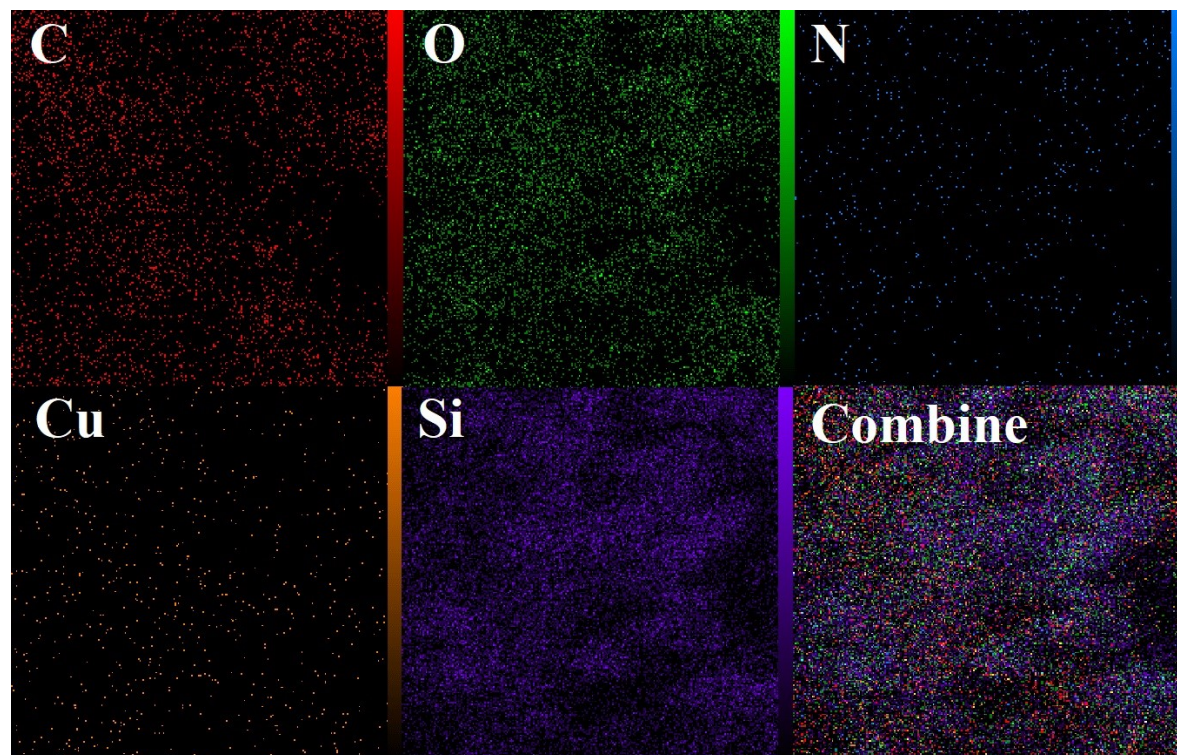
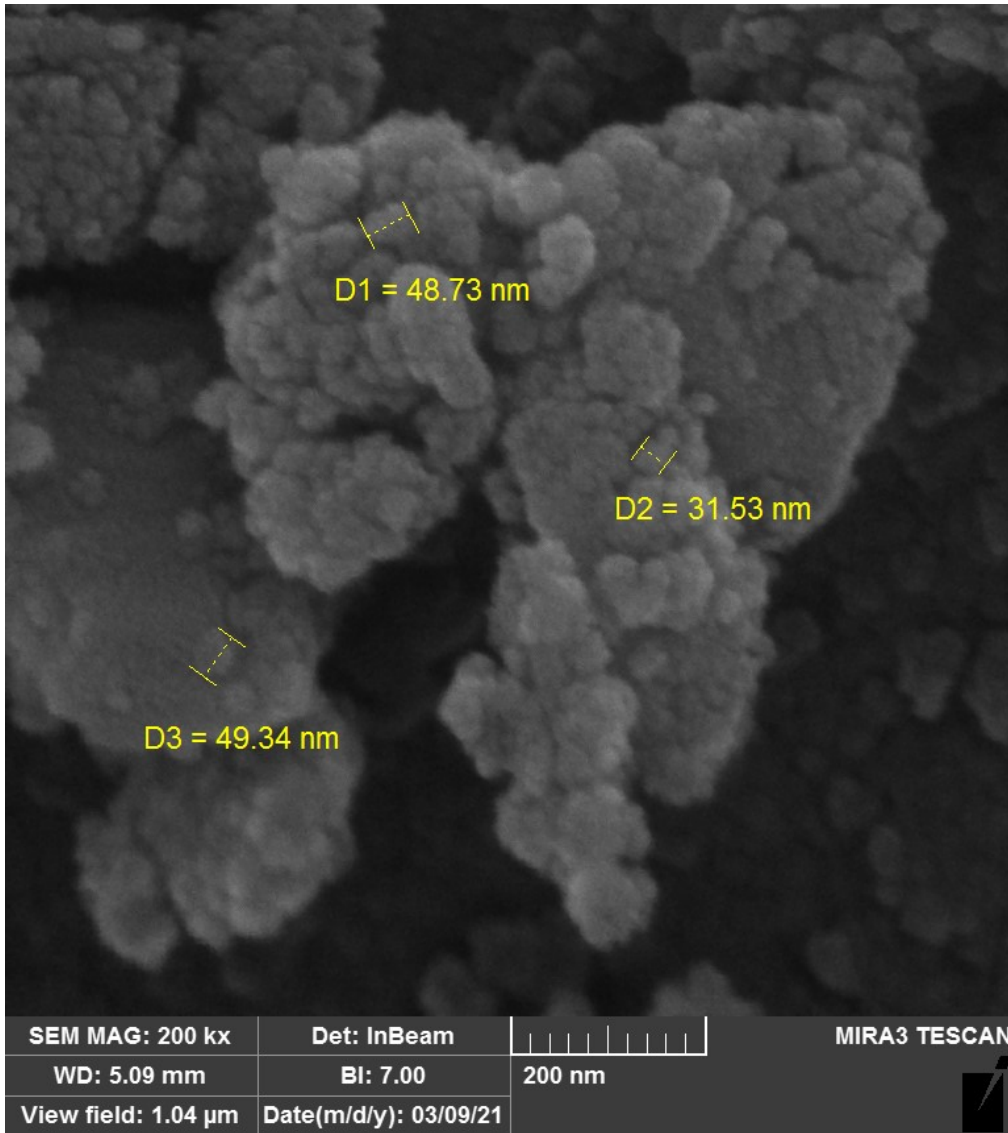
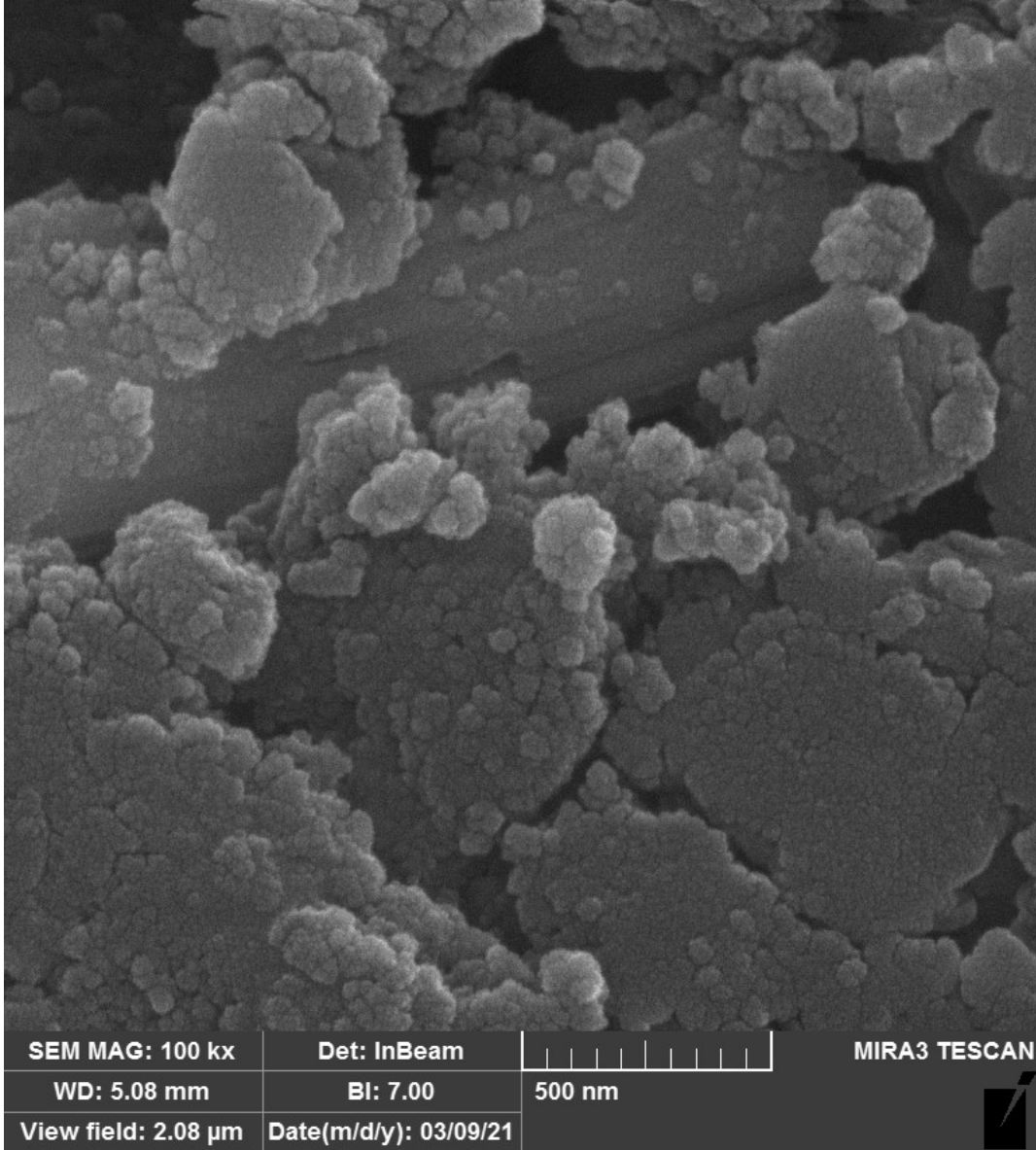


Fig. S26. EDS mapping images of the recovered **Si-Cu**





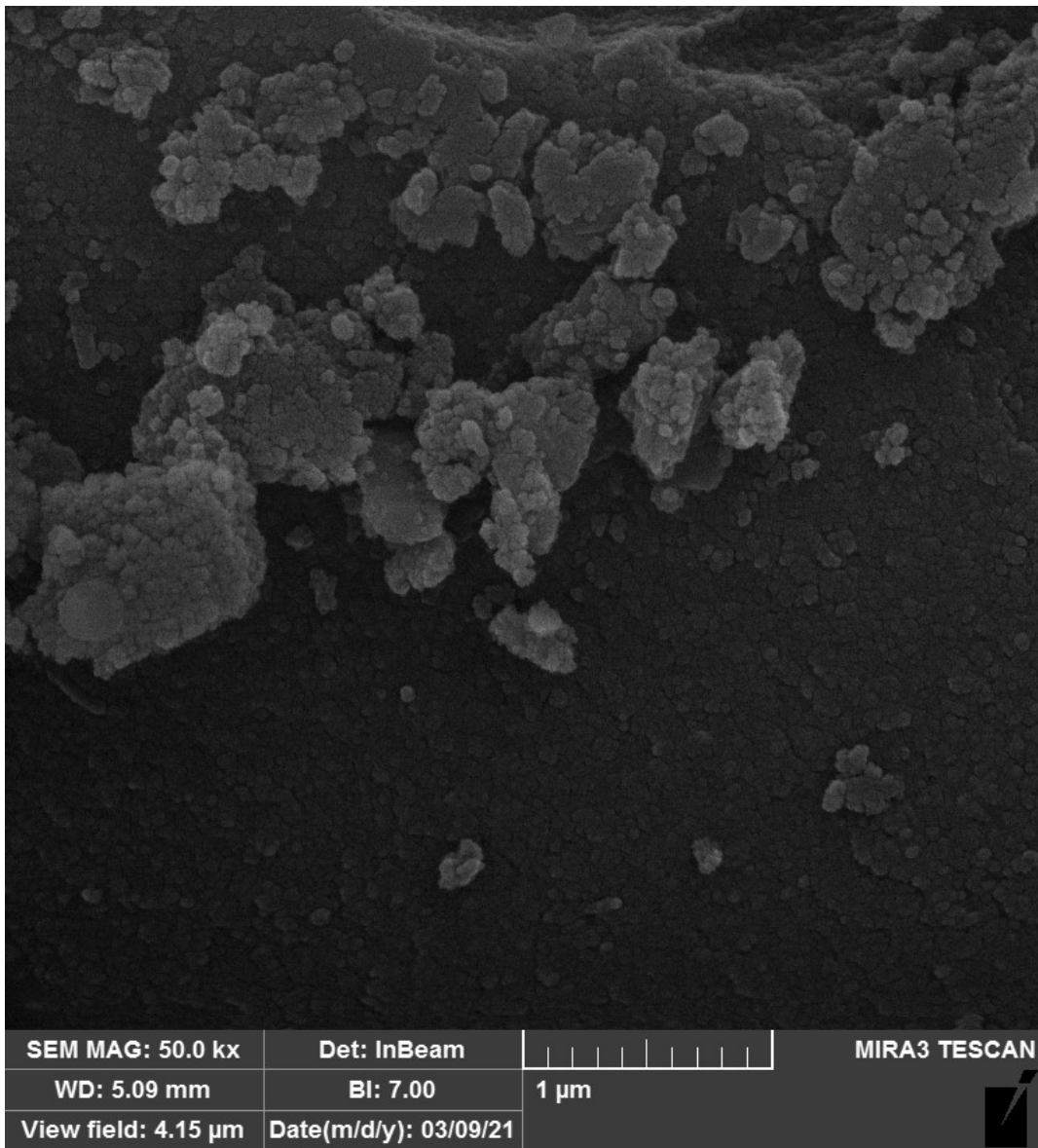


Fig. S27. SEM images of recovered catalyst

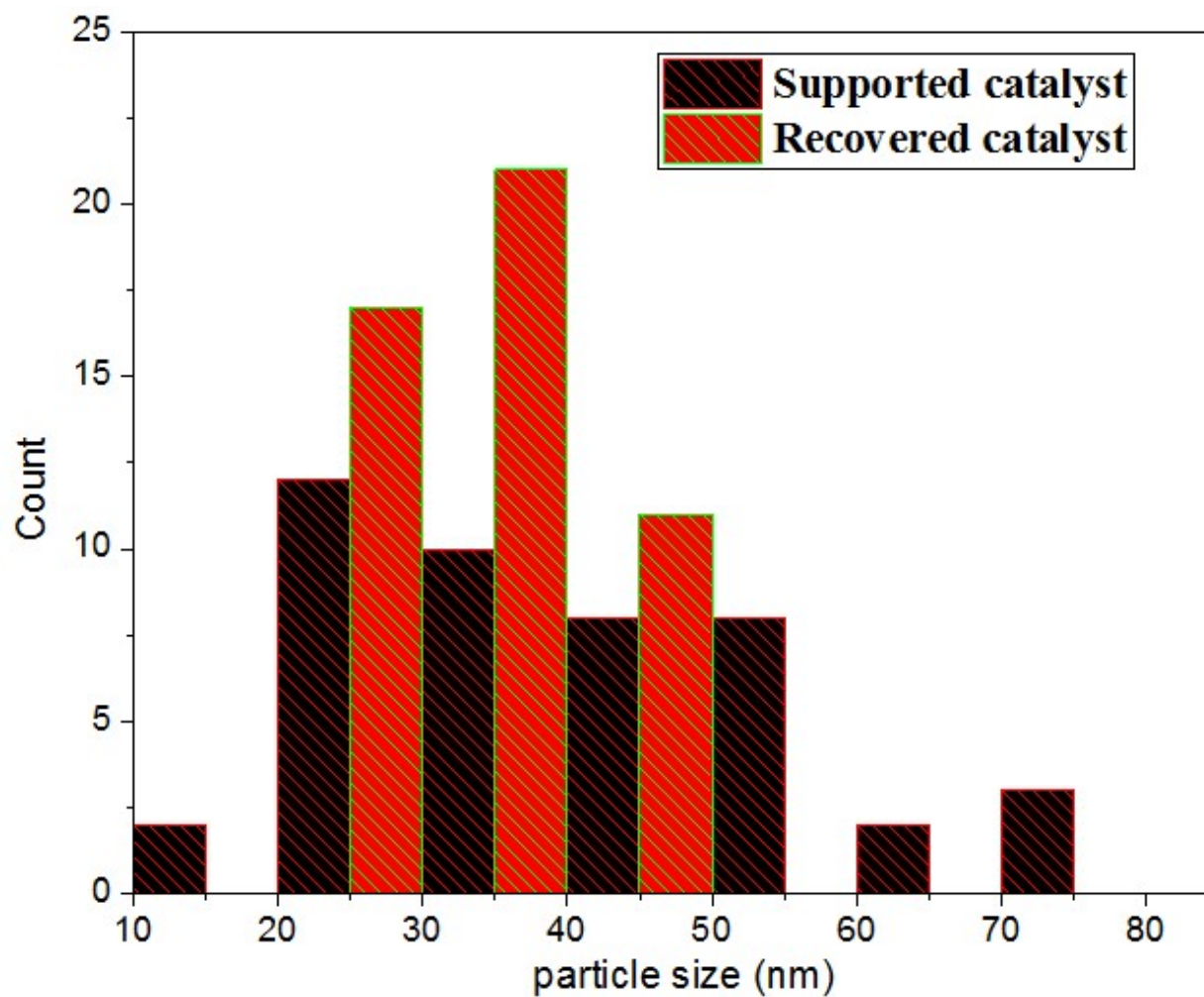


Fig. S28. Distribution of the size of particles obtained by using the analysis of variance (ANOVA) and Tukey's test

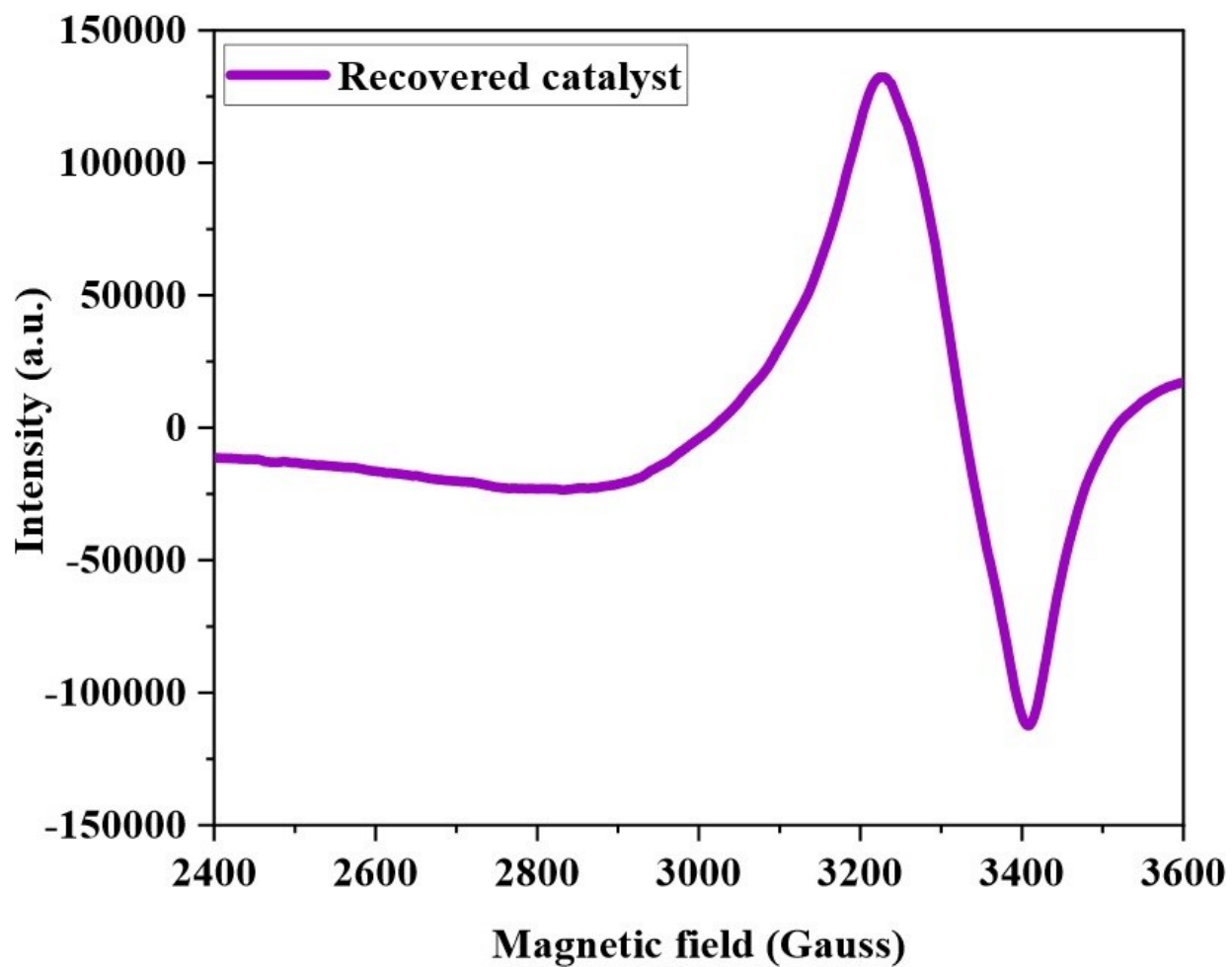


Fig. S29. The EPR spectrum of the recovered Si-Cu recorded at room temperature



Fig. 30. Green powder (Si-Cu) obtained from the reaction of compound **1** and propionyl chloride functionalized silica gel

ⁱ Agilent Technologies, 2010, *CrysAlis PRO* Version 1.171.35.15.