

An efficient approach for the green synthesis of biologically active 2,3-dihydroquinazolin-4(1H)-ones using EDTA coated magnetic copper nanocatalyst

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SUPPLEMENTARY INFORMATION (SI)

Supplementary Information (SI) includes XRD, SEM and TEM of recycled $\text{Fe}_3\text{O}_4@\text{EDTA}/\text{CuI}$ nanocatalyst; Green chemistry metric calculations, ^1H NMR and ^{13}C NMR spectra of all compounds.

Total No of Pages: 18, Total No of Tables: 0, Total number of Figures: 3.

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1. General Remarks

Chemicals and solvents were purchased from Sigma Adrich, Alfa-Aesar and Merck India Pvt. ^1H and ^{13}C spectra were recorded on a Jeol Spectrospin spectrometer at 400 MHz and 100 MHz respectively by keeping TMS as internal standard. CHNS Analyser was recorded on model Vario Micro Cube at USIC (University Science Instrument Centre), University of Delhi, Delhi, India. The X-ray diffractometer (Model No. D8 DISCOVER) at 2θ range of $2-90^\circ$ with $\text{Cu K}\alpha$ radiation. Chemical shift values were recorded in terms of δ and coupling constants (J) are in hertz (Hz). FTIR spectra were obtained on IRAffinity-1S Fourier Transform Infrared Spectrophotometer. Scanning Electron microscopy (SEM) measurement was performed on JEOL JSM 6610 at USIC, University of Delhi. Transmission electron microscopy (TEM) was obtained on a TECNAI G20 HR-TEM 200kV at SAIF (sophisticated analytical instrumentation facility), AIIMS, New Delhi, India. The elemental composition and electronic structure analysis were obtained from X-ray photoelectron spectra (XPS) of PHI 5000 Versa Probe III instrument at Institute Instrumentation Centre, Indian Institute of Technology, Roorkee – 247 667 (Uttarakhand), India. ICP-MS was measured on Agilent ICP-MS 7900 with UHMI at the ICP-MS Lab, (CRF Adopted Facility), IIT Delhi, INDIA.

2. Characterization of recycled $\text{Fe}_3\text{O}_4@\text{EDTA}/\text{CuI}$ nanocatalyst

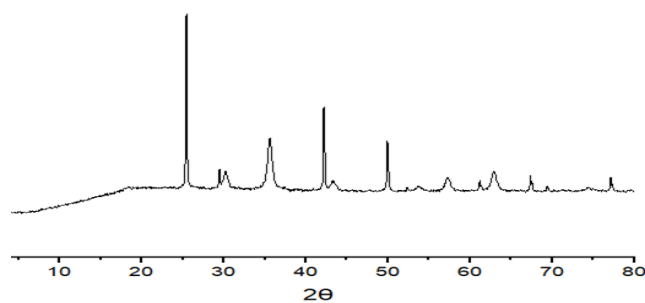


Figure S1: XRD of recycled catalyst

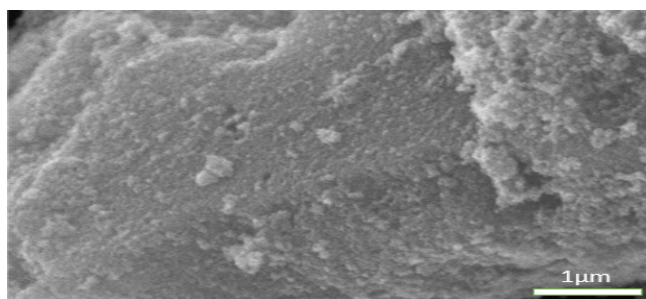


Figure S2: SEM image of recycled catalyst

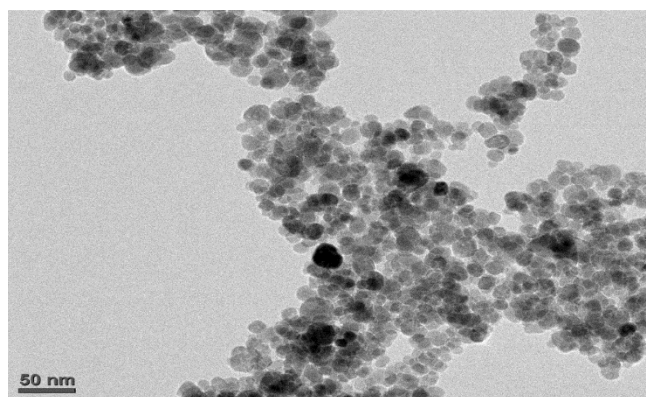
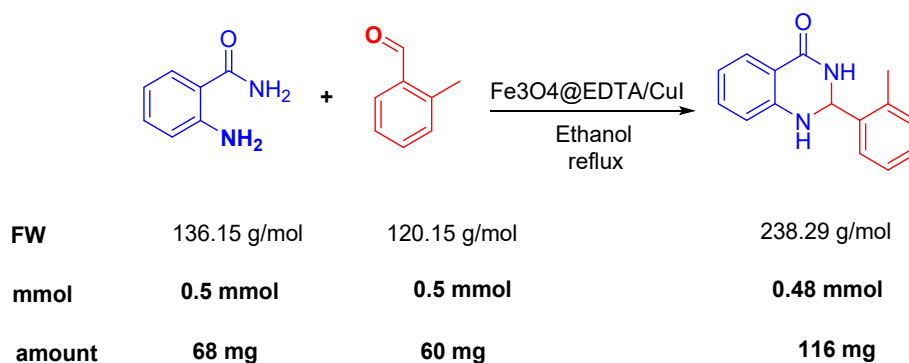


Figure S3: TEM image of recycled catalyst

3. Calculation of Green chemistry metrics

Calculation of Green chemistry metrics for 3c of our method



E-factor:

The ideal value of E-factor is zero.

E-factor = [total mass of raw materials - the total mass of product]/ mass of product.

E-factor of i = [(68 + 60)-116]/116

= 0.10.

Process mass intensity (PMI):

PMI= $\sum$ (mass of stoichiometric reactants)/[mass of product]

= (68+ 60)/ 116

= 1.10

Reaction mass efficiency (RME):

RME = [mass of product / $\sum$ (mass of stoichiometric reactants)] $\times$ 100

= [116/ (68 + 60)] $\times$ 100

= 90.62%

Carbon efficiency (CE):

CE= [Amount of carbon in product/ Total carbon present in reactants] $\times$ 100

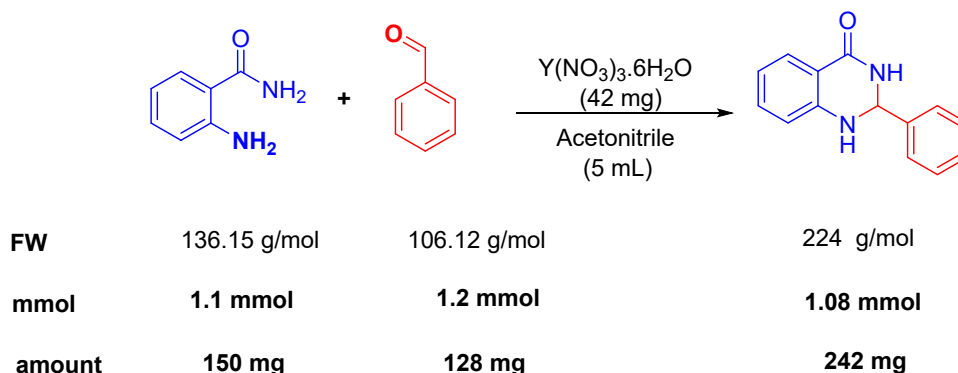
= [no. of moles of product $\times$ no. of carbons in product / (moles of a $\times$ carbons in 1i + moles of 2 carbons in 2)] $\times$ 100

= [0.48 $\times$ 15 / (0.5 $\times$ 7 + 0.5 $\times$ 8)] $\times$ 100

= [7.2 / (3.5 + 4)] $\times$ 100

= 96%

Calculation of Green chemistry metrics for previous reported method¹



E-factor:

The ideal value of E-factor is zero.

E-factor = [total mass of raw materials - the total mass of product]/ mass of product.

$$\text{E-factor of i} = [(42 + 150 + 128 + 3930) - 242] / 242$$

$$= 16.56.$$

Process mass intensity (PMI):

PMI = $\sum$ (mass of stoichiometric reactants + solvent) / [mass of product]

$$= (42 + 150 + 128 + 3930) / 242$$

$$= 17.56$$

Reaction mass efficiency (RME):

$$\text{RME} = [\text{mass of product} / \sum (\text{mass of stoichiometric reactants})] \times 100$$

$$= [242 / (42 + 150 + 128)] \times 100$$

$$= 75\%$$

Carbon efficiency (CE):

CE = [Amount of carbon in product / Total carbon present in reactants] x 100

$$= [\text{no. of moles of product} \times \text{no. of carbons in product} / (\text{moles of 1} \times \text{carbons in 1} + \text{moles of 2} \times \text{carbons in 2})] \times 100$$

$$= [1.08 \times 14 / (1.1 \times 7 + 1.2 \times 7)] \times 100$$

$$= 93\%$$

Reference

1. A.A. Khan, K. Mitra, A. Mandal, N. Baildya, M.A. Mondal, *Heteroat. Chem.*, 2017, **28**, 21379

^1H and ^{13}C NMR

