Mn doped ZrO₂ as a green, efficient and reusable heterogeneous catalyst

for the multicomponent synthesis of pyrano[2,3-d]-pyrimidine derivatives

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Contents	Pages
Materials, methods and instruments	2
Catalysts Characterization	2
Spectra's of substituted pyrano[2,3- <i>d</i>]-pyrimidine derivatives (4a-k)	3-34

Materials, methods and instruments

All chemicals and reagents required for the reaction were of analytical grade and were used without any further purification. Bruker AMX NMR spectrometer was used to record the ¹H NMR, ¹³C NMR and ¹⁵N NMR spectral values. The DMSO–d₆ solution was utilized for this while TMS served as the internal standard. TMS was further used as an internal standard for reporting the all chemical shifts in δ (ppm). The FT-IR spectrum for the samples was established using a Perkin Elmer Perkin Elmer Precisely 100 FT-IR spectrometer at the 400-4000 cm⁻¹ area. Purity of all the reaction products was confirmed by TLC using aluminum plates coated with silica gel (Merck Kieselgel 60 F254).

Characterization of Catalysts

Micromeritics Tristar-II porosity and surface area analyzer was used estimation of pore size, pore volume and surface area of the catalysts. BJH adsorption-desorption curves were obtained at -196 °C, to assess the particulate properties of the catalyst. All the catalyst materials were degassed by passing nitrogen overnight at 200 °C. Bruker D8 advance instrument with a Cu K radiation source by $\lambda = 1.5406$ was used for the X-ray diffraction data for the catalyst. Jeol JEM-1010 electron microscope and JEOL JSM-6100 microscope were used for TEM and SEM analysis respectively. In TEM analysis for particles distribution, size of 40-60 particles were averaged and with standard deviation. An emission current (100 μ A) by a Tungsten (W) filament with 12 kV accelerator voltage was employed for EDX-analysis of the SEM images. Elemental composition of the catalyst materials was established by using Inductively Coupled Plasma Optical Emission Spectrometer (Optima 5300 DV).



Spectra's of substituted pyrano[2,3-d]-pyrimidine derivatives (4a-k).









¹⁵N NMR (GHSQC) spectra of compound 4a



FT-IR spectra of compound 4a









¹⁵N NMR (GHSQC) spectra of compound 4b



FT-IR spectra of compound 4b









¹³C NMR spectra of compound 4c



¹⁵N NMR (GHSQC) spectra of compound 4c



FT-IR spectra of compound 4c



¹H NMR spectra of compound 4d









FT-IR spectra of compound 4d







¹³C NMR spectra of compound 4e



¹⁵N NMR (GHSQC) spectra of compound 4e



FT-IR spectra of compound 4e



¹³C NMR spectra of compound 4f





¹⁵N NMR (GHSQC) spectra of compound 4f



FT-IR spectra of compound 4f







¹³C NMR spectra of compound 4g



¹⁵N NMR (GHSQC) spectra of compound 4g



FT-IR spectra of compound 4g







¹⁵N NMR (GHSQC) spectra of compound 4h



FT-IR spectra of compound 4h



¹H NMR spectra of compound 4i









¹⁵N NMR (GHSQC) spectra of compound 4i



FT-IR spectra of compound 4i







¹³C NMR spectra of compound 4j



¹⁵N NMR (GHSQC) spectra of compound 4j



FT-IR spectra of compound 4j



¹H NMR spectra of compound 4k





¹³C NMR spectra of compound 4k



¹⁵N NMR (GHSQC) spectra of compound 4k



FT-IR spectra of compound 4k