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Supporting Information

Catalytic activity of microporous-mesoporous composite towards liquid phase oxidation of diphenylmethane

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General Information

Fumed silica (Sigma Aldrich), sodium aluminate (Kemphasol), sodium hydroxide (Merck), tetrapropylammonium bromide (Merck), cobaltous nitrate, CTAB (Rankem) were used for the synthesis of ZSM-5, ZSM-5/MCM-41 composite and Co loaded ZSM-5/MCM-41 composites. All the chemicals used were of analytical grade and obtained from commercial sources and used without further purification. Double distilled water was used throughout the experimental processes.

The synthesized materials were characterized by various techniques such as powder Xray diffraction, TGA, IR spectroscopy, UV-VIS DRS spectroscopy, SEM and EDX and N₂ adsorption-desorption analysis. The phase formation and crystallographic state of the synthesized samples were studied by Powder X-ray diffraction (PXRD) pattern and were recorded using a Rigaku Ultima IV X-Ray diffractometer (Cu Ka radiation, λ =1.5418 Å) at 40 kV and 40 mA. The FTIR spectra were recorded in the range 4000-400 cm⁻¹ using an IR Affinity-1 Fourier transform infrared spectrophotometer (SHIMADZU) using a KBr pallet. Thermogravimetric analysis (TGA) was done using Metter Toledo TGA/DSC1 STAR^e system in presence of N₂ atmosphere in the temperature range 50-700 °C at a heating rate of 10 °C/min. The N₂ adsorptiondesorption isotherm of the prepared samples were recorded at 77 K by using Micromeritics Tristar 3000 analyser. Prior to the measurement the samples were degassed at 110 °C for 4 h. To investigate the morphology and the presence of different elements in the synthesized samples scanning electron micrographs (SEM) and energy dispersive X -ray spectroscopic (EDX) analysis were recorded. The images were recorded in Zeiss SIGMA 300 scanning electron microscope. Diffuse reflectance UV-Vis spectra (DRS) of the Co loaded ZSM-5/MCM-41 samples were recorded with a Hitachi 4100 spectrometer in the range of 200-800 nm using barium sulphate as the standard reference for baseline correction. Finally, the products of the oxidation reaction were analysed using Perkin Elmer Clarus 500 gas chromatograph equipped with Elite-1 series of capillary column using flame ionization detector (FID). ¹H and ¹³C spectra of the isolated product were recorded on a Bruker 500 MHz spectrometer. Chemical shifts are given in δ units relative to the tetramethylsilane (TMS) signal as an internal reference in CDCl₃.

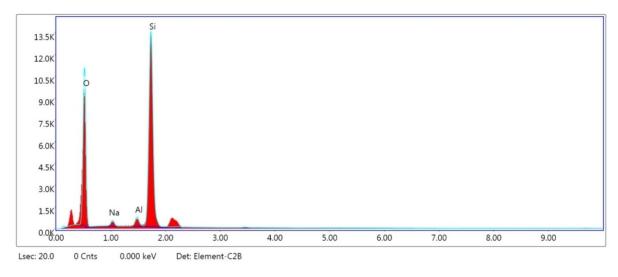


Fig1: EDX spectrum of ZSM-5

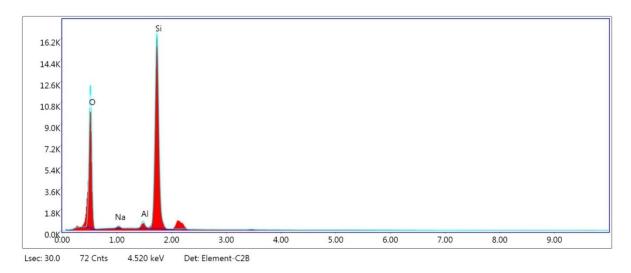


Fig 2: EDX spectrum of ZSM-5/MCM-41 composite

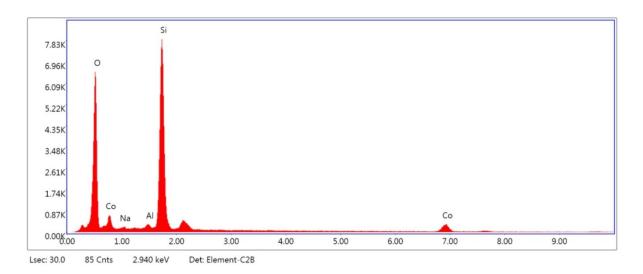


Fig 3: EDX spectrum of CZM-5

Table 1: Amount of metal ion	(Co) present in th	e prepared samples	s (wt%)(EDX data)
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Entry	Sample	Amount of metal ion (Co) in the samples (wt%)
	designation	(EDX data)
1	CZM-1	0.97
2	CZM-3	2.45
3	CZM-5	4.29
4	CZM-8	7.20
5	CZM-10	8.95

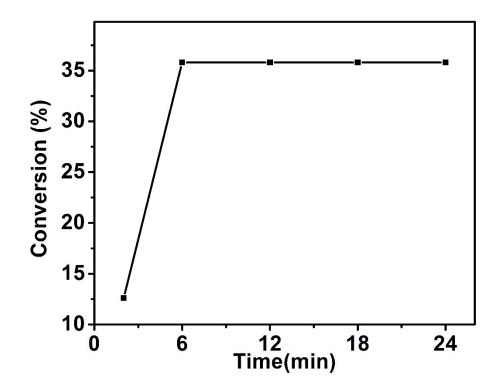


Fig 4: Leaching test for CZM-5 catalyst for conversion of diphenylmethane

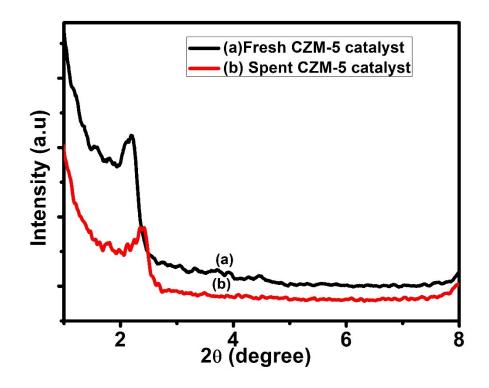
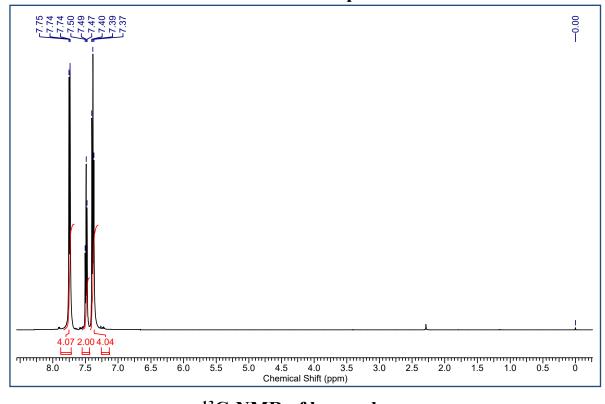


Fig 5: XRD pattern of (a) fresh CZM-5 (b) Spent CZM-5 catalyst

Experimental procedure

To a solution of DPM (6 mmol) in acetonitrile (5 mL), TBHP (6 mmol) was added and the temperature was set to 80 °C. Then 9 wt% of the catalyst w. r. t DPM was added slowly to the reaction mixture and the reaction was allowed to run for 24 h under reflux condition. After completion of the reaction, the reaction mixture was diluted with dichloromethane and filtered. The organic layer was washed several times with water, separated and treated with anhydrous Na₂SO₄. The solvent was removed under reduced pressure and the residue was purified by column chromatography using hexane:ethyl acetate (9:1) as eluent in silica gel 100-200 mesh to afford the pure product benzophenone as white solid with 68% isolated yield [m.p. 45-47 °C; ¹H NMR (500 MHz, CDCl₃) δ 7.75-7.74 (m, 4H), 7.49 (t, J =7.37 Hz, 4H), 7.39 (t, J = 7.89 Hz, 4H); ¹³C NMR (125 MHz, CDCl₃) δ 196.4, 137.6, 132.4, 130.0, 128.3].

Copies of ¹H and ¹³C NMR spectra:



¹H-NMR benzophenone

