Synthesis, Characterisation and Catalytic Applications of sulphated nano zirconia

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Sulphated nanocrystalline zirconia particles is prepared by template, micro emulsion, sol-gel and co-precipitation methods. Powder XRD showed that the peaks of the diffractogram are in agreement with the theoretical data of zirconia and the crystallite size is calculated by Sherrer equation. The calcinations at different temperatures showed that lower temperature help to form the catalytically active tetragonal phase in nanometric scale. The tetragonal phase is retained up to 500\(^0\)C. TG/DTA analyses of zirconia particles showed that the phase formation range is 350-400\(^0\)C. Temperature Pragrammed Desorption of ammonia shown that the synthesized ZrO\(_2\) surface is moderately acidic and showed maximum acidity below 300\(^0\)C and possesses negligible acidity above 500\(^0\)C. Sulphation increases surface acidity. TEM images confirm the nanometric crystallite site of the sample and SEM reveals that the sample is non-uniform shapeless crystallites with 0.2-1.0 \(\mu\)m cross sectional size. Most of the pores are found in meso region. UV-DRS spectra showed very strong absorption bands in the wavelength range 200-300 nm for zirconia nanocrystals and absorption edge of the inter band transition is blue shifted. IR bands in the frequency region of 1200-900 cm\(^{-1}\) showed the presence of sulphate groups on the surface. The catalytic activity study showed that sulphated zirconia can be used as an alternative to the Friedel-Crafts catalyst due to its acidic nature. The surface acidity and surface area have a deciding hand in the catalytic performance of sulphated nanocrystalline zirconia in the Friedel-Crafts benzylation reaction. Catalysts with moderate surface area can
accommodate sufficient sulphate ions at the catalyst surface and polarize the reactant benzyl chloride. The reaction appears to proceed through an electrophilic mechanism, involving the reaction of benzyl chloride with the acidic oxide catalyst resulting dechlorination and subsequent formation of electrophile.

Papers published


Conference Papers

5. Surface and morphological study of nanocrystalline sulphated zirconia prepared by microemulsion and sol-gel methods, *UGC sponsored National seminar on Recent Advances in Spectroscopy 31st July & 1st August, Mar Athanasius College, Kothamangalam, 2014*