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RESEARCH ARTICLE

Iron Oxide Impregnated SBA-15: Synthesis-Characterization and Its Catalytic Application in the Benzoylation of Benzene

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Trivalent iron impregnated SBA-15 (Fe-SBA-15) molecular sieve materials are prepared by a simple wet impregnation method. The formation of uniformly distributed iron oxide on the SBA-15 framework is evident from powder XRD studies. Furthermore, the resultant materials are systematically characterised using FT-IR, N_2 sorption and thermogravimetric analysis. The absence of X-ray diffraction peaks corresponding to iron oxide suggests that it is uniformly distributed in the SBA-15 channels. The synthesised materials are studied for the benzoylation of benzene and show good catalytic activity.

Keywords: Mesoporous Molecular Sieves, SBA-15, Wet-Impregnation, Benzoylation, Iron-Oxide.

1. INTRODUCTION

Friedel-Crafts acylation[1] is an important class of reactions in organic synthesis. The products of this reaction are intermediates in different fields, including pharmaceuticals, fine chemicals,[2] sorption,[3,4] dye degradation,[5] fragrances[6] and agrochemicals.[6] Traditionally, Friedel–Crafts acylation is catalysed in a homogeneous medium,[7] using hazardous soluble Lewis acids (e.g., $AlCl_3$, $TiCl_4$ or $FeCl_3$) or strong mineral acids (e.g., HF or H_2SO_4).[7] However, homogeneous catalysis requires tedious work to separate the products.[7] In addition, the formation of various side products and huge waste effluent pose severe environmental challenges.[7]

Alternatively, heterogeneous catalysis is of great industrial importance owing to its ease of handling, separation and reusability.[5] Several solid acid catalysts viz., ZSM-5,[8] clays,[9] HY zeolite,[10] Mg-hydrotalcite,[11] (mesoporous silica SBA-15, HMS and MCM-41) containing different metal ions[12–30] such as Al,[12,13] Ti,[14] Pt,[15] W,[15] Fe,[16,17] Pd,[18] Zr,[19] Mo,[20] Ni,[20] Ga,[8] Tl,[8] Cu[21] and In[21]) have been extensively studied and are efficient catalysts for Friedel-Craft reactions. In addition, mesoporous silica materials, in particular SBA-15, have received greater attention owing to their thick-walled 2D hexagonal pore walls with high surface areas and pore volumes, making them potential candidates for the benzoylation of aromatics.[22–24] Trivalent iron on inorganic frameworks has a range of applications in various fields, such as catalysis,[16,17] batteries,[25] gas sensors[26] and drug delivery.[27] It is of interest to introduce trivalent iron oxide on the external surface of the SBA-15 framework and explore its catalytic application for Friedel-Craft acylation.

In the present work, we report the preparation of iron oxide supported on ordered mesoporous SBA-15 using a wet impregnation method. The resultant materials were thoroughly characterised and utilised for the liquid phase benzoylation of benzene.

2. EXPERIMENTAL PROCEDURE

The typical synthesis of iron oxide loaded SBA-15 involved two primary steps, the synthesis of SBA-15 and the wet-impregnation of the trivalent iron source on SBA-15 and subsequent calcination at 550 °C.

2.1. Synthesis of SBA-15

SBA-15 was synthesised according to the procedure described elsewhere, using Pluronic P123 triblock copolymers (polyethylene oxide)-poly(propylene oxide)-poly(propylene oxide) (Aldrich)) $EO_nPO_{70}EO_n$

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