Promotion of organic reactions by interfacial hydrogen bonds on hydroxyl group rich nano solids

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Supporting Information

Experimental Details:

The preparation of metal hydroxides and metal oxides

The iron hydroxide nanoparticles were prepared via adding ammonia solution (25 %, 25 ml) to iron nitrate solution (0.5 M, 20 ml) quickly with vigorous stir. Then the brown deposition was gathered by centrifugation and dried in a 110 °C oven. The iron hydroxide with low surface area was prepared via adding ammonia solution to iron nitrate solution slowly, and the precipitation was dried in a vacuum oven at 60 °C. α -Fe₂O₃ was prepared by calcinations of low surface area iron hydroxide at 600 °C for 3h. Similar method was used to prepare γ -Al₂O₃ with different surface areas, except that both samples were calcined at 600 °C for 4 hours to generate γ -Al₂O₃.

For zinc hydroxide, 1.5g Zn(NO₃)₂.6H₂O and 0.8g NaOH were dissolved in 15ml distilled water separately. Then two solutions were mixed together and white deposit formed immediately. The white deposit was gathered by centrifugation and was washed three times by distilled water followed by drying at room temperature overnight. Zinc oxide was produced by the calcination of zinc hydroxide at 550°C for 5h in a furnace.

To prepare $Fe(OD)_3$, about 100 ml/min. ammonia gas was bubbled into 10 ml D_2O for 10 minutes, then the resulted NH₃.D₂O solution was added to anhydrous $Fe(NO_3)_3$ solution in D₂O as mentioned . The brown participation was recovered and washed with D₂O, then was dried for reaction.

In a typical experiment, 200 mg solid, 53 mg 2-methylindole (1) and 87 mg p-benzoquinone (2) were mixed with 2 ml THF. The mixture was then stirred at room temperature for certain time. After separating the solid by centrifugation, the clear solution was analyzed by GC or GC-MS. In some experiments, the product 3 was recovered and purified by a silica chromatograph column to obtain quantitative yields of each reaction.

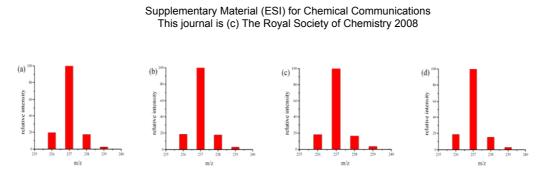
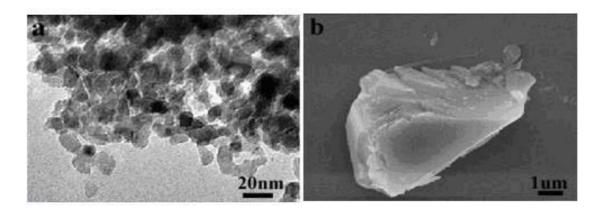


Figure S1: In order to study H/D exchange, a set of control experiments were done. (a) $Fe(OH)_3$; (b) $Fe(OD)_3$; (c) H_2O and (d) D_2O were used, and the molecular ion mass patterns of compound 3 in each experiment are showed, indicating no deuterium incorporation.



FigureS2: (a): TEM of iron hydroxide nano particles, and (b): SEM of iron hydroxide with low surface area