

Iron-Catalyzed Cross-Coupling Reaction: Recyclable Heterogeneous Iron Catalyst for Selective olefination of Aryl Iodides in poly(ethylene glycol) Medium

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Electronic Supplementary Information

Aryl iodides were prepared according to our previous procedure:

Iodoaryl derivatives synthesized from aniline derivatives using aryldiazonium silica sulfates according to our previous procedure¹ Aniline derivatives (2 mmol), silica sulfuric acid (1.5 g) and NaNO₂ (4 mmol) were ground with a pestle in a mortar for a few minutes. Two drops of water were added and the reaction mixture was ground until gas evolution stopped. Next, KI (5 mmol) was added to the mortar and grinding was continued. The mixture was diluted with EtOAc (12 mL) and filtered after vigorous stirring. The residue was extracted with EtOAc (3 × 12 mL) and the combined organic layer was washed with a 10% aq Na₂SO₃ solution and then dried over anhydrous Na₂SO₄. The solvent was evaporated in vacuum to afford desired aryl iodides.

The XPS analysis was used to detect the oxidation state of all species in the catalyst. Fig. S1 shows the survey spectrum of catalyst. From Fig. S1, it is obvious that Element C was present in the catalyst. Fig. S2 illustrates the C_{1s} spectrum, in which the peak centered at 292 eV is attributed to C species, which originated from the acetyl acetone and propyl moiety, suggesting that acetyl acetone existed in the catalyst. Fig. S3 displays the XPS spectrum of Fe_{2p}. The peak at 711 eV is attributed to Fe III species. From XPS analysis, it can be deduced that the iron(III) species are present in the catalyst composition. Figures S4 and S5 showed the XPS spectra of O_{1s} and Si_{2p} respectively.

Figure 6S shows the XRD patterns of SiO₂-FeIII (acac) catalyst, compared with original Fe(acac)₃.

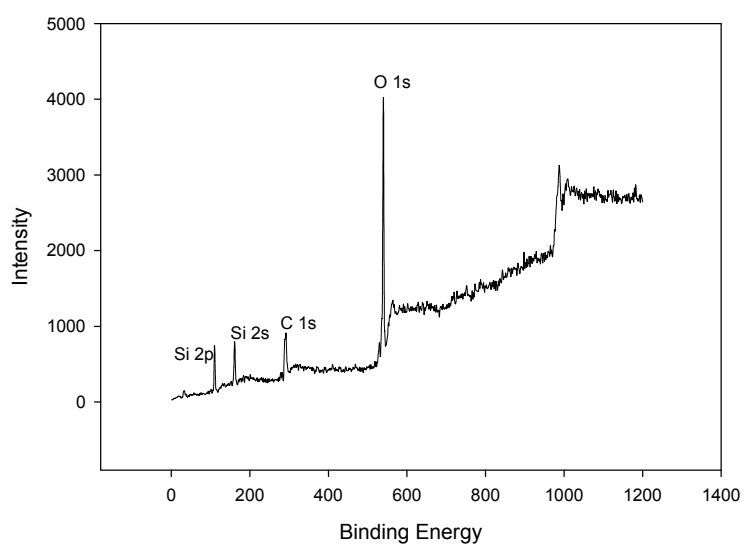


Fig. S1 Survey XPS spectrum of SiO₂-FeIII (acac)

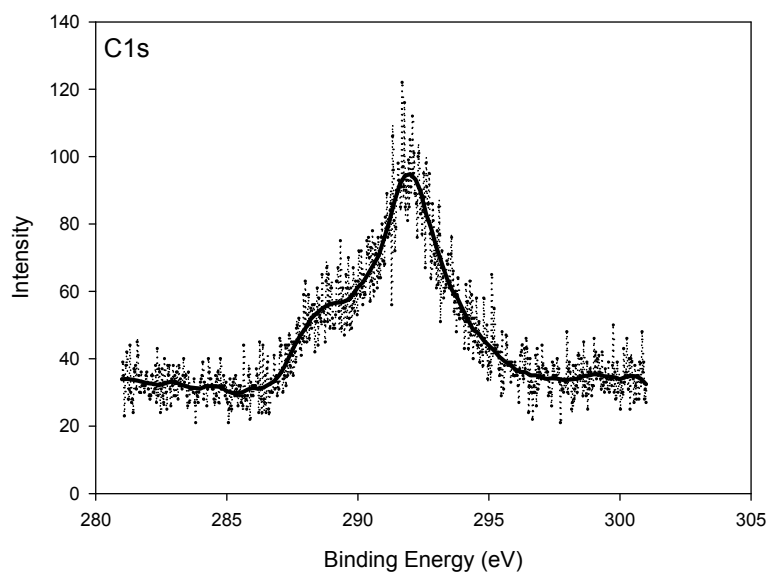


Fig. S2 XPS spectrum of the carbon 1s edge of the SiO₂-FeIII (acac) sample

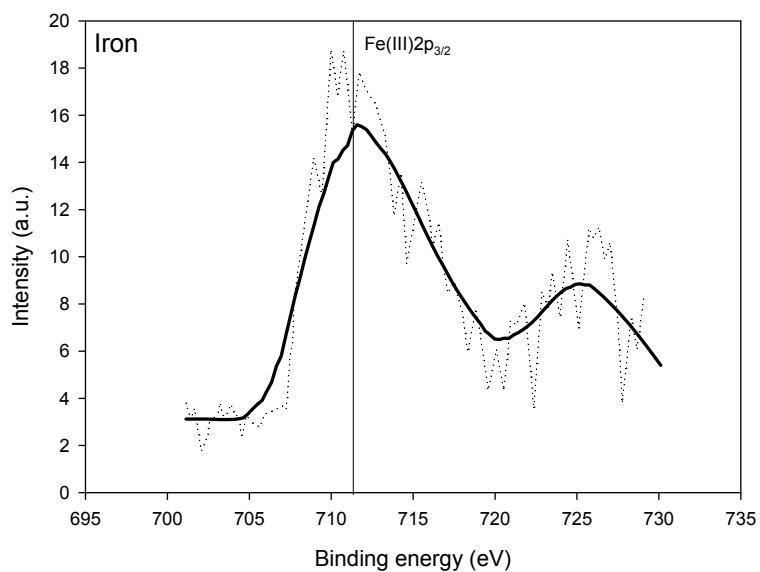


Fig. S3 XPS spectrum of the Fe 2p edge of the SiO₂-FeIII (acac) sample

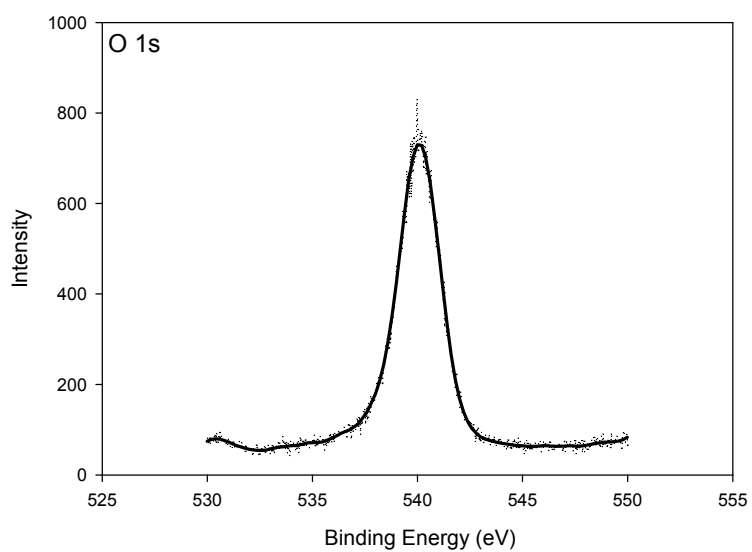


Fig. S4 XPS spectrum of the oxygen 1s edge of the SiO₂-FeIII (acac) sample

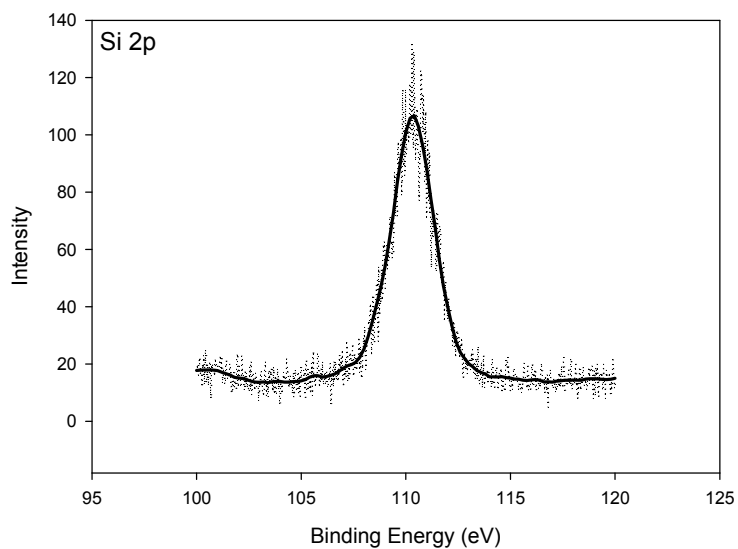


Fig. S5 XPS spectrum of the Si 2p edge of the SiO₂-FeIII (acac) sample

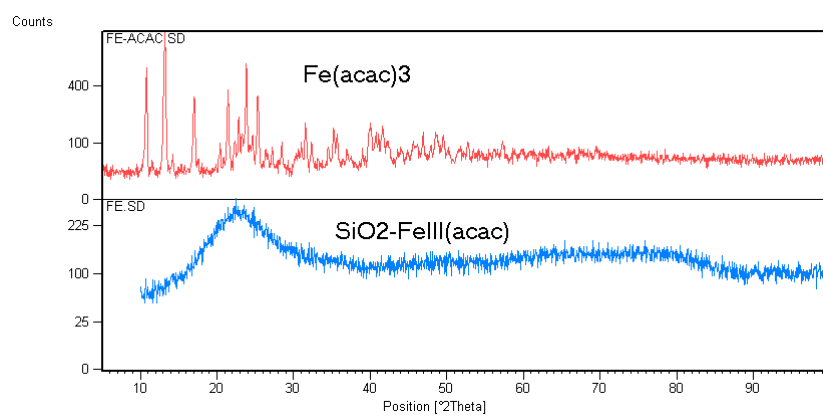


Fig. S6 XRD patterns of SiO₂-FeIII (acac) catalyst sample, compared with Fe(acac)₃.

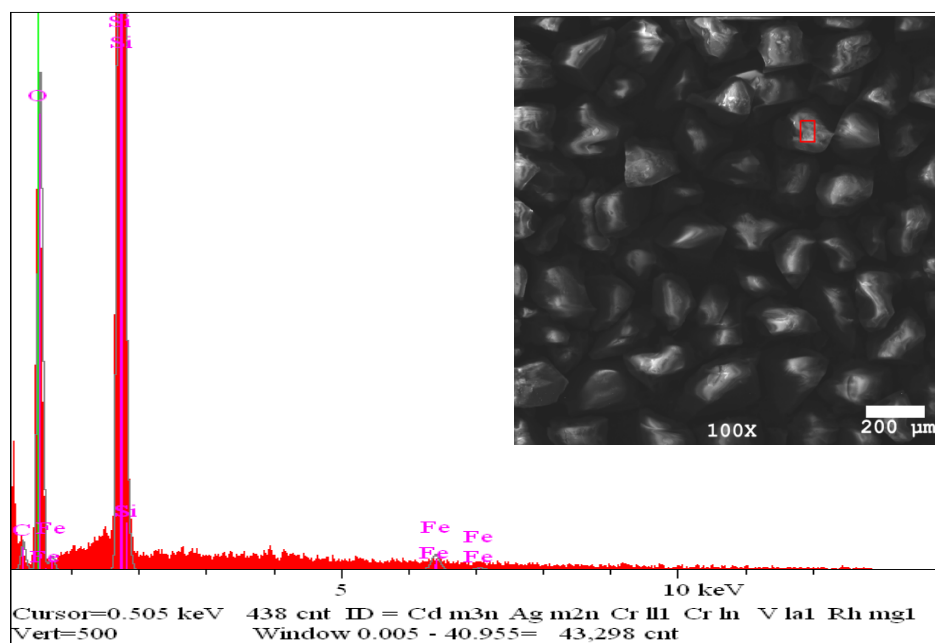


Fig. S7 EDX spectrum of SiO₂-FeIII(acac) catalyst sample.

References

- 1 A.Zarei, A. R. Hajipour, L. Khazdooz, *synthesis*, 2009, 941-944.