

Reduction of C=C Double Bonds by Hydrazine Using Active Carbons as Metal-Free Catalysts

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Supporting Information

ABSTRACT: A series of active carbons with or without chemical oxidation by nitric acid have been prepared, and their activity was evaluated as metal-free catalysts for the C=C double bond hydrogenation using hydrazine as reducing agent in the presence of oxygen. It was observed that an optimal treatment by nitric acid results in a sample with enhanced catalytic activity that is superior to that measured under the same conditions for the other carbon materials. Kinetic data suggest that the active sites in the process could be quinone/hydroquinone type redox centers introduced in the oxidative treatment of AC by nitric acid. Nitric acid-oxidized active carbon has a wide substrate scope and also exhibits a chemoselective C=C hydrogenation with respect to nitro group reduction compared to conventional Pd/C catalyst.

KEYWORDS: Heterogeneous catalysts, Carbocatalysis, Active carbon, Hydrazine, Olefin reduction



INTRODUCTION

Catalysis is currently dominated by the use of transition metals, in many cases precious noble metals.^{1–3} One current tendency in catalysis is to find alternatives to the use of critical metals used in the preparation of catalysts, replacing them by more abundant transition metals or even by metal-free catalysts.^{4,5} In this regard, the use of carbon catalysts is attracting considerable attention and one of the targets being to show the scope of this type of catalysts and particularly to apply them in reactions typically carried out by transition metals.^{6–9} In this context, the use of activated carbons as catalysts in the absence of added transition metals was known since long time ago, but mainly limited to the oxidation of pollutants, particularly NO_x in gas-phase.^{10,11} Due to its wide availability and sustainability, active carbons coming from biomass waste are appealing materials to develop metal-free catalysts.^{12,13} Within this line, one of the targets is to show that catalysts based on active carbons can promote reactions that up to now are almost exclusively catalyzed by noble metals.^{14,15}

One paradigmatic example metal catalyzed reactions is hydrogenation of C=C double bonds that is typically promoted by Pt, Pd, Rh, and other noble metals, being of interest to develop alternatives based on metal-free catalysts. Very recently, following the work of Su and co-workers with carbon nanotubes,^{16,17} it has been reported that graphitic carbon can act as metal free catalysts for hydrogenation of nitrobenzene using hydrazine as reducing agent.^{18,19} Continu-

ing with this line of research, herein we report that chemical oxidation of commercial active carbons increases their activity for hydrogenation of C=C double bonds by hydrazine. The leading idea based on the proposals of nature of active sites is that the modification of commercial carbon by chemical oxidation should improve the catalytic activity of this active carbon. As it has been proposed that carbonylic compounds, vicinal diketones at the periphery and defects of graphenic walls could be the active sites to activate hydrazine,²⁰ it can be speculated that oxidation of active carbons with nitric acid should be a favorable treatment to enhance the activity of these materials for hydrazine activation. The present manuscript provides experimental support to this hypothesis.

EXPERIMENTAL SECTION

Materials. Graphite (Aldrich, CAS 7782-42-5), diamond nanoparticles (ref: 636-444, ≥ 97%), activated carbon (AC, Noct SX Ultra, ref: 33663) and palladium supported on activated carbon (Sigma-Aldrich, ref: 205699) were provided by Sigma-Aldrich. Other reagents employed were analytical or HPLC grade were also provided by Sigma-Aldrich.

Catalyst Preparation. AC catalysts treated with nitric acid were obtained by submitting the commercial AC sample to nitric acid oxidation. One gram of AC (Noct SX Ultra) was dispersed in 25 mL

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