2. Experimental

2.1. Catalyst Preparation

Cu-HMS were synthesized following the procedures similar to those proposed by Tanev et al. via a neutral templating pathway using dodecylamine (DDA) as a surfactant.\(^1\) In a typical synthesis, 3.85 g of DDA was dissolved in 130 ml of water, then 32.5 ml of ethanol was then added to generate a 40:10 H\(_2\)O/EtOH solution of the surfactant. The surfactant solution was stirred for 15 min. At the same time, 0.35 g of copper chloride was added in the mixture of 20 ml ethanol and 23 ml tetraethyl orthosilicate (TEOS). Then the mixture was slowly added to the surfactant solution, stirred for about 2 h. The resultant solution was aged for 18 h at room temperature (25 °C) to obtain crystalline products. The solid precipitates were filtered out, dried at 120 °C over night, and calcined at 600 °C for 4 h. Finally, Cu-HMS was obtained for further experiments.

A Pd-containing solution was prepared by heat-dissolving palladium chloride (PdCl\(_2\)) in methanol solution at a temperature of 65 °C. Fully dried Cu-HMS supports were mixed with the solution and was then stirred for about 3 h to impregnate the Cu-HMS with Pd-containing solution. Thereafter, methanol was evaporated away from the mixture at a temperature of 65 °C under a reduced pressure. The residual mixture was heat-treated at 120 °C for one hour to get a solid catalyst. The total contents of the metal compound in terms of metallic palladium was 0.25 % by weight based on the weight of carrier.

2.2. Production and Analysis of Diethyl Carbonate (DEC)

Catalytic activity was measured by a computer-controlled continuous micro reactor system
(WFS-3015) with a quartz tubular reactor of 4 mm inner diameter. The reaction products collected by a cooling trap were taken out and sampled each hour, and analyzed by a gas chromatograph (GC) (4890D, Agilent) with a FID detector. The uncondensed gas products were introduced to the a gas chromatograph (GC-8A, Shimadzu) through an on-line six-way valve and analyzed by a TCD detector with a TDX-01 and Propak-Q packed column. The reaction conditions were steadily kept at a reaction temperature of 140 °C and a reaction pressure of 0.64 MPa.

2.3. Thermodynamic data

The Thermodynamic data of DEC was not found in the thermodynamics notebook. So it could be estimated by group contribution method. And the Gas standard thermodynamic data of other molecules was got from the thermodynamics notebook.
