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## Precise regulation of selectivity of supported nano-Pd catalysts using polysiloxane coatings with tunable surface wettability

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A facile method was developed for surface modification of supported nano-Pd with tailorable wettability. The obtained Pd/TiO<sub>2</sub>@POS catalytic materials could be used in controllable synthesis of styrene and ethylbenzene from hydrogenation of phenylacetylene, and the selective synthesis of imine and *N*-methylanilines in reductive amination reaction. The precise modification of the hydrophilicity/hydrophobicity of the catalyst surface is the crucial operation to realize this targeted transformation.

Heterogeneous catalysts are extensively used in industrial manufacture of bulk and fine chemicals due to their easy recoverability, and reusability.<sup>1, 2</sup> From the view of economical, energy-saving, and environmentally benign, development of highly selective heterogeneous catalytic processes is highly demanded.<sup>3, 4</sup> However, it is difficult to control the selectivity toward the desired products in heterogeneous catalysis when products can be further reacted to afford undesired byproducts or more than one reactive group is present in the substrates. For example, the selective hydrogenation of alkynes to generate alkenes is a key industrial process to manufacture polymer-grade alkenes and to synthesize new chemicals, pharmaceuticals, and materials.<sup>5</sup> Nevertheless, the highly selective synthesis of alkenes from alkynes remains a tremendous challenge because alkenes are prone to further hydrogenation to produce the alkanes.<sup>6, 7</sup> To improve the reaction selectivity over the heterogeneous catalysts, a number of strategies have been developed, such as adjusting the metal nanoparticle sizes and shapes,<sup>8-13</sup> constructing multimetallic nanoparticles with additional metals,14-19 generating strong metal-support interactions,<sup>20-22</sup> doping the supports<sup>23</sup> and



Recently, surface modification has become a powerful tool for enhancing selectivity in heterogeneous catalysis.<sup>25-34</sup> Owing to their flexibility and versatility, hydrophobic-hydrophilic surface modification represents an important set for the surface modification of heterogeneous catalysts.35-41 The selectivity of the product could be improved by influencing the adsorption/desorption of reaction intermediate on a hydrophobic-hydrophilic properties of a catalyst. Because hydrophobic surface could promote desorption of the hydrophilic molecules, modification of hydrophobic surface for enhancing the selectivity has been applied in a series of reactions, such as reduction of aromatic ketones to alcohols,<sup>35</sup> oxidation of aliphatic alcohols to acids,<sup>38</sup> oxidation of sulfide to sulfoxide.<sup>39</sup> Compared with modification of hydrophobic surface, the influence of hydrophilic surface on the product selectivity is rarely studied. Recently, Xiao and co-workers presented the selective hydrogenation of furfural to furan using a Pd@S-1-OH catalyst with appropriate hydrophilicity.<sup>41</sup> Moreover, it is still challenging to extend this strategy to other heterogeneous catalytic reactions.

Here, we report a facile method for the preparation of polysiloxane (POS)-modified  $Pd/TiO_2$  materials. The  $Pd/TiO_2@POS$  catalysts can be precisely tailored from hydrophilic to hydrophobic (Fig. 1). The obtained  $Pd/TiO_2@POS$  catalysts feature good selectivity in controllable synthesis of styrene and ethylbenzene, which is the first example of application of surface wettability in selective hydrogenation of phenylacetylene. Meanwhile, controllable synthesis of *N*-methyleneaniline and *N*-methylamines can be achieved by adjusting the hydrophilicity/hydrophobicity of the catalyst, too.

The hydrophilic/hydrophobic polysiloxane (POS)-modified catalysts were firstly prepared by deposition–precipitation method to afford Pd/TiO<sub>2</sub> followed by hydrolytic condensation of *n*-hexadecyltrimethoxysilane (HDTMS) and TEOS onto the

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pristine Pd/TiO<sub>2</sub> to generate the Pd/TiO<sub>2</sub>@POS catalysts (Fig. 1). In order to control the hydrophilicity/hydrophobicity of the catalyst samples, the volume of HDTMS was adjusted from 0.12 to 2.06 mL whilst keeping the other synthesis parameters constant.



Fig. 1 Schematic illustration for the controllable preparation of hydrophilic/hydrophobic  $Pd/TiO_2@POS$  catalysts.

In order to investigate the wettability nature of the modified nano-Pd samples, water contact angles ( $CA_{water}$ ) of the catalyst samples were measured. The influence of the amounts of HDTMS on wettability of the Pd/TiO<sub>2</sub>@POS coatings is shown in Table S1. The pristine Pd/TiO<sub>2</sub> sample showed a CA<sub>water</sub> of 22.9° in air. When the volume of HDTMS was low (0.12 mL), the water miscibility of Pd/TiO<sub>2</sub>@POS-1 catalyst was apparently emaciated to circa 44.5°. The CA<sub>water</sub> increased from 44.5° to 144.3° step by step with increasing the volume of HDTMS from 0.12 to 1.21 mL. Further increasing of HDTMS volume to 2.06 mL increased the CA<sub>water</sub> to 154.5°, which indicates that the modified catalyst became superhydrophobic. The above results clearly suggested that the POS coating could transform the surface character of the nanocomposite from hydrophilic to hydrophobic and even superhydrophobic.

The Pd/TiO<sub>2</sub> and Pd/TiO<sub>2</sub>@POS samples were characterized by scanning electron microscopy (SEM), transmission electron microscopy (TEM), XRD, XPS and N<sub>2</sub> adsorption-desorption studies to reveal their structures. Firstly, the morphologies of the materials were measured by SEM and TEM. The SEM images of the samples are shown in Fig. 2. As is shown in Fig. 2a, the Pd/TiO<sub>2</sub> sample had a spherical shape with a relatively smooth surface and a diameter of approximately 30-70 nm. After modification by POS, the original spherical morphology of Pd/TiO<sub>2</sub> was almost preserved, but the modified samples showed a rough surface and an enlarged thickness of the spherical shape. This phenomenon was observed clearly when changing the volume of HDTMS. When the volume increased from 0.12 to 2.06 mL, the resultant materials increased in particle size gradually. The results implied that the assembly of POS coatings took place controllably on the surface of Pd/TiO<sub>2</sub>.

The TEM images of samples are shown in Fig. S1. As illustrated in Fig. S1a, the mean particle diameter of Pd nanoparticles in pristine Pd/TiO<sub>2</sub> was  $2.1\pm0.5$  nm, and these nanoparticles were homogeneously dispersed on the TiO<sub>2</sub> surface. After coated with a POS layer, the Pd nanoparticles were not visible in the TEM image, as they were covered by the POS<sub>ie</sub>coating million addition, the TEM images also revealed that 1319 the POS precursors were completely coated with the POS coatings after subjected to ammonia-assisted sol-gel process.



Fig. 2 SEM images of samples. (a) Pd/TiO<sub>2</sub>, (b) Pd/TiO<sub>2</sub>@POS-1, (c) Pd/TiO<sub>2</sub>@POS-2, (d) Pd/TiO<sub>2</sub>@POS-3, (e) Pd/TiO<sub>2</sub>@POS-4, (f) Pd/TiO<sub>2</sub>@POS-5.

Moreover, the XRD patterns of the samples are shown in Fig. S2. The Pd/TiO<sub>2</sub> sample showed mixed phases of anatase and rutile TiO<sub>2</sub>. Neither a palladium oxide nor a metallic palladium diffraction pattern was detectable in Pd/TiO<sub>2</sub> catalyst, which might be ascribed to that the Pd species in these catalysts were amorphous or well dispersed on the surface of TiO<sub>2</sub>. Compared with Pd/TiO<sub>2</sub>, the Pd/TiO<sub>2</sub>@POS sample exhibited a new diffraction peak located at 20.7°. This peak is the typical diffraction peak of silica (PDF#76–0931), which strongly proves successful modification of POS coatings on the surface of Pd/TiO<sub>2</sub>.

Next, the XPS studies of the catalysts were performed to determine the chemical state of Pd species. As shown in Fig. S3, the Pd  $3d_{5/2}$  signal of Pd/TiO<sub>2</sub> appeared at 335.1 eV, which suggested that metallic Pd was mainly formed on the surface of Pd/TiO<sub>2</sub>. No peak of Pd  $3d_{5/2}$  was observable in the Pd/TiO<sub>2</sub>@POS samples, which might be attributed to the POS coating on the surface of Pd/TiO<sub>2</sub>. In the XPS survey spectra of the Pd/TiO<sub>2</sub>@POS catalysts, intensive Si 2p (103.0 eV) and Si 2s (153.7 eV) peaks appeared as well as Ti 2p peak disappeared simultaneously (Fig. S4). The results clearly demonstrated successful modification of the POS coating on the surface of the Pros coating on the surface of the pristine Pd/TiO<sub>2</sub>, which is consistent with the XRD results.

The N<sub>2</sub> adsorption–desorption tests showed that the BET surface areas of the Pd/TiO<sub>2</sub>, Pd/TiO<sub>2</sub>@POS-1, Pd/TiO<sub>2</sub>@POS-2, Pd/TiO<sub>2</sub>@POS-3, Pd/TiO<sub>2</sub>@POS-4, and Pd/TiO<sub>2</sub>@POS-5 were 55.325, 37.548, 24.116, 25.587, 27.182, and 11.932 m<sup>2</sup> g<sup>-1</sup>, respectively (Table S1). The BET surface areas of the modified materials present a slight decrease with respect to Pd/TiO<sub>2</sub>. In addition, the Pd/TiO<sub>2</sub>@POS samples give similar average pore radius(Table S1). By the use of ICP-AES, the Pd loadings of the

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 $Pd/TiO_2@POS$  catalysts were measured as 0.22, 0.07, 0.03, 0.04, and 0.07 wt%, respectively (Table S1).



Fig. 3 Catalytic hydrogenation of phenylacetylene over various Pd/TiO\_@POS catalysts. Reaction conditions: phenylacetylene (1.0 mmol), H<sub>2</sub> (1.0 atm), EtOH (4.0 mL), 50 °C, 2 h.

We have further checked whether the surface hydrophilization/hydrophobization of  $Pd/TiO_2@POS$  catalysts could really regulate the catalytic performance in catalytic reactions. The first model reaction is the catalytic hydrogenation of phenylacetylene as the tuning of the selectivity to styrene and ethylbenzene is quite difficult (Fig. 3 and Table S3). In view of the elementary steps in catalysis, desorption of styrene from the catalyst surface has a significant effect on the selectivity of reaction. Specifically, if hydrophobic styrene could rapidly desorb from hydrophilic catalyst surface, it would avoid the further hydrogenation of styrene to ethylbenzene and thus increase the selectivity of alkene. On the other hand, hydrogenation of phenylacetylene to ethylbenzene would be realized when hydrophobic styrene still adsorbs on hydrophobic catalyst surface. The reactions were performed by using the same dosage of nano-Pd catalyst and 1.0 mmol of phenylacetylene in EtOH at 50 °C under 1 atm of H<sub>2</sub> for 2 h. The reaction over Pd/TiO<sub>2</sub>@POS-1 was sluggish and only 20% conversion of phenylacetylene was obtained, although the good styrene selectivity of 91.7% was observed. Given that the CA<sub>water</sub> of Pd/TiO<sub>2</sub>@POS-1 is 44.5°, the low conversion should be attributed to difficult adsorption of phenylacetylene on a hydrophilic surface. When Pd/TiO<sub>2</sub>@POS-2 was used as catalyst, the conversion of phenylacetylene increased to 87% and selectivity to styrene remained high (91.7%). As expected, ethylbenzene product was generated with further increasing the  $CA_{water}$  of the catalyst. For example, the Pd/TiO<sub>2</sub>@POS-3, Pd/TiO<sub>2</sub>@POS-4, and Pd/TiO<sub>2</sub>@POS-5 catalysts led to 99% conversion of phenylacetylene with 99% ethylbenzene selectivity, probably because styrene could not rapidly desorb from hydrophobic catalyst surface and was further reduced to generate ethylbenzene. By applying Pd/TiO<sub>2</sub> as catalyst, >99% conversion of phenylacetylene with 67.9% selectivity to styrene were obtained. Clearly, these results were similar to those obtained by hydrophilic Pd/TiO<sub>2</sub>, i.e., Pd/TiO<sub>2</sub>@POS-1 and Pd/TiO<sub>2</sub>@POS-2. From the above results, we could draw a

conclusion that modification by hydrophilic/hydrophobic POS coatings on the nano-Pd surface could<sup>Q</sup>hided<sup>3</sup>fegulate<sup>8</sup>the selectivity of hydrogenation of phenylacetylene. The Pd/TiO<sub>2</sub>@POS catalyst with appropriate hydrophilicity exhibits good selectivity for the formation of styrene while the Pd/TiO<sub>2</sub>@POS catalyst with appropriate hydrophobicity displays good selectivity toward ethylbenzene. The Pd contents in the solution were tested by ICP-AES but it was out of the test limit (0.05 ppm). So the influence of leached palladium on the reaction can be neglected.



**Fig. 4** Reductive amination of aniline with paraformaldehyde over various  $Pd/TiO_2@POS$  catalysts. Reaction conditions: aniline **1** (1.0 mmol), paraformaldehyde **2** (1.0 mmol), H<sub>2</sub> (1.0 atm), toluene (4.0 mL), 80 °C, 4 h.

These surface-modified nano-Pd catalysts can also be applied to the reductive amination of aniline with paraformaldehyde for controllable synthesis of imine and N-methylanilines (Fig. 4 and Table S4). By applying Pd/TiO<sub>2</sub> as catalyst, 86% conversion of aniline with 60.7% N-methyleneaniline selectivity were obtained. With the hydrophilic Pd/TiO<sub>2</sub>@POS-1 as a catalyst, 84% conversion of aniline with 90.1% N-methyleneaniline selectivity and 9.9% N-methyl aniline selectivity were obtained. The selectivity of N-methylanilines increased to 21.1% if Pd/TiO<sub>2</sub>@POS-2 was used as catalyst. As expected, high selectivity toward N-methylanilines was achieved by increasing the CA<sub>water</sub> of catalyst, i.e., 94.0% selectivity was achieved in the presence of Pd/TiO<sub>2</sub>@POS-4. The enhanced selectivity to Nmethylanilines can be explained by the high hydrophobicity of the POS coatings, in which hydrophobic imine was easily accommodated into the shell of the catalyst followed by hydrogenation. The results clearly demonstrated that the selectivity of imine and N-methyl aniline could be regulated by changing the hydrophilic/hydrophobic of catalyst surface. The reductive amination of aniline with paraformaldehyde involved three main steps, i.e., the addition of aniline to formaldehyde, imine formation via dehydration and imine hydrogenation to generate N-methylaniline. The hydrophilic surface of Pd/TiO<sub>2</sub>@POS-1 benefits desorption of hydrophobic intermediate to selective afford imine product, while the hydrophobic surface of Pd/TiO<sub>2</sub>@POS-4 favors accumulation of hydrophobic N-methyleneaniline intermediate followed by hydrogenation to form N-methylanilines.

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As the selectivity of these two reactions is time-dependent, typical catalysts with hydrophilic and hydrophobic properties, i.e.,  $Pd/TiO_2@POS-2$  and  $Pd/TiO_2@POS-4$ , were compared with varied reaction times (Fig. S6 and 7). Clearly, higher selectivity to styrene and N-methyleneaniline were achieved over hydrophilic  $Pd/TiO_2@POS-2$  while ethylbenzene and N-methyl aniline were obtained over hydrophobic  $Pd/TiO_2@POS-4$ . So the selectivities were mainly tuned by the surface properties of catalysts but not reaction time.

In summary, we have developed a convenient method for surface modification of nano-Pd using POS coatings with controllable wettability. The successful fabrication of POS coatings is confirmed by detailed characterizations. The resultant Pd/TiO<sub>2</sub>@POS catalysts exhibit good selectivity in the hydrogenation of phenylacetylene to styrene and ethylbenzene. Furthermore, controllable formation of imine and *N*-methylanilines in reductive amination reaction can also be realized by regulating the hydrophilicity/hydrophobicity of the catalysts, too. This work may promote the development of controllable preparation of heterogeneous catalysts via precise modification of the catalyst surface.

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### **Conflicts of interest**

There are no conflicts to declare.

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