

Direct Transformation of Furfural to 1,2-Pentanediol Using a Hydrotalcite-Supported Platinum Nanoparticle Catalyst

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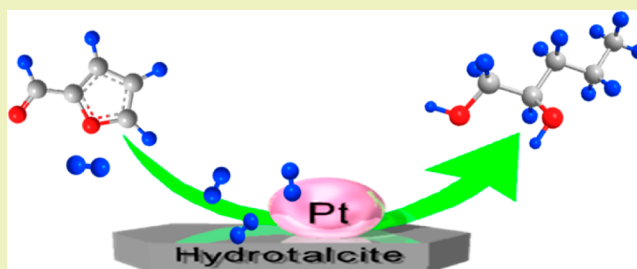
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S Supporting Information

ABSTRACT: Hydrotalcite-supported Pt nanoparticles catalyze the direct transformation of furfural to 1,2-pentanediol in a high yield of 73% under additive-free conditions. The Pt nanoparticle catalyst is easily recoverable and reusable while maintaining high activity and selectivity.

KEYWORDS: Hydrogenolysis, Furfural, 1,2-Pentanediol, Hydrotalcite-supported Pt nanoparticle catalyst



INTRODUCTION

Recent requirements to decrease the emission of carbon dioxide have led to the utilization of renewable resources such as plant biomass as carbon neutral feedstocks for the production of commodity chemicals.^{1–3} Furfural is a lignocellulosic material manufactured at the rate of 5 million tons per year by the dehydration of hemicelluloses, such as xylose and arabinose, and has attracted attention as a potential C₅ resource for the synthesis of chemicals such as furfuryl alcohol and 1,5-pentanediol.^{4–7}

1,2-Pentanediol (1,2-PeD) is a valuable chemical used as a monomer in the production of polyesters⁸ and an intermediate in the synthesis of fungicides⁹ and is also a component of disinfectants, printing inks,¹⁰ and cosmetics.¹¹ Conventional processes for the production of 1,2-PeD rely on petroleum resources and require multistep reactions, an example being the oxidation of 1-pentene to 1,2-epoxypentene and the subsequent hydrolysis to afford 1,2-PeD.⁸ From the viewpoint of green sustainable chemistry, the development of a new synthesis of 1,2-PeD from biomass-derived chemicals is highly desirable. Despite the extreme usefulness of furfural as a C₅ resource, the transformation of furfural to 1,2-PeD has not been widely studied, and to date, a catalyst system using PtO₂/γ-Al₂O₃ has been reported for the selective synthesis of 1,2-PeD.¹² Other catalyst systems of PtO₂ with FeCl₂¹³ and Pt/Co₂AlO₄¹⁴ are also known to catalyze the hydrogenolysis of furfural, but these methods suffer from low yields of 1,2-PeD (below 30%). The development of efficient catalytic systems for the direct transformation of furfural to 1,2-PeD would thus be very beneficial.¹⁵ Herein, we report that a catalyst consisting of hydrotalcite-supported Pt nanoparticles (Pt/HT) is capable of

efficiently promoting the direct hydrogenolysis of furfural to 1,2-PeD without the presence of any additives. Notably, 1,2-PeD was obtained in 73% yield from furfural, a yield much greater than those obtained from previously reported catalyst systems.^{13,14} Furthermore, this Pt/HT catalyst is reusable while maintaining its activity and selectivity.

EXPERIMENTAL SECTION

Pt/HT was prepared by the impregnation method using an aqueous solution of H₂PtCl₆ and hydrotalcite (Tomita Pharmaceutical, AD-500NS), followed by reduction with KBH₄ (Pt: 1.9 wt %).^{16–18} The powder X-ray diffraction (XRD) pattern of the resulting Pt/HT clearly showed the preservation of the layered structure of HT following impregnation by the Pt species and exhibited no diffraction peaks due to Pt metal, indicating the high dispersion of Pt nanoparticles (Pt NPs) on the HT support (Figure S1, Supporting Information). Transmission electron microscopy (TEM) demonstrated the formation of Pt NPs with an average diameter of 3.7 nm on the surface of the hydrotalcite (Figure S2, Supporting Information), while in situ Pt L_{III}-edge XAS analysis of Pt/HT supported the formation of Pt(0) NPs (Figure S3, Supporting Information). Pt/HT was stable in air; the catalytic performance of Pt/HT stored in a vial for two months showed the same catalytic performance as that of a fresh sample.

RESULTS AND DISCUSSION

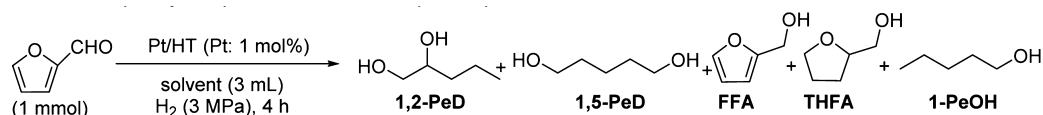
The hydrogenolysis of furfural was conducted under 3 MPa of H₂ using the Pt/HT catalyst (Table 1). Notably, 1,2-PeD was obtained in 73% yield together with THFA (14% yield), 1,5-

Received: May 17, 2014

Revised: August 29, 2014

Published: September 19, 2014

Table 1. Hydrogenolysis of Furfural Catalyzed by Pt/HT



entry	solvent	temp. (K)	conv. (%) ^a	yield (%) ^a					
				1,2-PeD	1,5-PeD	FFA	THFA	1-PeOH	others
1	2-PrOH	333	>99	16	28	0	50	2	4
2	2-PrOH	373	>99	43	13	0	42	1	1
3	2-PrOH	423	>99	73	8	trace	14	3	2
4	2-PrOH	453	>99	58	8	0	11	3	20
5	<i>n</i> -BuOH	423	>99	69	8	trace	15	3	5
6	1,4-dioxane	423	>99	61	8	0	24	2	5
7	toluene	423	>99	60	7	0	21	2	10
8 ^b	EtOH	423	>99	71	9	trace	12	3	5
9	water	423	>99	18	6	48	14	1	13
10	MeOH	423	>99	1	2	93	1	0	3
11	DMF	423	>99	0	0	91	1	0	0

^aConversion and yields were determined by GC-MS using an internal standard technique. Yields of others were obtained by the differences between converted furfural and the sum of the yields of 1,2-PeD, 1,5-PeD, FFA, THFA, and 1-PeOH. ^bReaction time was 8 h.

Table 2. Hydrogenolysis of Furfural in 2-PrOH Using Various Catalysts^a

entry	catalyst	conv. (%) ^a	Pt NP size (nm)	yield (%) ^b					
				1,2-PeD	1,5-PeD	FFA	THFA	1-PeOH	others
1	Pt/HT	>99	3.7	73	8	trace	14	3	2
2 ^c	Pt/HT	>99	3.7	73	8	0	16	3	0
3 ^d	Pt/HT	>99	3.8	72	8	0	16	3	1
4	Pt/MgO	>99	3.4	68	7	trace	23	2	0
5	Pt/CeO ₂	>99	4.5	41	5	37	15	2	0
6	Pt/ γ -Al ₂ O ₃	>99	5.4	28	4	36	16	3	13
7	Pt/TiO ₂	>99	4.1	trace	2	51	2	2	33 ^e
8	Pt/HAP	76	5.5	trace	1	55	4	1	15 ^e
9	Pt/SiO ₂	34	5.4	0	0	24	1	0	9
10	Pd/HT	>99	—	0	0	0	81	0	19
11	Ru/HT	>99	—	4	1	45	38	0	12
12	Cu/HT	>99	—	0	0	89	0	0	11
13	Ir/HT	>99	—	0	0	81	0	0	19
14	Ag/HT	85	—	0	0	64	0	0	21
15	Rh/HT	60	—	0	0	27	5	0	28
16	Pt black + HT	>99	—	trace	1	89	3	1	6

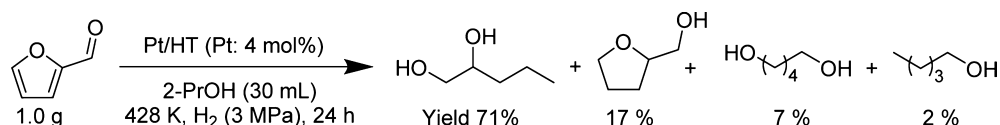
^aFurfural (1 mmol), catalyst (metal: 1 mol %), 2-PrOH (3 mL), 423 K, and H₂ (3 MPa). ^bConversion and yields were determined by GC-MS using an internal standard technique. Yields of others were obtained by the differences from converted furfural to the sum of the yields of 1,2-PeD, 1,5-PeD, FFA, THFA, and 1-PeOH. ^cFirst reuse. ^dSecond reuse. ^e2,2'-Oxybis(methylene)difuran was included in others.

pentanediol (1,5-PeD, 8% yield), and 1-pentanol (1-PeOH, 3% yield) following the complete conversion of furfural at 423 K in 2-propanol (2-PrOH) solvent (Table 1, entry 3). This yield of 1,2-PeD is comparable to PtO₂/ γ -Al₂O₃¹² and is much greater than those obtained from previously reported catalyst systems consisting of PtO₂ + FeCl₂ (27%)¹³ or Pt/Co₂AlO₄ (16%).¹⁴ No formation of acetone or the condensation product of furfural with acetone was observed, revealing that 2-PrOH did not act as a hydrogen source under H₂.¹⁵ Interestingly, the reaction temperature strongly affects the 1,2-PeD yield. The yield of 1,2-PeD increased from 16% to 73% on increasing the temperature from 333 to 423 K (Table 1, entries 1–3), while the yield of THFA and 1,5-PeD decreased at higher temperature (Table 1, entry 4). With regard to solvents, 2-PrOH gave the best results among various solvents used (Table 1, entries 3 vs 5–11). EtOH afforded a 71% yield of 1,2-PeD but required a longer reaction time of 8 h (Table 1, entry 8).

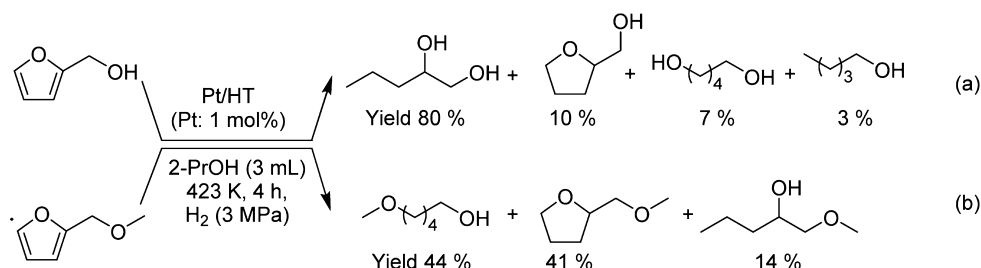
n-BuOH, 1,4-dioxane, and toluene were all found to be suitable solvents (Table 1, entries 5–7), while water resulted in nonselective hydrogenolysis (Table 1, entry 9). MeOH and DMF did not promote 1,2-PeD synthesis but gave 93% and 91% of furfuryl alcohol (FFA), respectively (Table 1, entries 10 and 11).

The various supported Pt NPs catalysts were subsequently tested using 2-PrOH as the solvent under 3 MPa of H₂ (Table 2). Under these conditions, Pt/MgO also exhibited high activity, giving 68% yield of 1,2-PeD (Table 2, entry 4), while Pt/CeO₂ and Pt/Al₂O₃ afforded 1,2-PeD in lower yields of 41% and 28%, respectively (Table 2, entries 5 and 6).²⁰ Pt/TiO₂, Pt/HAP, and Pt/SiO₂ did not give 1,2-PeD, but rather generated FFA as a major product (Table 2, entries 7–9). The significant effect of the catalyst metals on the 1,2-PeD yield was observed in the reaction of furfural (Table 2, entries 1 vs 10–15). In contrast to Pt/HT, Pd/HT and Ru/HT did not provide 1,2-

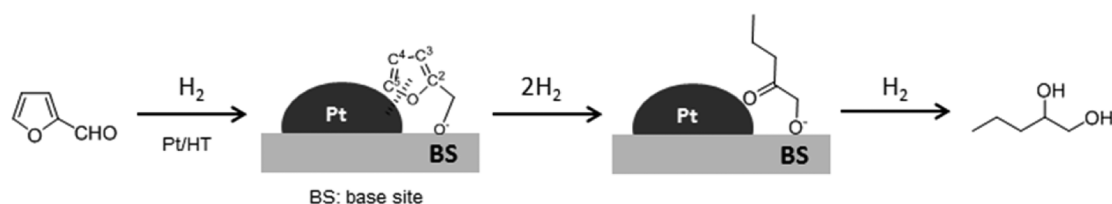
Scheme 1. Gram-Scale Reaction of Furfural Using Pt/HT Catalyst



Scheme 2. Hydrogenolysis of Furfuryl Alcohol (a) and Furfuryl Methyl Ether (b) using Pt/HT Catalyst



Scheme 3. Proposed Reaction Path for Hydrogenolysis of Furfural to 1,2-Pentanediol



PeD at all, but instead produced THFA in 81% and 38% yields, respectively (Table 2, entries 10 and 11).²¹ Cu/HT, Ir/HT, and Ag/HT afforded FFA as the major product by the reduction of only the formyl group (Table 2, entries 12–14). Notably, a physical mixture of Pt black and HT gave a high yield of FFA together with a trace amount of 1,2-PeD (Table 2, entry 16). These results clearly show that immobilization of Pt nanoparticles on the basic HT support is essential for the selective formation of 1,2-PeD.

Following the hydrogenolysis of furfural, the Pt/HT catalyst was easily recovered from the reaction mixture by centrifugation and was reusable without appreciable loss of the activity or selectivity for 1,2-PeD (Table 2, entries 2 and 3).²² The Pt content in the used catalyst was the same as in the fresh one evidenced by ICP-AES analysis (Pt: 1.9 wt %). TEM observations and XRD analysis of the used catalyst showed that both the size of Pt NP and the layered structure of the HT support were maintained following the hydrogenolysis reaction (Figures S1 and S2, Supporting Information), demonstrating the high durability of Pt/HT. Furthermore, the Pt/HT catalyst was shown to be applicable to a gram-scale reaction; 1.0 g of furfural was completely converted to give 1,2-PeD in a 65% isolated yield (71% GC-MS yield) (Scheme 1).

To elucidate the reaction path of furfural to 1,2-PeD, the progress over time of the hydrogenolysis reaction was investigated (Figure S4, Supporting Information). The yield of FFA was observed to increase with increases in the reaction time. After complete consumption of furfural, 1,2-PeD was formed as the amount of FFA decreased. During hydrogenolysis, trace amounts of 1-hydroxy-2-pentanone were found by GC-MS analysis, but the formation of dihydrofurfuryl alcohols was not detected. When FFA was employed as the substrate under reaction conditions similar to those of entry 1 in Table 2, 1,2-PeD was obtained in a significantly high yield of 80% (Scheme 2a).²³ The reaction of THFA in place of furfural

did not occur at all, although Tomishige and other groups reported that 1,5-PeD could be obtained from THFA.^{24–28} As a control, the hydroxy group of FFA was protected by a methyl group to afford furfuryl methyl ether. The hydrogenolysis of furfuryl methyl ether resulted in the formation of 5-methoxypentanol and tetrahydrofurfuryl methyl ether in 44% and 41% yields, respectively; 1-methoxy-2-pentanol was obtained only in a 14% yield (Scheme 2b). These results indicate that 1,2-PeD appears to be produced via the formation of the intermediates FFA and 1-hydroxy-2-pentanone.^{12,29} The furan ring and hydroxymethyl group of FFA are the key functions leading to the selective formation of 1,2-PeD.

On the basis of the above results, a reaction path for the synthesis of 1,2-PeD is proposed in Scheme 3. Initially, the Pt/HT catalyst preferentially reduces the formyl group of furfural to give FFA. Subsequent C⁵–O¹ bond scission of the furan ring of FFA followed by hydrogenation affords 1-hydroxy-2-pentanone, after which reduction of the keto group gives 1,2-PeD.²¹ The excellent catalytic performance of Pt/HT described above may be due to the cooperative behavior of the Pt NPs and HT, in which the surface basic sites of HT strongly interact with the hydroxymethyl moiety of the FFA intermediate, giving an alcoholate species,^{30–32} and the furan ring adsorbs on the surface of Pt NPs.

Polar hydrogen species are generated at the interface between Pt NPs and the basic sites of HT,³³ and the attack of these polar hydrogen species on the adsorbed furan ring leads to selective C⁵–O¹ bond scission,¹⁸ possibly assisted by the upright adsorption configuration on Pt NPs.^{34–36} This is followed by hydrogenation to give 1,2-PeD. The strong interaction between Pt NPs and HT is also responsible for the high durability of Pt/HT during the recycling experiments.

CONCLUSION

We have succeeded in the direct synthesis of 1,2-PeD from furfural as a biomass-derived platform chemical using a Pt/HT catalyst under additive-free conditions. The cooperative catalysis of Pt NPs and basic HT drastically increases the selectivity for 1,2-PeD. Furthermore, the Pt/HT catalyst was easily recoverable and reusable without appreciable loss of either activity or selectivity.

ASSOCIATED CONTENT

Supporting Information

Experimental details including catalyst preparation, characterization, and reaction procedures. This material is available free of charge via the Internet at <http://pubs.acs.org>.

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Notes

The authors declare no competing financial interest.

ACKNOWLEDGMENTS

This study was supported by grants from NEDO and JSPS KAKENHI (23360357 and 24246129). The authors thank Dr. Tetsuo Honma (JASRI) for Pt L_{III} -edge XAS measurements. The TEM observations were performed at a facility in the Research Center for Ultra-High Voltage Electron Microscopy, Osaka University. T. Mizugaki also wishes to acknowledge the financial support of the Shorai-Foundation for Science and Technology.

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