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Regioselective hydrogenation of 1-naphthol over supported Pt and Pd catalysts for producing high-temperature jet fuel stabilizer

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Abstract

Supported Pt and Pd catalysts were examined for selective hydrogenation of 1-naphthol to form tetrahydronaphthol (THNol). Five kinds of supports were used: HY zeolite with SiO_2/Al_2O_3 of 40, HM zeolite with SiO_2/Al_2O_3 of 38, γ-Al_2O_3, SiO_2 and TiO_2. Among these five supports, only TiO_2-supported catalysts displayed selectivity for 1-naphthol hydrogenation towards 1,2,3,4-THNol. Under the same reaction conditions, Pd/TiO_2 catalysts show much higher catalytic activity and higher selectivity to 1,2,3,4-THNol than Pt/TiO_2 catalysts, and more tetralin was observed in product distribution for Pd catalysts. On the other hand, Pt/TiO_2 showed higher selectivity for 5,6,7,8-THNol. By using Pt and Pd bimetallic catalysts, much higher selectivity to 1,2,3,4-THNol has been achieved. Product distribution depends strongly on Pt/Pd ratio. The selectivity of 1,2,3,4-THNol decreases with the increase in Pt/Pd weight ratio. The bimetallic catalyst with Pt/Pd weight ratio of 0.3 shows the highest 1,2,3,4-THNol selectivity up to 22.6 mol% at 150°C, 500 psi and 60 min. Tetralone has been found to be an intermediate for hydrogenation of 1-naphthol to 1,2,3,4-THNol. Further hydrogenation of 1,2,3,4-THNol towards tetralin is much faster than that of 5,6,7,8-THNol. © 2001 Elsevier Science B.V. All rights reserved.

Keywords: Regioselective hydrogenation; Supported Pt and Pd catalysts; Tetrahydronaphthol; 1-Naphthol

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1. Introduction

The present work deals with the catalytic production of specific hydroaromatic compounds as high-temperature stabilizers for jet fuels. Until recently, the major concern of jet fuel stability only involved thermal oxidation stability and storage stability, because the current operating fuel temperature of all the commercial and military jet fuels is below 300°C, where the fuel degradation is controlled by autoxidation reactions [1,2]. With the development of high-Mach aircraft, fuel thermal stability has become more crucial [3–6]. Fuel in such aircraft is expected to experience temperature in the range of 400–500°C [3–5]. One of the critical problems in developing thermally stable jet fuels for high-Mach aircraft is the thermal decomposition and formation of solids from hydrocarbon fuels in the pyrolytic regime [5–9]. Previous studies at Pennsylvania State University have demonstrated that during the pyrolytic degradation of coal and petroleum-derived jet fuels, hydrogen transfer form Hydrogen-donors, such as those present in coal-derived JP-8C jet fuel, could play an important role in suppressing thermal solid formation [6,10]. In previous papers [11–14], it has been reported that the formation of carbonaceous materials in jet fuel can be retarded by hydrogen donors such as tetrahydronaphthalene (THN), tetrahydroquinoline (THQ), and tetrahydronaphthol (THNol) and aromatic alcohol-type molecules such as benzyl

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alcohol and benzenedimethanol. More recent work at Pennsylvania State University has shown that THNol (especially the 1,2,3,4-isomer), THN as well as benzyl alcohol are effective hydrogen donors for capping aliphatic radicals formed at temperature of >400°C while transforming into relatively stable products [15].

The present work was motivated by the need to produce effective hydrogen donors as high-temperature fuel stabilizers by selective hydrogenation of aromatic compounds that are abundant in coal-derived liquids [16]. Hydrogenation of aromatics is exothermic and is therefore thermodynamically favored at a lower temperature [17,18]. However, in conventional hydrotreating processes, a reaction temperature above 325°C is typical, which consequently results in relatively higher content of aromatics at equilibrium composition. Conventional supported Ni-Mo and Co-Mo catalysts become active only at relatively high temperatures. Consequently, hydrogenation of aromatics at higher temperatures requires higher hydrogen pressure to offset the limitation of thermodynamic equilibrium conversion.

This study aims at examining the potential of supported Pt, Pd and their bimetallic catalysts for low-temperature hydrogenation of 1-naphthol to produce tetrahydronaphthol, particularly 1,2,3,4-tetrahydro-1-naphthol. The results revealed that by using proper support and bimetallic species, we can selectively promote the hydrogenation towards 1,2,3,4-tetrahydro-1-naphthol or 5,6,7,8-tetrahydro-1-naphthol.

2. Experimental

2.1. Catalyst precursor and reactant

Catalysts with one metal precursor loaded on different supports were prepared from HY zeolite with SiO_2/Al_2O_3 of 40, HM zeolite with SiO_2/Al_2O_3 of 37.5, γ-Al_2O_3, SiO_2 and TiO_2 (see Table 1, used as received), by incipient wetness impregnation of an aqueous solution of either tetraamineplatinum chloride ($[Pt(NH_3)_4]Cl_2$, Aldrich, 99.995% Pt, metal base) or tetraaminepalladium chloride ($[Pd(NH_3)_4]Cl_2$, Aldrich, 99.995% Pd, metal base). Bimetallic catalysts were prepared by co-impregnation method by using both $[Pt(NH_3)_4]Cl_2$ and $[Pd(NH_3)_4]Cl_2$. Following drying in vacuo at 100°C for 3 h, the catalysts were calcined in air at 450°C for 3 h. For all the catalysts used, the metal loading was kept at 1 wt.%.

2.2. Catalyst characterization

Characterization by temperature-programmed-reduction (TPR) was carried out for all the catalysts on a Micromeritics AutoChem 2910 (USA) analyzer. In a typical TPR experiment, about 0.4 g of catalyst was loaded in a U-shaped quartz adsorption cell. Before TPR run, the sample was heated in Ar for 30 min at 350°C in order to remove moisture and impurities and then cooled to room temperature in Ar flow (10 ml/min). TPR profile of catalyst sample was recorded by following the temperature program with a heating rate of 10°C/min between 25 and 500°C in 5.12% H_2–Ar gas mixture.

Metal dispersion on catalyst surface was determined by using H_2 pulse chemisorption method using the same apparatus. In a typical experiment, 0.2 g of catalyst sample was loaded in a U-shape quartz adsorption cell and the sample was prereduced by following the temperature program with a heating rate of 10°C/min between 25 and 350°C in H_2 with flow rate of 20 ml/min. The reduced sample was held at 350°C in Ar flow (20 ml/min) for 60 min and then cooled down to ambient temperature in Ar flow. After 30 min

Table 1
Types of the supports and their nominal properties

Support ID	Material type	Surface area (m^2/g)	SiO_2/Al_2O_3 mole ratio	Na_2O (wt.%)	Supplier and code
HY	Y-zeolite	750	40	0.03	PQ-CBV-740
HM	Mordenite	512	37.5	0.07	PQ CBV-30A
TiO_2	Titania	49			Degussa. P-25
Al_2O_3	γ-alumina	199			KF S-1
SiO_2	Silica-gel	499			Aldrich-28,851-9

holding in Ar flow at ambient temperature, H_2 pulse chemisorption profile was recorded using 10% H_2–Ar gas mixture until the discretely injected gas volumes emerges unchanged.

2.3. Catalyst evaluation

Catalytic tests were done in a 25 ml stainless-steel microautoclave reactor. The reactor system is T-shaped with most internal volume in the horizontal tube that contains the catalyst and reactants. The reactor was charged with 0.05 g catalyst, 0.5 g 1-naphthol (TCI, 99.99%), and 2.0 g of *n*-tridecane solvent. The charged reactor was flushed with H_2 to desired pressure to start test. The reactor was then mounted on a holder and immersed in a fluidized sand bath heater. Mixing was accomplished by vertical agitation (254 strokes/min). At the end of the test, the reaction was quenched by immersing the reactor into cold water. After cooling, the contents of the reactor were then washed by acetone onto a filter, and the solid was dried. Solution products were identified by GC-MS (HP5890 GC-HP 5971 MSD) and quantitatively analyzed by GC-FID. For both GC instruments, the column was a 30 m × 0.25 mm i.d. DB-17 fused silica capillary column and the oven temperature program was 80–290°C at 6°C/min. GC-MS analysis indicates that cracking of *n*-tridecane, if any, was negligible under the conditions used. More details of analytical procedures are available elsewhere [19].

3. Result and discussion

3.1. Effect of different supports

Table 2 shows the results of 1-naphthol hydrogenation over monometallic Pt and Pd catalysts prepared using different kinds of support. All reactions were done under 500 psi at 150°C, reaction time was 90 min for Pt catalysts and 30 min for Pd catalysts. It is apparent that even over shorter period of reaction time, Pd catalysts on all the supports show higher conversion than Pt catalyst on the same kind of support. Both kinds of zeolite supported catalysts display no selectivity for 1,2,3,4-THNol. Under the conditions employed, TiO_2 supported catalysts appeared to be the only one showing selectivity to 1,2,3,4-THNol and much higher selectivity to total THNL (1,2,3,4-THNol + 5,6,7,8-THNol).

In an attempt to compare the present results with previous literature data on effects of support or supported catalyst, we have conducted a literature search using SCI-Expanded database (1986–1999) of Institute of Scientific Information, but this search yielded no publications on hydrogenation of 1-naphthol [20]. Therefore, we were not able to compare our results with literature. On the other hand, it is known in the literature that TiO_2-supported catalysts may have metal-support interaction such that it favors the hydrogenation of oxygen-containing compounds. For example, Vannice has demonstrated that metal-support interactions (MSI) in Pt/TiO_2 can markedly shift selectivity towards the formation of, and retention of

Table 2
Effects of different supports[a]

Catalysts	Naphthol conversion (wt.%)	Product yield distribution (mol%)			
		1,2,3,4-THNol	5,6,7,8-THNol	Tetralone	THN + DeHN
Pt/HY	33	0	7.6	14.3	7.2
Pt/HM	58.4	0	23.9	16.3	15.4
Pt/Al_2O_3	36.9	0	24.2	10.6	2
Pt/TiO_2	84	2.8	55.5	12.2	6.6
Pd/HY	59.8	0	15.9	15.4	28.3
Pd/HM	100	0	23.2	13.1	57
Pd/Al_2O_3	100	1.6	28.2	13.7	55.7
Pd/TiO_2	97.8	11.2	34.2	20.5	29.4
Pd/SiO_2	100	0	35.2	0	64.8

[a] All reactions were done at 150°C, 500 psi; Pt catalysts: 90 min, Pd catalysts: 30 min.

Table 3
Hydrogenation of 1-naphthol over Pt and Pd monometallic catalysts

Catalysts	Temperature (°C)	Pressure (psi)	Time (min)	Naphthol conversion (%)	Product yield distribution (mol%)				
					DeHN	THN	Tetralone	1,2,3,4-THNol	5,6,7,8-THNol
Pt/TiO_2 (1 wt.%)	200	300	90	84.2	4.3	13.4	23.7	1.6	52.9
	200	500	90	100.0	7.6	25.6	1.9	1.4	47.5
	200	500	60	55.4	3.7	9.4	24.6	2.0	58.6
	150	500	60	36.7	5.6	1.0	25.2	2.4	65.7
Pd/TiO_2 (1 wt.%)	200	500	30	100.0	4.3	62.6	1.0	4.1	28.1
	200	300	30	100.0	2.2	59.4	4.0	7.2	31.5
	200	300	60	100.0	1.8	59.3	3.6	8.1	33.2
	150	500	60	100.0	2.1	52.6	0.4	8.0	42.0

high selectivity to, the intermediate unsaturated alcohol [21]. It is possible that the metal-support interaction between TiO_2 and metal atoms contributed partly to the regioselective hydrogenation of 1-naphthol. However, at the present stage, we do not have any direct evidence for the contribution of the metal-support interaction.

3.2. Hydrogenation of 1-naphthol over TiO_2 supported Pt and Pd monometallic catalysts

Results of 1-naphthol hydrogenation for TiO_2-supported Pt and Pd monometallic catalysts are shown in Table 3. Product distribution depends strongly on the type of metal used as well as the reaction conditions. It is apparent that Pd catalyst exhibits much higher activity for 1-naphthol conversion than Pt catalyst. On the other hand, Pd catalyst gave more hydrogenolysis product such as tetralin. It should be noted that Pd catalyst has higher selectivity to 1,2,3,4-THNol than Pt catalyst, whereas Pt catalyst presents higher selectivity for 5,6,7,8-THNol. By controlling reaction condition (200°C, 300 psi or 150°C, 500 psi), selectivity of 1,2,3,4-THNol for Pd catalyst can reach to 8 mol%.

The observed product distribution indicates the competitive adsorption during simultaneous ring-hydrogenation and C–O bond cleavage reaction. Process selectivity is interpreted in terms of reactant/catalyst interactions where naphthol ring adsorption is viewed as occurring via the aromatic pi-electron system and/or near the hydroxyl group where the latter interaction promotes either hydrogenation or hydrogenolysis, the latter represents a direct loss of selectivity with respect to THNol formation.

3.3. Hydrogenation of 1-naphthol over TiO_2 supported Pt–Pd bimetallic catalysts

Results for 1-naphthol hydrogenation at 150°C over Pt and Pd bimetallic catalysts are shown in Table 4. It is clear that bimetallic catalysts display much higher selectivity for 1,2,3,4-THNol. Product distribution strongly depends on Pt/Pd ratio. With the increase of Pt/Pd mole ratio, the selectivity for 1,2,3,4-THNol decreases. Relative to the monometallic Pd catalyst, the bimetallic catalyst with 0.3 Pt/Pd ratio (total metal loading kept at 1 wt.%) shows the highest 1,2,3,4-THNol selectivity, up to 22.6% at 150°C, 500 psi and 60 min, even though the 1-naphthol conversion decreased by roughly 10%.

TPR characterization results are shown in Fig. 1. For single metal catalysts, Pt catalyst displays much higher reduction temperature (376.3°C) than that of the Pd catalyst (283.1°C), this is consistent with the reaction result that Pd catalyst shows higher catalytic activity in terms of 1-naphthol conversion since all reaction were done at a low temperature, 150°C. When the two metals are loaded into bimetallic catalysts, the reduction temperatures fall in between the corresponding monometallic ones. There are two possible effects involved in bimetallic catalyst system prepared

Table 4
Hydrogenation of 1-naphthol over Pt–Pd bimetallic catalysts[a]

Catalysts	Pressure (psi)	Time (min)	Naphthol conversion (%)	Product yield distribution (mol%)				
				DeHN	THN	Tetralone	1,2,3,4-THNol	5,6,7,8-THNol
Pt–Pd/TiO_2 (1 wt.%, Pt/Pd = 3)	300	60	71.6	5.0	10.9	23.1	3.5	54.6
	300	90	74.2	7.4	12.6	24.9	3.9	52.3
	500	60	69.0	3.0	9.3	22.4	3.6	59.6
	500	90	89.1	2.4	16.1	12.5	8.8	57.0
Pt–Pd/TiO_2 (1 wt.%, Pt/Pd = 0.33)	300	60	90.3	2.7	12.4	33.1	12.6	38.1
	300	90	99.2	1.9	27.8	9.0	17.6	41.9
	500	60	90.8	2.3	24.5	8.2	22.6	40.8
	500	90	100	2.1	40.7	7.8	11.0	38.3

[a] All reactions were done at 150°C.

by co-impregnation. One is that one metal can prevent the other metal from agglomerating, the other is that the two metals can combine together and form new species different from the parental ones. It is worthy to mention that there is a specific peak for the bimetallic catalysts, particularly for the one with Pt/Pd weight ratio of 0.3. The TPR peak has even higher temperature than the highest single metal reduction temperature (Pt, 376.3°C). This observation suggests that some new bimetallic phase is formed in this catalyst system which may be responsible for the higher selectivity toward 1,2,3,4-isomer of THNol.

If Pd is the main metal species in the bimetallic catalytic system that leads the reaction towards the formation of 1,2,3,4-THNol, the existence of Pt could prevent the agglomeration of Pd particles during the catalyst preparation and reaction processes. Characterization of both monometallic and bimetallic catalysts was done by H_2 pulse chemisorption in order to measure metal dispersion and metal particle size;

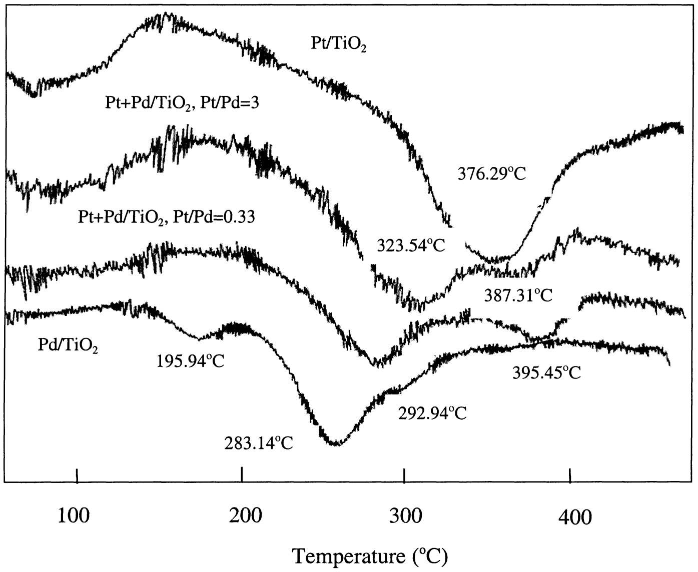

Fig. 1. TPR characterization results.

Table 5
H_2 pulse chemisorption result of TiO_2-supported Pt, Pd catalysts

Catalysts	Metal dispersion	Particle diameter (nm)	Metal surface area (m^2/g sample)
Pt/TiO_2	30.54	3.71	0.76
Pd/TiO_2	18.84	5.95	0.84
Pt–Pd/TiO_2 (Pt/Pd = 3)	66.72	1.70	1.64
Pt–Pd/TiO_2 (Pt/Pd = 0.3)	106.06	0.89	5.62

the results are shown in Table 5. Based on the values of observed H_2 pulse chemisorption data, it is apparent that bimetallic catalysts display much higher metal dispersion and smaller metal particle size compared to monometallic catalysts and the one with Pt to Pd weight ratio of 0.3 has the highest metal dispersion among all the catalysts tested. It should be noted that a stoichiometric ratio of hydrogen: metal of 1:1 was used. The metals in Pt–Pd/TiO_2 (Pt/Pd = 0.3) gave higher than 100% in the value of metal dispersion. These may be cases where the true stoichiometric ratio of hydrogen: metal is higher than the unity. Further verification of these values is needed.

3.4. Reaction network

Product analysis for reactions under various conditions indicated that there are three types of by-products (tetralin, decalin and tetralone) in the reaction system. In order to understand the way in which those products were formed, reactions with different time period, from 2 to 50 min, were carried out and the results are shown in Fig. 2 for the reaction of 1-naphthlol with hydrogen at 150°C and 300 psi. At the beginning of the reaction, 2 min with 1-naphthol conversion of 22%, the yield of THN is zero and the yield of 1,2,3,4-THNol is very low, about 1%. On the other hand, the yield of both tetralone and 5,6,7,8-THNol is relatively high compared to other products found, especially with 5,6,7,8-THNol close to 20%. The results suggest that 1-tetralone and 5,6,7,8-THNol are primary products. The curves representing 1-tetralone, 1,2,3,4-THNol and 5,6,7,8-THNol pass through a maximum, which means that those compounds are intermediates, being converted into one of the other compounds in the network. At about 15 min, 1-tetralone concentration starts to drop sharply, while at the same time, 1,2,3,4-THNol increases significantly. This suggests that 1,2,3,4-THNol is possibly formed by further hydrogenation of tetralone. This is

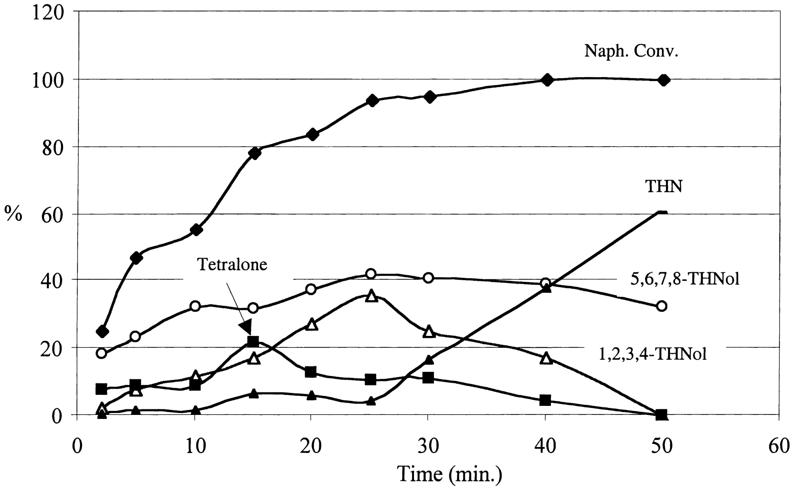

Fig. 2. Hydrogenation of 1-naphthol over Pt–Pd/TiO_2 (Pt/Pd = 0.3), 150°C, 300 psi.

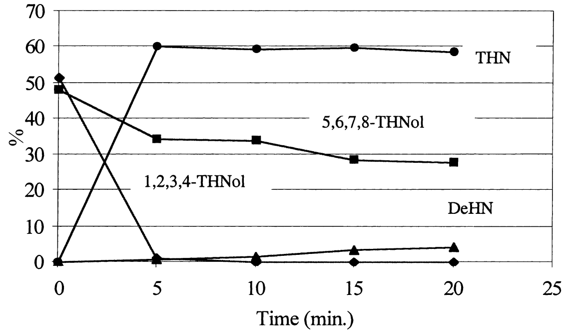

Fig. 3. Hydrogenation of THNol mixture over Pt–Pd/TiO_2 (1 wt.%, Pt/Pd = 0.3), 150°C, 300 psi.

confirmed by tetralone hydrogenation results shown in Fig. 4. At around 25 min, as 1,2,3,4-THNol decreases, the concentration of THN starts to increases dramatically. 5,6,7,8-THNol also contributes to the formation of THN, but in very small amount since the curve representing 5,6,7,8-THNol stays relatively steady during reaction time.

To further clarify the relative reactivity of the intermediates, the hydrogenation of mixture of the two tetrahydro-naphthol isomers (50%:50%) was carried out at the same reaction conditions, 150°C, 300 psi. The results are shown in Fig. 3. Starting with almost half of each isomer, the concentration of 1,2,3,4-THNol dropped to near 0 while the concentration of THN increased to about 60% within the first 5 min. On the other hand, 5,6,7,8-THNol stayed relatively stable. Small amount of DeHN was found after 10 min. The relatively higher stability of 5,6,7,8-THNol than 1,2,3,4-THNol is the result of conjugation effect between hydroxyl group and benzene ring.

Hydrogenation of tetralone was carried out by using Pt and Pd bimetallic catalyst with Pt/Pd weight ratio of 0.3 at 150°C, 500 psi; the reaction results are shown in Fig. 4. 1,2,3,4-THNol and THN were the only products observed under the condition employed. It is apparent that 1,2,3,4-THNol is an intermediate during the hydrogenation and its further hydrogenation leads to the formation of THN.

According to these results, we proposed a reaction network for the hydrogenation of 1-naphthol shown in Fig. 5. The reaction pathway is consistent with the one proposed for the hydrodeoxgenation of 1-naphthol by others [22–24] except that no 1,2,3,4-THNol was observed because of the constant H_2 pressure during the reaction.

On the basis of the results of this study, a reaction mechanism involving adsorption of 1-naphthol on the sites around the interface between the noble metal particles and the support is envisioned for the selective formation of tetralone and 1,2,3,4-THNol. Hydrogenation of phenol to cyclohexanone over Pd/MgO and Pd/Al_2O_3 was reported by Neri et al. [22]. Higher selectivity of cyclohexanone was found on Pd/MgO compared to Pd/Al_2O_3, which is explained by

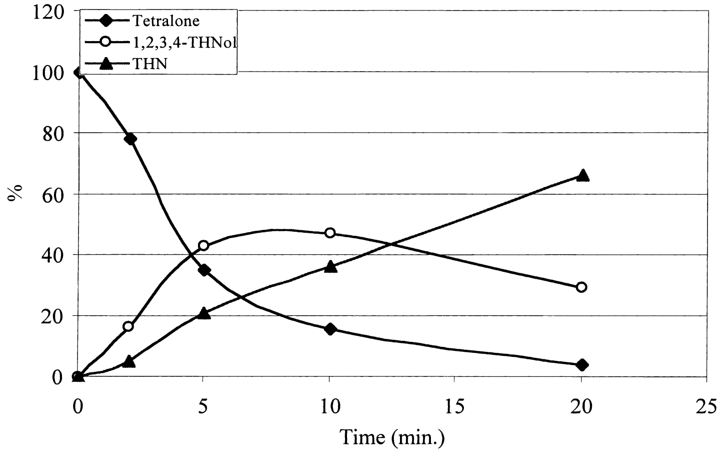

Fig. 4. Hydrogenation of tetralone over Pt–Pd/TiO_2 (1 wt.%, Pt/Pd = 0.3), 150°C, 500 psi.

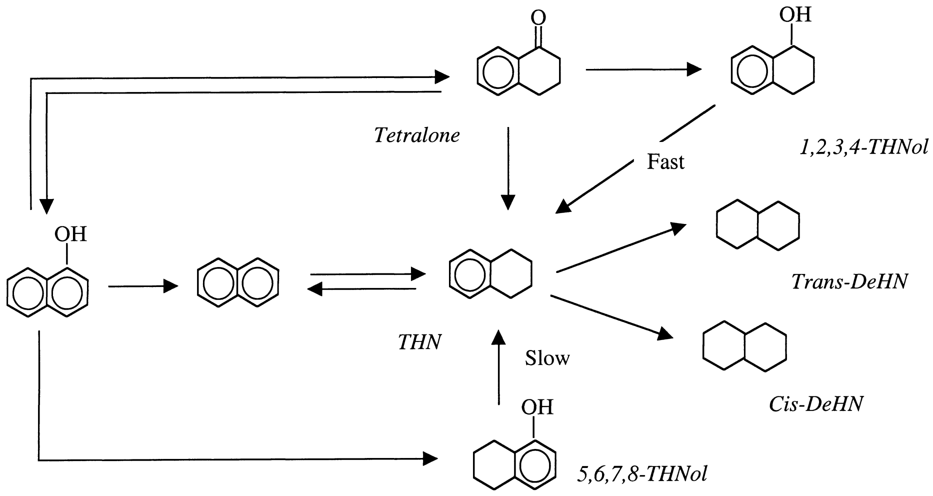

Fig. 5. Reaction network of 1-naphthol hydrogenation at low temperature.

different forms of adsorbed phenol. They concluded that the acidity of the support also plays an important role in selective hydrogenation of benzene ring, rather that removal of hydroxyl group.

4. Conclusion

We have established that the catalytic hydrogenation of 1-naphthol can be tailored to produce more 1,2,3,4-THNol or 5,6,7,8-THNol by selecting appropriate support and noble metal species. Pd supported on TiO_2 catalyst shows higher selectivity than Pt catalyst. Relative to the cases with monometallic catalysts, the selectivity for 1,2,3,4-THNol can be tripled by using bimetallic catalyst Pt–Pd/TiO_2 with appropriate Pt/Pd ratio.

Tetralone has been found to be an intermediate for the hydrogenation of 1-naphthol to 1,2,3,4-THNol. Further hydrogenation of THNol toward tetralin and decalin can occur more easily in the case of 1,2,3,4-THNol than in the case of 5,6,7,8-THNol. Therefore, the control of H_2 pressure, reaction temperature and time period is also very important in order to reach and maintain high 1,2,3,4-THNol selectivity.

A number of fundamental questions remain to be answered in future study, and further work is in progress.

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