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Chemoselective synthesis of imine and secondary amine from nitrobenzene and benzaldehyde by Ni_3Sn_2 alloy catalyst supported on TiO_2

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Keywords: Ni ₃ Sn ₂ /TiO ₂ alloy catalyst Nitrobenzene Benzaldehyde Imine Secondary amine	Ni ₃ Sn ₂ /TiO ₂ alloy catalyst prepared by hydrothermal method was applied for the direct one-pot reductive imination of benzaldehyde with nitrobenzene in the presence of H ₂ gas as a reducing agent. A desired imine, benzylideneaniline, was produced in an excellent yield of 90% without the formation of any by-products. This is due to the chemoselective molecular recognition of Ni ₃ Sn ₂ alloy nanoparticles supported on TiO ₂ for the nitro group over the carbonyl group. By prolonging the reaction time, the desired imine was converted into the corresponding secondary amine, <i>N</i> -phenylbenzylamine, with a remarkably high yield of 80% through the catalytic hydrogenation of the C—N bond. Finally, we demonstrated that it was easy to reuse the catalyst up to five times.

1. Introduction

Nitrogen-containing compounds such as primary, secondary, and tertiary amines, imines, and azo compounds have attracted enormous attention for a long history [1–4]. In particular, imines are highly desirable due to their wide applications as electrophilic reagents for diverse organic reactions such as reductions, additions, condensations, and cycloadditions [5–7]. The formation of the C=N bond involved in the synthesis of imines is one of the most important areas in synthetic organic chemistry [6,8–12].

Traditionally, imines have been synthesized from the dehydrative condensation between primary amines and carbonyl compounds in the presence of an acid catalyst, the self-condensation of primary amines upon oxidation, and the oxidation of secondary amines [7,13–17]. The one-pot conversion of alcohols with primary amines has also been developed for producing imines although a large excess of oxidants was required [7,18]. Compared with these methods using primary amines, one-pot reductive imination of carbonyl compounds directly from nitroarenes is conceived to be especially attractive from the viewpoint of green sustainable chemistry because it does not require prior reduction of nitroarenes [1,19]. In spite of the great progress being made in the selective imine formation, there are scarcely any available reports dealing with the direct one-pot reductive imination of carbonyl compounds with nitroarenes by non-noble metal-based heterogeneous

catalysts.

For the cascade reductive imination to be successful, not carbonyl compounds but nitroarenes should be reduced first. Then, the coupling between *in situ* formed primary amines and carbonyl compounds occurs rapidly without the aid of any catalyst and reaches equilibrium (Scheme 1). From our continuing works on Ni₃Sn₂ alloy catalysis, we discovered that the highly dispersed Ni₃Sn₂ alloy nanoparticles on TiO₂ support were efficient for the chemoselective hydrogenation of a broad range of functionalized nitroarenes [20]. Notably, other reactive groups such as olefin, chloride, ketone, and ester groups remain unaffected before almost complete exhaustion of the nitro group. Due to the prominent efficiency of Ni₃Sn₂/TiO₂ alloy catalyst for the chemoselective nitro hydrogenation, we envisioned that the Ni₃Sn₂/TiO₂ alloy-catalyzed strategy could offer a new efficient protocol for the direct one-pot reductive imination of carbonyl compounds with nitroarenes.

Secondary amines, which are important building blocks used for the synthesis of various functional organic molecules such as polymers, additives, dyes, pharmaceuticals, herbicides, surfactants, and bioactive molecules, can be obtained *via* the one-pot reductive imination of carbonyl compounds with nitroarenes in the presence of H_2 gas as a reducing agent (Scheme 1) [1,21–23]. To the best of our knowledge, the direct synthesis of secondary amines from carbonyl compounds and nitroarenes has been performed mainly by noble metal-based heterogeneous catalysts. In this context, our developed non-noble metal-based

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alloy catalyst Ni_3Sn_2/TiO_2 was applied for the direct one-pot reductive amination of carbonyl compounds with nitroarenes.

2. Experimental section

2.1. Materials

All chemicals were commercially available and used as received without further purification. NiCl₂, SnCl₂·2H₂O, NaOH, TiO₂, nitrobenzene, benzaldehyde, aniline, benzyl alcohol, naphthalene, 1,4-dioxane, *p*-xylene, *o*-xylene, mesitylene, methanol (MeOH), ethanol (EtOH), tetrahydrofuran (THF), triethylamine (NEt₃), and *N*,*N*-dimethylacetamide (DMA) were supplied from Wako. Benzylideneaniline and *N*-phenylbenzylamine were purchased from TCI.

2.2. Catalyst preparation

 Ni_3Sn_2 alloy catalyst supported on TiO_2 were prepared by hydrothermal method in accordance with the procedure reported earlier [20]. Briefly, Ni(II) and Sn(IV) precursors were mixed at Ni:Sn molar ratio of 1.5 at room temperature. TiO_2 was then added into the mixture as a support and stirred for 1 h at room temperature. The resulting solution was hydrothermally treated at 423 K for 24 h, filtered, washed with distilled water, and dried under vacuum at room temperature overnight. Finally, the recovered powder was reduced under H₂ atmosphere at 673 K for 1 h to obtain 10 wt% Ni_3Sn_2/TiO_2 alloy catalyst.

2.3. Catalytic test

The one-pot reaction of benzaldehyde with nitrobenzene was carried out in a high-pressure autoclave with a pressure gauge, a magnetic stirrer, and an oil bath. Typically, Ni_3Sn_2/TiO_2 alloy catalyst (0.10 g), nitrobenzene (1.0 mmol), benzaldehyde (1.0 mmol), naphthalene (0.3 mmol) as an internal standard material, and mesitylene solvent (5.0 mL) were placed into a glass reaction tube and stirred at room temperature for a selected time. After the autoclave was sealed, pure H₂ gas was introduced in order to remove air from the system and kept at a desired pressure, and then the reaction system was heated to a given temperature. After the reaction, the reaction mixture was taken out of the reaction system and analyzed by gas chromatography. The products were identified by gas chromatography (GC-8A, Shimadzu, using a flame ionization detector) equipped with a flexible quartz capillary column coated with Silicon OV-17. The conversion and yields of the products were calculated using an internal standard method.

3. Results and discussion

3.1. Effect of solvent

In this work, we began our study by using nitrobenzene and benzaldehyde as starting materials. The desired products are imine intermediate benzylideneaniline (1) and over-hydrogenated secondary amine *N*-phenybenzylamine (2). The by-product is benzyl alcohol (3).

With Ni₃Sn₂/TiO₂ alloy catalyst, we aimed to evaluate the catalytic property for the one-pot reaction of benzaldehyde with nitrobenzene in different reaction solvents under H₂ 1.0 MPa at 423 K (Table 1). The physicochemical properties such as the particle size of Ni₃Sn₂ alloy, H₂ and CO uptakes, BET surface area, and average pore volume are shown in our previous report [20]. Initially, 1,4-dioxane solvent was employed (Table 1, entries 6 and 7). The favored hydrogenation of nitrobenzene over benzaldehyde proceeded, providing the desired imine benzylideneaniline (1) as a consequence of the condensation between in situ formed aniline and benzaldehyde. No formation of the by-product benzyl alcohol (3) was observed during the early stage of the reaction. This is due to the chemoselective molecular recognition of Ni₃Sn₂ alloy nanoparticles supported on TiO2. The imine yield was 37% at the benzaldehyde conversion of 40% after 12 h. The product distribution differed depending on the reaction time. After 24 h, the other desired secondary amine N-phenylbenzylamine (2) through the catalytic hydrogenation of the C=N bond in benzylideneaniline was obtained in a moderate yield of 50%, along with a large amount of benzyl alcohol. These results indicate that 1,4-dioxane solvent was not suitable for the one-pot synthesis of the nitrogen-containing compounds.

The reaction solvent has been reported to strongly affect the catalytic activity and product distribution [24]. Therefore, the effect of the solvent on the catalytic performance of Ni_3Sn_2/TiO_2 alloy catalyst for the one-pot reaction was investigated to explore the best solvent under identical reaction conditions (H₂ 1.0 MPa, 423 K). The results are summarized in Table 1. The use of protic polar solvents such as MeOH

Table 1

Effect of solvent on the catalytic performance of Ni_3Sn_2/TiO_2 alloy catalyst for the direct one-pot reaction of benzaldehyde with nitrobenzene^a.

Entry	Solvent	Conv (%) ^b	Yield	(%) ^c	Sol (04)d	
	Solvent	COIIV. (70)	1	2	3	361. (70)
1	MeOH	100	0	58	40	58
2	EtOH	100	5	52	37	57
3	DMA	100	17	17	57	34
4	THF	25	22	0	0	88
5	NEt ₃	58	52	0	0	90
6	1,4-Dioxane	40	37	0	0	93
7 ^e		100	5	50	45	55
8	o-Xylene	90	82	0	0	91
9 ^e		100	21	54	25	76
10	Mesitylene	64	64	0	0	100
11 ^e		90	90	0	0	100
12	p-Xylene	78	74	0	0	95
13 ^e		82	78	0	0	95

^a Reaction conditions: nitrobenzene, 1.0 mmol; benzaldehyde, 1.0 mmol; Ni₃Sn₂/TiO₂ alloy catalyst, 0.10 g; solvent, 5.0 mL; naphthalene, 0.30 mmol; H₂, 1.0 MPa; reaction temperature, 423 K; reaction time, 12 h. The conversion and yields were determined by GC using an internal standard technique.

^b Conversion of benzaldehyde.

^c Yields of entries 1, 2, and 3 represent benzylideneaniline, *N*-phenylbenzylamine, and benzyl alcohol, respectively, as shown in Scheme 1.

^d Selectivity to benzylideneaniline and *N*-phenylbenzylamine.

e Reaction time, 24 h.



Scheme 1. Synthetic pathway of nitrogen-containing compounds starting from nitrobenzene and benzaldehyde.

and EtOH produced the highest catalytic activity but had a negative impact on the selectivity to the imine and secondary amine (Table 1, entries 1 and 2). Some researchers have proposed that a strong interaction between protic polar solvents (e.g., C1-C4 primary alcohols) and 2-butanone by hydrogen bonding dramatically lowers the activation energy barrier and significantly enhances the catalytic activity [25,26]. The catalytic activity of Ni₃Sn₂/TiO₂ alloy catalyst in aprotic polar DMA solvent, was next to protic polar solvents (Table 1, entry 3). The use of other aprotic polar solvents such as THF, NEt₃, and 1,4-dioxane led to low-to- moderate catalytic activity (Table 1, entries 4-6). This is probably due to the strong adsorption of the solvent molecules on the catalyst surface blocking the accessibility of the active Ni sites [26-28]. Non-polar solvents such as *p*-xylene, *o*-xylene, and mesitylene showed a lower catalytic activity but provided the imine in higher yields without a hint of benzyl alcohol formation, when compared with aprotic polar DMA solvent (Table 1, entries 8, 10, and 12). It can be concluded that the catalytic activity is higher in a more polar solvent with the following order: protic polar solvent > aprotic polar solvent > non-polar solvent.

The direct one-pot reaction of benzaldehyde with nitrobenzene in the non-polar solvents steadily proceeded with increasing the reaction time (Table 1, entries 9, 11, and 13). Although the complete conversion of benzaldehyde was achieved using *o*-xylene after 24 h, the yield of the imine largely decreased and the secondary amine was obtained in 54% yield together with the by-product (Table 1, entry 9). In contrast, selective formation of the imine was accomplished by employing mesitylene and *p*-xylene (Table 1, entries 11 and 13). The benzaldehyde conversions were 90% and 82% less than 100%, respectively. On the basis of these results, it can be assumed that the formation of the desired secondary amine *N*-phenylbenzylamine and the by-product benzyl alcohol is inhibited before the complete exhaustion of benzaldehyde. Mesitylene yielded the desired imine compound almost quantitatively and therefore was shown to be the best solvent for the Ni₃Sn₂/TiO₂ alloy-catalyzed reductive imination of benzaldehyde with nitrobenzene.

Fig. 1 presents the time profile for the Ni₃Sn₂/TiO₂ alloy-catalyzed one-pot reaction of benzaldehyde with nitrobenzene in mesitylene. At a reaction time of 6 h, the *in situ* formed aniline (\blacklozenge) was condensed with benzaldehyde, giving rise to only the desired imine product (\blacktriangle). The yield of the imine (\bigstar) steeply increased with increasing the reaction time along with a small amount of aniline (\blacklozenge), and it reached 90% after 24 h. The formation of the corresponding secondary amine (\blacklozenge) was observed at 30 h when the benzaldehyde conversion was almost full, more precisely, 98%. The yield of the secondary amine (\blacklozenge) dramatically increased to a remarkably high yield of 80% at only 6 h. The control of



Fig. 1. Time profile for the one-pot reaction of benzaldehyde with nitrobenzene by Ni_3Sn_2/TiO_2 alloy catalyst in mesitylene. Reaction conditions: nitrobenzene, 1.0 mmol; benzaldehyde, 1.0 mmol; Ni_3Sn_2/TiO_2 alloy catalyst, 0.10 g; mesitylene, 5.0 mL; naphthalene, 0.30 mmol; H₂, 1.0 MPa; reaction temperature, 423 K.

the reaction time in mesitylene can allow an efficient one-pot synthesis of the imine and secondary amine. Notably, the yield of the secondary amine (\bullet) was slightly lower than that of the parent imine (\blacktriangle). This is because *in situ* formed H₂O resulted in recovery of aniline (\blacklozenge) and benzaldehyde as a consequence of the imine hydrolysis [29].

3.2. Effect of the addition of molecular sieve 3 Å

The reverse transformation of the imine benzylideneaniline into benzaldehyde and aniline in the presence of the *in situ* produced water prompted us to add molecular sieve 3 Å into the catalytic system for the removal. The time profile is illustrated in Fig. 2. In comparison with Fig. 1, it has been proven that the addition of the molecular sieve plays a significant role in accelerating the imine formation step during the present one-pot reaction. However, the reverse reaction into aniline and benzaldehyde proceeded regardless of the presence of the molecular sieve. A similar yield of the imine and secondary amine to that of the catalytic system without the molecular sieve was obtained at shorter reaction times of 19 h and 30 h, respectively.

3.3. Effect of H_2 pressure

Optimization of the reaction conditions was carried out for the onepot reaction of benzaldehyde with nitrobenzene using Ni_3Sn_2/TiO_2 alloy catalyst. First, the effect of H₂ pressure was investigated at 423 K in mesitylene. The results are given in Table 2. A lower H₂ pressure of 0.6 MPa decreased the yield of the imine (Table 2, entry 1). This is because the hydrogenation of nitrobenzene to aniline is sensitive to the initial H₂ pressure. The results presented in Table 2, entries 1 and 2 show that the kinetic behavior of Ni₃Sn₂/TiO₂ alloy catalyst during this process enables the selective formation of the imine before the complete exhaustion of benzaldehyde. To further improve the catalytic efficiency, we performed the one-pot reaction under a higher H₂ pressure of 2.0 MPa (Table 2, entry 3). In this situation, the imine was converted into the corresponding secondary amine in a moderate yield, accompanied by the unwanted side product because the benzaldehyde conversion reached 100%.

3.4. Effect of reaction temperature

Next, the effect of reaction temperature was studied under H_2 1.0 MPa in mesitylene. The results are listed in Table 3. A relatively high



Fig. 2. Time profile for the one-pot reaction of benzaldehyde with nitrobenzene by Ni_3Sn_2/TiO_2 alloy catalyst in mesitylene. Reaction conditions: nitrobenzene, 1.0 mmol; benzaldehyde, 1.0 mmol; Ni_3Sn_2/TiO_2 alloy catalyst, 0.10 g; mesitylene, 5.0 mL; naphthalene, 0.30 mmol; H₂, 1.0 MPa; reaction temperature, 423 K; molecular sieve 3 Å, 0.10 g.

Table 2

Effect of H_2 pressure on the catalytic performance of Ni_3Sn_2/TiO_2 alloy catalyst for the one-pot reaction of benzaldehyde with nitrobenzene^a.

Entry	H ₂ pressure (MPa)	Conv. (%) ^b	Yield (%) ^c			0-1 (0/)d
			1	2	3	Sel. (%)
1	0.6	73	68	0	0	93
2	1.0	90	90	0	0	100
3	2.0	100	20	54	22	74

^a Reaction conditions: nitrobenzene, 1.0 mmol; benzaldehyde, 1.0 mmol; Ni₃Sn₂/TiO₂ alloy catalyst, 0.10 g; mesitylene, 5.0 mL; naphthalene, 0.30 mmol; reaction temperature, 423 K; reaction time, 24 h. The conversion and yields were determined by GC using an internal standard technique.

^b Conversion of benzaldehyde.

^c Yields of entries 1, 2, and 3 represent benzylideneaniline, *N*-phenylbenzylamine, and benzyl alcohol, respectively, as shown in <u>Scheme 1</u>.

^d Selectivity to benzylideneaniline and *N*-phenylbenzylamine.

Table 3

Effect of reaction temperature on the catalytic performance of Ni_3Sn_2/TiO_2 alloy catalyst for the one-pot reaction of benzaldehyde with nitrobenzene^a.

Entry	Temp. (K)	Conv. (%) ^b	Yield (%) ^c			Sel.
			1	2	3	(%) ^d
1	403	81	75	0	0	93
2	423	90	90	0	0	100
3	443	100	54	22	20	76

^a Reaction conditions: nitrobenzene, 1.0 mmol; benzaldehyde, 1.0 mmol; Ni₃Sn₂/TiO₂ alloy catalyst, 0.10 g; mesitylene, 5.0 mL; naphthalene, 0.30 mmol; H₂, 1.0 MPa; reaction time, 24 h. The conversion and yields were determined by GC using an internal standard technique.

^b Conversion of benzaldehyde.

^c Yields of entries 1, 2, and 3 represent benzylideneaniline, *N*-phenylbenzylamine, and benzyl alcohol, respectively, as shown in <u>Scheme 1</u>.

^d Selectivity to benzylideneaniline and *N*-phenylbenzylamine.

yield of 75% for the imine was obtained at 403 K. The increase of the reaction temperature to 443 K increased the benzaldehyde conversion to 100%. This is why the formation of the secondary amine and benzyl alcohol occurred.

3.5. Reusability test

Using the optimized reaction conditions, the reusability of Ni₃Sn₂/ TiO₂ alloy catalyst was investigated for the one-pot reaction of benzaldehyde with nitrobenzene, and the result is shown in Table 4. After the reaction, the catalyst was separated from the reaction solution by centrifugation, thoroughly washed with acetone, dried in vacuum overnight, and then reused for the next run under the same reaction conditions. Ni₃Sn₂/TiO₂ alloy catalyst can be reused at least five times for the one-pot reaction. The selectivity of the imine remained constant at 100% during a period of five successive runs. A slight decay in the catalytic activity was observed after the first run, likely due to the oxidation of the catalyst surface [30]. However, XRD analysis showed that the spent catalyst had no appreciable change relative to the fresh catalyst (Fig. 3). The XRD patterns of the fresh and spent catalysts exhibited three characteristic peaks at $2\theta = 30.8^{\circ}$, 42.5° , and 44.2° , which correspond to the Ni₃Sn₂ (101), Ni₃Sn₂ (102), and Ni₃Sn₂ (110) diffraction peaks, respectively [31].

4. Conclusions

We have developed a simple, eco-friendly, and cost-effective catalytic system for the direct one-pot reductive imination and amination of benzaldehyde with nitrobenzene. The relationship between the catalytic activity and solvent properties used was revealed. In non-polar mesity-lene solvent, Ni_3Sn_2/TiO_2 alloy catalyst transformed the starting

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Table 4

Reusability test of Ni_3Sn_2/TiO_2 alloy catalyst for the one-pot reaction of benz-aldehyde with nitrobenzene^a.

Run	1	2	3	4	5
Conv. (%) ^b	90	85	80	78	75
Sel. (%) ^c	100	100	100	100	100

^a Reaction conditions: nitrobenzene, 1.0 mmol; benzaldehyde, 1.0 mmol; Ni₃Sn₂/TiO₂ alloy catalyst, 0.10 g; mesitylene, 5.0 mL; naphthalene, 0.30 mmol; H₂, 1.0 MPa; reaction temperature, 423 K; reaction time, 24 h. The conversion was determined by GC using an internal standard technique.

^b Conversion of benzaldehyde.

^c Selectivity to benzylideneaniline.



Fig. 3. XRD patterns of (a) the fresh and (b) spent Ni₃Sn₂/TiO₂ alloy catalysts.

materials into the desired imine benzylideneaniline in an excellent yield of 90% after 24 h, without the formation of the by-product benzyl alcohol. The catalyst efficiency is ascribed to the high chemoselectivity for the nitro group over the carbonyl group. Prolonging the reaction time to 36 h produced the corresponding secondary amine *N*-phenylbenzylamine with a remarkably high yield of 80% through the catalytic hydrogenation of the C=N bond. The addition of the molecular sieve into the catalytic system has a positive effect on the reaction time to produce the imine and secondary amine. In summary, the one-pot reductive imination and amination of benzaldehyde with nitrobenzene was accomplished using Ni₃Sn₂/TiO₂ alloy catalyst under the optimized reaction conditions (H₂ 1.0 MPa, 423 K). This is the first time that the two nitrogen-containing compounds have been synthesized over a nonnoble metal catalyst using an easily available and environmentally benign hydrogen source.

CRediT authorship contribution statement

Nobutaka Yamanaka: Conceptualization, Methodology, Validation, Writing - original draft. Takayoshi Hara: Data curation. Nobuyuki Ichikuni: Data curation. Shogo Shimazu: Supervision, Writing - review & editing, Conceptualization.

Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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