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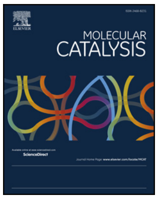

Selective amination of 1,2-propanediol over Co/La_3O_4 catalyst prepared by liquid-phase reduction

Chuan-Jun Yue*, Kai Di, Li-Ping Gu, Zhen-Wei Zhang, Lin-Lin Ding

School of Chemical Engineering and Material, Changzhou Institute of Technology, Changzhou 213022, PR China

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ABSTRACT

The catalytic coupling of alcohol and ammonia is an environmentally friendly process. Cobalt-based catalysts, modified by supports (including CeO_2, Fe_3O_4, Nb_2O_5, La_3O_4 and Al_2O_3), and prepared by the liquid-phase reduction, were used for the amination of 1,2-propanediol. The screened nano-Co/La_3O_4 catalyst exhibited an excellent catalytic performance of 68% conversion and 89% selectivity toward 2-amino-1-propanol under optimal conditions. The characterizations of the catalyst was performed by XRD, XPS, BET, TEM, TG, and CO_2-TPD, revealing a relatively large specific surface area, strongly alkaline sites and a Co-La-O transition phase, which were responsible for the selective catalysis of 1,2-propanediol. The efficient construction of cobalt-based catalysts on the basis of the active species is key to improving the efficiency of the reaction process.

1. Introduction

Organic compounds containing C— bonds are often important functional molecules; hence they are not only found as the structural components of active compounds such as vitamins, hormones, alkaloids, neurotransmitters, and natural toxicants, but are also widely used in fine chemicals such as agrochemicals, pharmaceuticals, detergents, dyes, food additives, and lubricants [1,2]. Among all amines, primary amines are usually precursor molecules for the synthesis of secondary amines, tertiary amines, and quaternary ammonium salts.

To date, there are more than ten types of synthetic methods for the preparation of primary amines, including the nucleophilic substitution of halogenated hydrocarbons [3], the catalytic hydrogenation of nitrile and nitro compounds [4], the conversion of carbonyl compounds [5], the catalytic addition of olefins [6], and the catalytic coupling of alcohols and ammonia [7]. From the view of green chemical, especially when concerning non-toxic and sustainable raw materials, catalytic coupling by the aforementioned methods are promising route for the green synthesis of primary amines, for there are a large number of alcohols in the biomass, which can safely and sustainably substitute the petrochemical feed stock in chemical industry [8]. Glycerol is a typical biomass that has garnered widespread attention, and can be converted to all kinds of fine chemicals, such as 1,2-propanediol, propanol, and glycol, providing alternative materials for the chemical processes [9–11]. Theoretically, primary amines are attainable by the removal of water from the reaction between affordable ammonia and biomass alcohols. However, the improvement of the conversion and selectivity of the reaction remains an issue [12]. Through this work, the efficient construction of a catalyst is the core means of addressing this problem.

Catalyzing the amination of alcohols requires designing an effective catalyst for matching the reaction process to the substrate. As an example, it is known that the catalytic amination of mono alcohols proceeds through the homogeneous catalytic system represented by the [Ir] and [Ru] complexes [13,14] and the heterogeneous catalytic system represented by nickel- and ruthenium-based catalysts [15–17] and bimetallic catalytic systems [18,19]. The tuning of the support or ligand to the metal center is one of the strategies for effective catalyst design. For nickel-based catalysts, Ni/Al_2O_3 catalyst by two preparation methods exhibited good catalytic amination activity for secondary alcohols [20] and selectivity for nitrile product [21], respectively, nickel with the highly alkaline support LaAlSiO contributed to the selective catalytic amination of isopropanol [22], and an Al-Ce support enhanced the catalytic activity of Ni for the amination of 1-octadecyl alcohol [23]. These studies improved the understanding of the catalytic process and revealed that the active center of the catalyst possessed an excellent dehydrogenation and hydrogenation nature. On the other hand, it was found that the catalytic amination of glycerol by Ru/C exhibited higher activity but the poor selectivity with respect to the same reaction conditions with polyols [24]. It is well known that there are many primary and secondary hydroxyl groups present in biomass, and 1,2-propanediol is suitable as a model polyol for the fundamental research in the catalytic amination, as it contains only a primary and a secondary

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* Corresponding author.
E-mail address: ywangjung@163.com (C.-J. Yue).

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hydroxyl group. In addition, the 2-aminpropanol product obtained from 1,2-propanediol is an important intermediate for the pharmaceutical Levofloxacin. Nevertheless related reports are few although cobalt-based and ruthenium-based catalytic systems had an effect on the catalytic amination of 1,2-propanediol [25,26], with high activity and selectivity under the mild reaction conditions. Fortunately, cobalt-based catalysts exhibited the potential to efficiently catalyze the amination reaction under relatively mild conditions, and the controlled cobalt-based catalyst could also catalyze the dehydrogenation of inert alkanes [27,28]. Thus it is necessary to prepare a highly efficient cobalt-based catalyst to be applied for the amination of 1,2-propanediol.

The liquid reduction method is one simple and effective ways to prepare the active components of the heterogeneous catalyst [29,30]. In this paper, cobalt-based catalysts with different textures were prepared by this method and used in the catalytic amination of 1,2-propanediol.

2. Experimental

2.1. Materials

All the reagents used were analytical grade and without further purification before use, and the purity of nitrogen was 99.999%.

2.2. Preparation and characterization of catalysts

Catalyst preparation was based on reported procedure with slight modification [31]. A total of 1 g of $CoCl_2·6H_2O$ was dissolved in a three-necked flask with 20 mL of absolute ethanol, followed by the addition of a certain amount of the support. A paste containing 3.5 g of NaOH and 3 mL of 80% $N_2H_4·H_2O$ was added in a dropwise fashion with vigorous stirring. After 20 min, 10 mL of ethanol containing 0.001 g $NaBH_4$ was added dropwise. After complete reduction, the reaction system was filtered. The filter cake was completely washed with deionized water and ethanol, and was transferred to vacuum chamber at 120 °C for 1 h, and finally calcined at 180 °C for 2 h under a nitrogen atmosphere. The catalyst powder was obtained by cooling to room temperature.

The catalysts were characterized by the undermentioned technologies. X-ray diffraction (XRD) patterns were collected on D/MAX2500 V (Rigaku, Japan) for bulk phase and structure analysis. Specific surface area measurements (BET method) were carried out by an ASAP 2000 physical adsorption instrument (Micromeritics Instrument Corp.). The oxidation state and surface composition were recorded using an X-ray photoelectron spectrometry (XPS) (Kratos Ultra Axis DLD). Transmission electron microscopy (TEM) images were acquired using a JEOL JEM-1230 transmission electron microscope. Thermostabilities of the samples were determined on PerkinElmer's Pyris 1 TGA device (Setsys 170 Thermogravimetric Analyzer, Setaram, France), the experiments were carried out in N_2 atmosphere from room temperature to 500 °C at a ramp rate of 5 °C/ min. Temperature programmed desorption (CO_2-TPD) measurement was carried out on Micromeritics Auto Chem II 2920 apparatus. The CO_2 adsorption was performed at 50 °C for 30 min by using CO_2/He mixture gas(volume ratio, 10/90) at a flow rate of 25 $mL·min^{-1}$.

2.3. Procedure for catalytic reaction

The typical procedure for the catalytic amination of alcohol was as follows. A total of 2 mL of 1,2-propanediol, 18 mL of aqueous ammonia and a certain amount of cobalt-based catalyst were placed in a nitrogen atmosphere reactor. The reactor was sealed after nitrogen blowing with a slight agitation, and then nitrogen gas was charged to 10 atm. The reaction system was then heated for a certain period of time. After the completion of the reaction, the catalyst was filtered and the filtrate was analyzed with a GC or GC–MS (GCQ, Thermo Finnigan) spectrometer to determine the conversion of 1,2-propanediol and the selectivity toward 2-aminopropanol by the quantitative method using isopropanol as an internal standard.

Table 1
Effect of support on the reaction of 1,2-propanediol.

Support	Conversion/%	Selectivity/%			
		2A1H	1A2H	1A2A	Others
none	< 1	n.d.	n.d.	n.d.	100
CeO_2	3.1	43.8	26.7	10.8	18.7
Fe_3O_4	24.7	49.2	30.4	15.3	5.1
Nb_2O_5	36.7	39.9	35.9	13.5	10.7
La_3O_4	39.9	75.0	17.8	3.0	4.2
Al_2O_3	49.7	53.7	20.6	12.7	13.0

Reaction conditions: 0.1 g catalyst with 10% Co loading, 2 mL 1,2-propanediol, 18 mL aqueous ammonia, 6 h, 160 °C. 2A1H: 2-amino-1-propanol, 1A2H:1-Amino-2-propanol, 1A2A: 1, 2-diamino propane,others:propylene glycol ether etc.

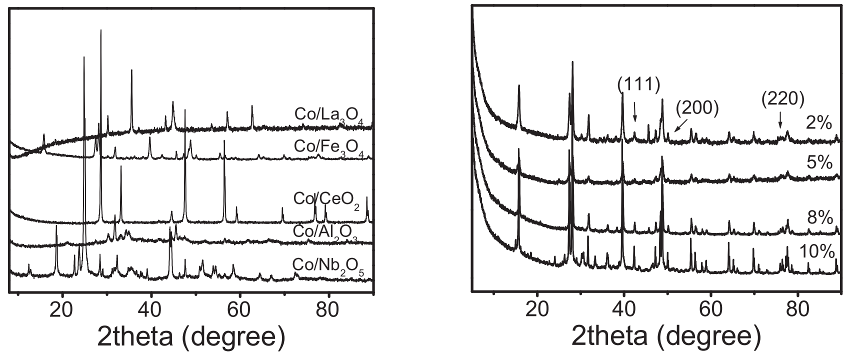
3. Results and discussion

3.1. Screening of catalytic system based on cobalt

The cobalt-based catalyst exhibited activity in the catalytic amination of 1,2-propanediol while the modification of the support to cobalt contributed to the further improvement of the catalytic efficiency [25].

Table 1 shows that amination results of 1,2-propanediol catalyzed by cobalt-based catalysts with different supports prepared by liquid-phase reduction. The catalytic amination of 1,2-propanediol over cobalt modified by lanthanum and alumina oxide showed excellent selectivity (75.0%) to 2A1H (2-amino-1-propanol) and conversion (49.7%) while other modified cobalt catalysts did not present better catalytic efficiency. At the same time, other products like 1A2H (1-Amino-2-propanol) and 1A2A (1,2-diamino propane) were formed in the catalytic amination by supported cobalt. Obviously, the catalytic activity of elemental cobalt was poor, revealing a relationship between catalysis and the support material. The results of XRD analysis of the catalysts are shown in Fig. 1(a). Cobalt modified by different supports had differing phase structures; the powdering of alumina was higher while the crystal face of cerium oxide was relatively complete. In addition, there were different degrees of acid or base active sites on the supports [32]. From the catalytic selectivity of the product, lanthanum oxide was found to be a suitable alternative support.

The molar ratio of cobalt to lanthanum in Co/La_3O_4 catalysts had an effect on the catalytic performance. Table 2 shows the results of amination of 1,2-propanediol catalyzed by Co/La_3O_4 with different Co/La molar ratio. The variance in Co/La molar ratio did not bring obvious changes to the catalytic efficiency, inferring that the catalytic efficiency was closely related to the effective interfacial interaction between cobalt and La_3O_4 [33]. When Co/La molar ratio increased from 0.14 to 0.30, the decreased amplitude of conversion only was 28.6%. The highest conversion and selectivity to 2A1H at the Co/La molar ratio of 0.14 were 68.6 and 89.3%, respectively,with the lowest selectivity for 9.4% 1A2H, less than 1% 1A2A and 1.3% other products. When Co/La molar ratio was 0.06, the catalytic efficiency was low due to the support masking the cobalt thus preventing effective interfacial contact with 1,2-propanediol. XRD showed that each catalyst had a diffraction peak characteristic of metallic cobalt at the double diffraction angle of 44.5, 51.8 and 76.3. At the Co/La molar ratio of 0.14, the peak of the surface pointing to the (111) crystal plane at 44.5 disappeared, and it was presumed that it had a strong interaction with the lanthanum oxide interface. However, other low diffraction peaks indicated that the pulverization degree of the catalyst was relatively high. These results helped to elucidate the structure-activity relationship of catalytic amination.



(a) (b)

Fig. 1. XRD patterns for Cobalt-based catalysts (a) and La_3O_4 support (b).

Table 2
Effect of Co/La molar ration on the reaction of 1,2-propanediol amination.

Co/La (mol)	Conversion/%	Selectivity/%			
		2A1H	1A2H	1A2A	Others
0.30	40.0	75.0	17.9	2.9	4.2
0.24	53.9	78.2	15.3	2.1	4.4
0.14	68.6	89.3	9.4	< 1	1.3
0.06	11.2	29.1	20.3	2.2	48.4

Reaction conditions: 0.1 g catalyst, 2 mL 1,2-propanediol, 18 mL aqueous ammonia, 6 h, 160 °C.

3.2. Characterization of the catalyst

In order to further reveal the structure-activity relationship of catalytic amination, the Co/La_3O_4 (labeled as Cat0) catalyst with 5% cobalt loading was characterized by XPS, TEM, BET, CO_2-TPD, and TG.

Fig. 2 shows the XPS results for Cat0. The general image (outer figure) indicates that the catalyst contains elements such as lanthanum, cobalt, and oxygen, which were consistent with the element composition of the reagents used during catalyst preparation. The bond energy analysis (internal figure) of cobalt indicates that there were two peaks near 778 and 792 eV, that the former was attributed to the Co2p bond energy peaks of Co, and the latter was ascribed to oxidized cobalt and Co, respectively. From the peak area, the content of the oxidation state of cobalt was relatively few from estimation based on split peak fitting [34]; thus the liquid-phase reduction method used to prepare the Cat0 catalyst was sufficient to ensure that the cobalt was reduced to a simple substance. Also, there was almost no possibility that the cobalt was oxidized during the preparation of the nitrogen atmosphere. Therefore, it could be inferred that the oxygen of CoO was likely derived from the lanthanum oxide, that is, cobalt and lanthanum oxide worked each other in the post-processing of preparation, which was consistent with the results revealed by XRD wherein a Co-La-O transition interface point was identified in Cat0.

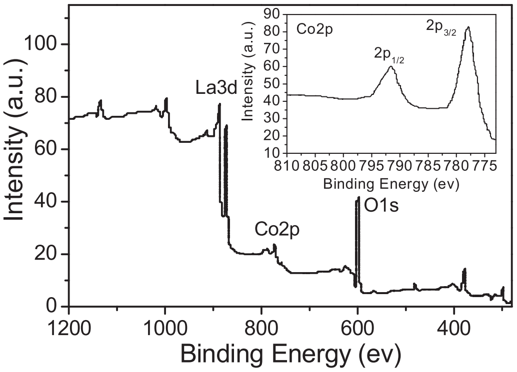


Fig. 2. XPS survey spectrum (outside) and Co2p photoelectron spectra from Co/La_3O_4(inside).

Fig. 3 shows the TEM image of Cat0. In general, the particles of the catalyst were dispersed and the texture was loose. This porosity was not only manifested as an overall accumulation, but also by the aggregated of small particles. The degree of partial aggregation of small particles was relatively low, due to the tendency of the two phases of cobalt and lanthanum oxide had to disperse to form a uniform phase, while the formation of CoO prevented the further fusion of the two phases to some extent [33]. Since the preparation of the catalyst by the above method did not require high temperature calcination [31]. Based on statistical calculation, the size of the particles was about 50 nm on average, although due to the loose aggregation the actual particle size was below 10 nm. This was reliably the same as the particle size found by XRD analysis, and the presence of the transient interface exhibited the above-described catalytic amination performance.

Fig. 4 shows the results of nitrogen adsorption and desorption of Cat0. From the perspective of a loop, it clearly presented that the catalyst had mesoporous characteristics, which were constructed by the interaction of cobalt and lanthanum oxide, especially the (111) crystal plane of cobalt. Macroscopically, loose particles accumulate to form mesopores, as seen in the XRD and TEM results. As a result, a relatively large specific surface area was obtained with BET and Langmuir surface areas of 5.05 and 30.6 m^2/g, respectively, which were larger than the usual specific surface area of lanthanum oxide. The large surface area contributed to the reactive adsorption interface of the molecules.

The surface basicity of Cat0 was characterized by CO_2-TPD. As shown in Fig. 5, there were two types of basic sites of different strength on the surface of the catalyst. As the desorption temperature rose to 580.9 and 732.6 °C, the almost equivalent peaks appeared, which corresponded to the medium-strength and strong basic position, respectively, originating from the oxygen of the isolated hydroxyl and the bridging oxygen on the surface of the lanthanum oxide support [32]. The presence of two types of basic sites and the intrinsic Lewis acid sites of the lanthanum facilitated the adsorption of the substrate for catalysis.

Thermogravimetric analysis of Cat0 was also performed (see supplemental material), primarily reflecting the stability properties of the catalyst. As temperature increased, only a dehydration process was seen; no new species or phase formation or transformations were seen, indicating that the catalyst was relatively thermostable.

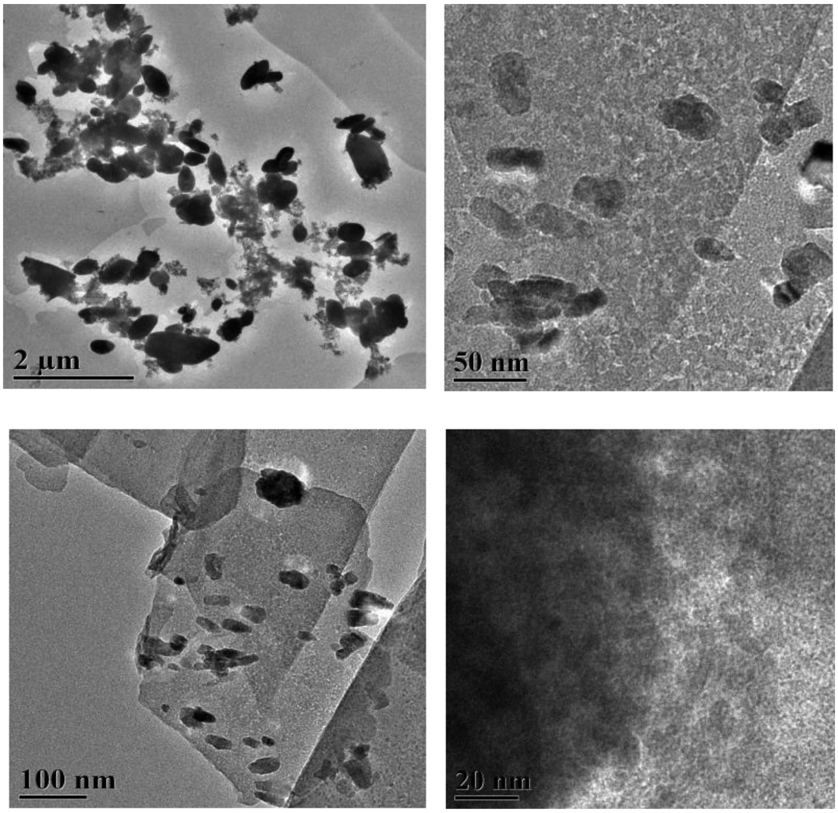


Fig. 3. TEM images for Cat0.

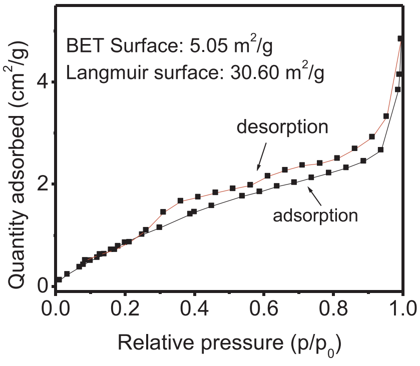


Fig. 4. N_2 adsorption-desorption isotherms for Cat0.

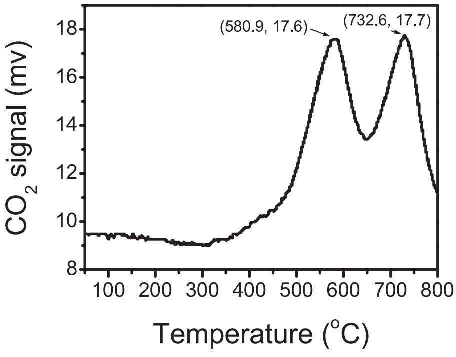


Fig. 5. CO_2-TPD profiles for Cat0.

Table 3
Effect of reaction temperature on the reaction of 1,2-propanediol amination.

T/ °C	Conversion/%	Selectivity/%			
		2A1H	1A2H	1A2A	Others
120	30.8	77.5	15.8	5.7	1.0
160	68.6	89.2	9.6	< 1	1.2
180	70.3	75.4	8.4	6.8	9.4
200	72.1	55.8	5.9	6.5	31.8

Reaction conditions: 0.1 g catalyst with 5% loading, 2 mL 1,2-propanediol, 18 mL aqueous ammonia, 6 h.

Table 4
Effect of reaction time on the reaction of 1,2-propanediol amination.

t/h	Conversion/%	Selectivity/%			
		2A1H	1A2H	1A2A	Others
4	50.4	56.5	37.4	5.1	< 1
6	68.6	89.4	9.4	< 1	1.2
8	75.3	80.1	10.5	7.2	2.6
10	78.7	75.1	8.3	10.9	5.7

Reaction conditions: 0.1 g catalyst with 5% loading, 2 mL 1,2-propanediol, 18 mL aqueous ammonia, 160 °C.

3.3. *Catalytic performance*

The efficiency of the amination of the representative polyol 1,2-propanediol was related to reaction conditions other than the composition and texture of the catalyst with reaction temperature and time as the main influencing parameters.

Table 3 lists the effect of temperature on the reaction. As the reaction temperature increased

from 120 to 200 ℃, the conversion increased from 30.8% to 72.1%.

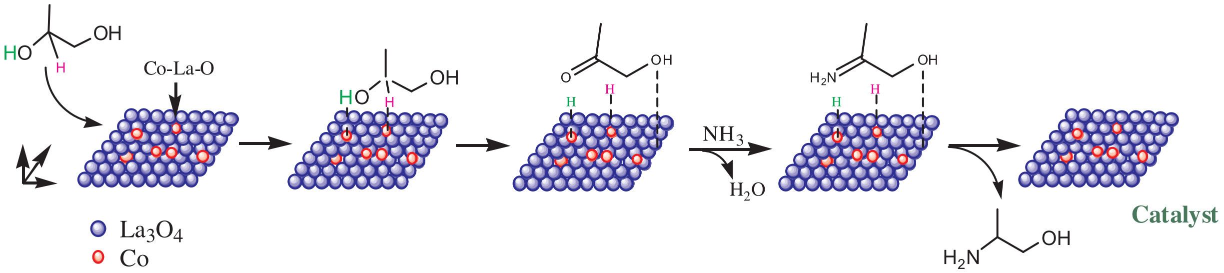


Fig. 6. Schematic diagram of selective amination process for 1,2-propanediol.

When the temperature was higher than 160 °C, the selectivity toward 2A1H decreased from 89.3 to 55.8%, while the 1A2H selectivity was right decreased with the elevated temperature, because the product was condensed with the feed stock as increased temperatures, resulting in the consumption of the product. Compared with the reported results [25], the cobalt-based catalyst applied here showed a relatively high catalytic efficiency at low temperatures. The method of catalyst preparation led to the specific texture of the catalyst. The Co/La$_3$O$_4$ prepared by liquid-phase reduction method exhibited simple cobalt and a relatively high specific area, which helped with the catalysis of surface reactions such as dehydrogenation, condensation and hydrogenation.

The reaction time was an important parameter for evaluating catalytic efficiency (Table 4). As the reaction time increased, conversion also increased from 50.4 to 78.7% at 4 and 10 h, respectively, though the selectivity to 2A1H of the reaction decreased from 89.3% due to further conversion of the desired product to 1A2A. In addition, the results presented that the catalyst had a distinct catalytic kinetic process for 2A1H,1A2H and 1A2A.

According to the above test results and the relative process reported [15–24,35–37], the catalytic dehydrogenation of the hydroxyl group was recognized as the key step prompted by the excellent dehydrogenation property of cobalt. However, the single cobalt was unable to complete the selective amination of 1,2-propanediol. Therefore the presence of basic lanthanum oxide was required to complete the selectively catalytic reaction (Fig. 6). So, it could be interpreted that the dehydrogenation process was performed on the Co-La-O interface where the higher acidity of the secondary hydrogen with respect to the primary hydrogen resulted in the reaction selectivity based on the basicity of the catalyst. GC—MS analysis of the product showed no intermediate ketone, thus the Co-H species was formed after the catalytic reaction dehydrogenation on the Co at the interface. This species had been confirmed through experimentation [38]. After dehydrogenation, the active intermediate reacted with ammonia to form an imine group which was then hydrogenated by Co-H to obtain the aminatived product.

4. Conclusions

Cobalt-based catalysts can be prepared by the liquid-phase reduction, resulting in simple cobalt with dehydrogenation ability and tunable catalytic performance based on support modification. Co/La$_3$O$_4$ presented an effective catalytic activity for the amination of 1,2-propanediol, indicating that catalytic activity was related to the catalyst with the special structure derived from the preparation method. The catalyst prepared by liquid-phase reduction had an efficient active species for the selective amination of 1,2-propanediol. Compared with the catalyst prepared by co-precipitation method reported in the literature [25], not only was the surface area enhanced, but also catalytic active species were found directly present in the interface of the support. As a result, the efficiency of the catalytic reaction was improved. In addition, the reaction conditions were relatively mild. In future work, the improvement of the catalyst based on its active species will be key to further enhancement of catalytic dehydration and condensation efficiency.

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Appendix A. Supplementary data

Supplementary material related to this article can be found, in the online version, at doi:https://doi.org/10.1016/j.mcat.2019.110539.

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