

Hydrogenation of Cinnamaldehyde over an Ionic Cobalt Promoted Alumina-Supported Palladium Catalyst

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ABSTRACT

Nano γ -alumina-supported transition metal-based mono- ($\text{Pd}^0/\gamma\text{-Al}_2\text{O}_3$ or $\text{Co}^{\text{III}}/\gamma\text{-Al}_2\text{O}_3$) and bimetallic catalysts ($\text{Pd}^0\text{Co}^{\text{III}}/\gamma\text{-Al}_2\text{O}_3$), were prepared by wet impregnation, oxidation and *in situ* reduction of the palladium. The palladium(II) acetate [$\text{Pd}^{\text{II}}_3(\text{OOCCH}_3)_6$], cobalt(II) acetate [$\text{Co}^{\text{II}}(\text{OOCCH}_3)_2 \cdot 4\text{H}_2\text{O}$] and palladium, cobalt bridging tetraacetate [$\text{Pd}^{\text{II}}\text{Co}^{\text{II}}(\text{OOCCH}_3)_4 \cdot \text{H}_2\text{O} \cdot 2\text{CH}_3\text{COOH}$] were used as catalyst precursors to prepare a 7.4 % mol catalyst/support loading. The oxidation states of these activated catalysts were studied by XPS. These catalysts were evaluated for hydrogenation of cinnamaldehyde in a biphasic reaction under 3 MPa of hydrogen at 80 °C. The $\text{Pd}^0/\gamma\text{-Al}_2\text{O}_3$ selectively catalyzed the hydrogenation of C=C bond in cinnamaldehyde to form phenylpropanol (100 %). In contrast, catalysis with $\text{Pd}^0\text{Co}^{\text{III}}/\gamma\text{-Al}_2\text{O}_3$ hydrogenated both C=C and C=O bonds consecutively, forming 65 % phenylpropanal and 35 % phenylpropanol with $\text{TOF} = 47.2 \text{ h}^{-1}$. No reaction occurred when $\text{Co}^{\text{III}}/\gamma\text{-Al}_2\text{O}_3$ was used.

KEYWORDS

Bimetallic heterogeneous catalyst, palladium, cobalt, hydrogenation, unsaturated aldehydes.

1. Introduction

The catalytic hydrogenation of α,β -unsaturated aldehydes is a useful transformation in organic synthesis to produce fine chemicals.¹ To selectively hydrogenate carbon–carbon double bonds (C=C) in molecules containing other functional groups like aldehydes is a very important process in the fragrance, flavouring and pharmaceutical industry.^{2–5} In α,β -unsaturated aldehydes the reduction of the carbonyl group (C=O) is in competition with the hydrogenation of the C=C bond, where the hydrogenation of the C=C bond is thermodynamically more favoured by about 35 kJ/mol.⁶ Consecutive hydrogenation can also occur first, leading to the formation of the saturated aldehyde followed by the formation of the saturated alcohol, or hydrogenation to form the unsaturated alcohol and then reduction to form the saturated alcohol. Depending on the reaction conditions and catalyst type, hydrogenation can be selective for a specific functional group or it can proceed in steps to form the fully hydrogenated product.

Cinnamaldehyde is one of the most popular substrates to test catalytic performance of a hydrogenation catalyst since two different unsaturated bonds (C=C and C=O) are present in the same molecule.^{7–10} The hydrogenation of cinnamaldehyde can proceed *via* two routes as shown in Fig. 1, depending on the catalyst and reaction conditions. The different products that can form are hydrocinnamaldehyde, cinnamyl alcohol and phenyl propanol.

Palladium is well known to be very active for C=C bond hydrogenation,^{11–13} and in the hydrogenation of unsaturated aldehydes it gives almost 100 % yield in saturated aldehyde.⁸ The selectivity of Pd towards the formation of saturated carbonyls as products, is explained by the preferred adsorption of the C=C bond on Pd.¹⁴

It has been shown that promotion of metallic catalysts with an ionic compound increase the hydrogenation rate of the

C=O bond of unsaturated aldehydes,¹⁵ due to the activation of the C=O bond by the ionic species.

This article reports on the preparation and characterization of an ionic Co(III)-promoted $\text{Pd}^0/\text{Al}_2\text{O}_3$ catalyst, using the organometallic single-source catalyst precursor [$\text{Pd}^{\text{II}}\text{Co}^{\text{II}}(\mu\text{-OOCCH}_3)_4 \cdot \text{H}_2\text{O} \cdot 2\text{CH}_3\text{COOH}$] (1). The catalytic activity for the hydrogenation of cinnamaldehyde to hydrocinnamaldehyde and phenyl propanol under mild conditions, i.e. 80 °C and 3 MPa H_2 were investigated. Comparison was made with the unpromoted $\text{Pd}^0/\text{Al}_2\text{O}_3$ catalyst and the promoted catalyst $\text{Pd}^0\text{Co}^{\text{III}}/\text{Al}_2\text{O}_3$ catalyst prepared by 1 and the physical mixing of the two mono-metallic acetate precursors, [$\text{Pd}_3(\text{OOCCH}_3)_6$] (2) and [$\text{Co}(\text{OOCCH}_3)_2 \cdot 4\text{H}_2\text{O}$] (3).

2. Experimental

2.1. Chemicals

Ethanol, palladium(II) chloride, cobalt(II) acetate tetrahydrate were purchased from Aldrich and used without any further purification. Catalyst support used was nano $\gamma\text{-Al}_2\text{O}_3$ with a surface area of $220 \text{ m}^2 \text{ g}^{-1}$. The attenuated total reflectance, ATR FTIR spectra were recorded on a Nexus FTIR equipped with a Nicolet Smart Golden Gate. XPS data were recorded on a Kratos AXIS Ultra spectrometer, equipped with a monochromatic $\text{Al K}\alpha$ X-ray source and a delay-line detector. Spectra were obtained using the aluminium anode ($\text{Al K}\alpha = 1486.6 \text{ eV}$) operating at 150 W, with the survey scans a constant pass energy of 160 eV was used and with the region scans a constant pass energy of 40 eV was used. The background pressure was $2 \times 10^{-9} \text{ mbar}$ and data was fitted on CasaXPS. Temperature programmed reduction (TPR) experiments was performed by measuring the H_2 consumption of an oxidized $\text{Pd}^{\text{IV}}\text{Co}^{\text{III}}/\text{Al}_2\text{O}_3$ catalyst as a function of temperature. The sample was heated at a rate of 5 K min⁻¹ to a temperature of 973 K in a 10 mL min⁻¹ H_2/N_2 gas flow (4 % H_2 in N_2). Tripalladium(II) hexaaacetate [$(\text{Pd}^{\text{II}}_3(\text{OOCCH}_3)_6)$, 2, was prepared according to published procedures.¹⁶

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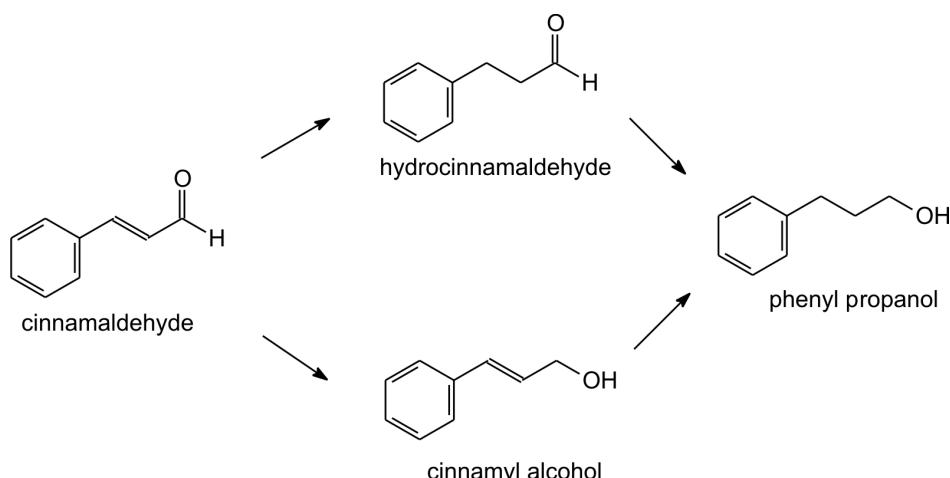


Figure 1 Hydrogenation of cinnamaldehyde.

2.2. Preparations

2.2.1. Palladium-Cobalt Bridging Tetracarboxylate Paddlewheel $[Pd^{II}Co^{II}(COOCH_3)_4] \cdot 2H_2O \cdot CH_3COOH$, 1

$[Pd^{II}Co^{II}(COOCH_3)_4] \cdot H_2O \cdot 2CH_3COOH$ was prepared according to published procedures.^{17,18}

A mixture of **2** (340 m, 0.5 mmol) and **3** (362 mg, 1.45 mmol) in 10 mL acetic acid was refluxed for 2 h. The mixture was left to cool slowly, overnight. Brown precipitate was filtered off and washed with acetic acid and water and dried. Yield 710 mg (1.43 mmol, 98 %). IR (cm^{-1}): 1699 (ν_{as} COO), 1610 ν_{as} COO), 1508 (ν_{as} COO), 1419 (ν_s COO), 1377 (ν_s COO), 1325 (ν_s COO). The crystal structure of this compound is reported elsewhere.¹⁷

2.2.2. Incipient Wet Impregnation of Nano γ -Al₂O₃ with **1**, **2** and **3**

1 (or **2** and/or **3**) (0.6 mmol) dissolved in acetone was slowly added to nano γ -Al₂O₃ (830 mg, 8.14 mmol), the mixture was stirred for 24 h and the solvent was removed under *vacuo* to yield a catalyst loading of 21–22 molecules nm⁻² or $3.6 (\pm 0.1) \times 10^{-5}$ mol m⁻² or 7.4 % mol catalyst mol⁻¹ solid support.

2.2.3. Oxidation of **1**, **2** and/or **3** on Nano γ -Al₂O₃

Oxidation of the freshly prepared **1** or **2** and/or **3**/nano γ -Al₂O₃ was achieved by placing the sample in an oven. It was then oxidized under O₂/Ar flow of 250 mL min⁻¹, while increasing the temperature from 25 °C to 200 °C at a rate of 10 °C min⁻¹, the sample was then kept at this temperature for 30 min before the temperature was further increased at a rate of 10 °C min⁻¹ to 500 °C. The sample was then kept at this temperature for 4 h before it was allowed to cool spontaneously overnight. Oxidation of sample prepared **1**/nano γ -Al₂O₃ produced Pd^{IV}Co^{III}/ γ -Al₂O₃, **4**.

2.2.4. In situ Reduction of the Pd

The oxidized catalyst was loaded into a 50 mL self-made reactor. After the reactor was flushed three times with H₂ and heated to a temperature of 80 °C, the H₂ pressure was increased to 3 MPa and the reduction of Pd with H₂ *in situ* was allowed to proceed for 2 h. *In situ* reduction of the Pd produced Pd⁰Co^{III}/ γ -Al₂O₃, **5**, (prepared by **1**), Pd⁰+Co^{III}/ γ -Al₂O₃, **6** (prepared by physically mixing **2** and **3** in the same solution), Pd⁰/ γ -Al₂O₃, **7** (prepared by **2**) and Co^{III}/ γ -Al₂O₃, **8** (prepared by **3**).

2.2.5. Hydrogenation of Cinnamaldehyde with Pd⁰Co^{III} or Pd⁰ or Co^{III}/ γ -Al₂O₃ Catalyst

In a typical experiment, after loading of the catalyst Pd^{IV}Co^{III} or

Pd^{IV} or Co^{III}/ γ -Al₂O₃ (30 mg) and *in situ* reduction of palladium (Pd⁰Co^{III} or Pd⁰/ γ -Al₂O₃), cinnamaldehyde (3 mL, 23.8 mmol) was added to the 50 mL self-made reactor. The reaction was allowed to proceed for 24 h at a temperature of 80 °C, and H₂ pressure of 3 MPa.

Turnover frequency was calculated by:

$$\text{TOF} = \frac{\left(\frac{\text{mol converted substrate} \times \% \text{ conversion}}{\text{mol of catalyst}} \right)}{\text{time}}$$

3. Results and Discussion

The heterobimetallic compound **1**, of the form [Pd^{II}Co^{II}(COOCH₃)₄]·H₂O·2CH₃COOH, was prepared by reacting **2** with the **3**, in glacial acetic acid to yield the desired product in almost quantitative yield (98 %). Water is an axial ligand, associated with the Co via hydrogen interaction. There are also two acetic acid molecules associated between two of the [Pd^{II}Co^{II}(COOCH₃)₄] complexes, which can be seen in the ATR FTIR of the compound (Fig. 2). Since this compound is known and its crystal structure was already determined,¹⁷ comparison of IR data confirmed that the correct product was obtained.

1, **2**, **3** and the physically mixed solution of **2** and **3** were deposited onto the γ -Al₂O₃ to produce mono- and bimetallic heterogeneous catalysts by means of wet impregnation followed by oxidation. XPS results of the oxidized samples revealed that Pd^{IV} ($3d_{5/2} = 337.7$ eV) and Co^{III} ($2p_{3/2} = 781.1$ eV) was present on the surface (see Fig. 3). These binding energies are within experimental error the same that was obtained for **1** impregnated by means of spin coating onto a Si(OH)/SiO₂/Si(100) two dimensional wafer.¹⁸

A temperature programmed reduced (TPR) study was performed on **4**, [Pd^{IV}Co^{III}/ γ -Al₂O₃] and Fig. 4 shows the rate of consumption of hydrogen on **4**. Three reduction peaks were observed, a sharp, well-defined peak at 82 °C and two large broad peaks at 121 °C and 542 °C. The TPR peak at 82 °C corresponds to the reduction of Pd, it is at a slightly higher temperature than reported values for Pd on alumina, 67 °C¹⁹ and 73 °C.²⁰ This change is possibly due to the presence of Co and the acetate ligands, which shields the Pd, making H₂ consumption more difficult. The flat broad peak at 121 °C is probably due to the consumption of H₂ by the organic ligand, the other larger broad peak at 542.9 °C is due to H₂ consumption on Co. Literature reports that Co on alumina shows two sharp, well-defined TPR peaks at 549 °C and 635 °C,²¹ which is in contrast to what was

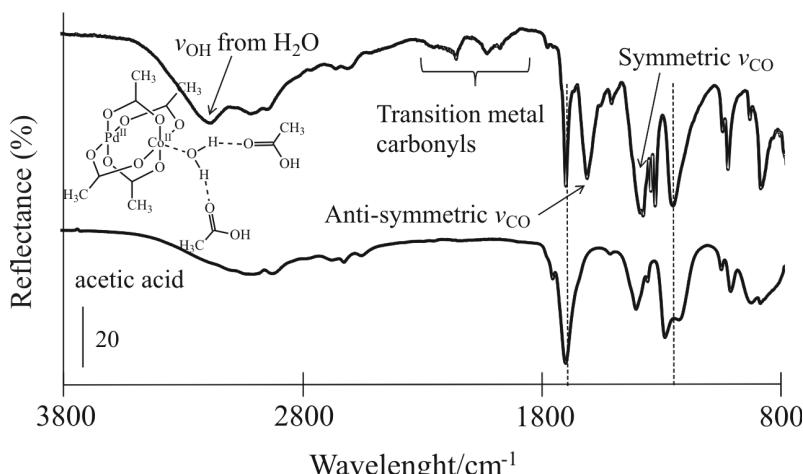


Figure 2 Infrared spectra of $[Pd^{II}-Co^{II}(OOCCH_3)_4] \cdot H_2O \cdot 2CH_3COOH$ (top) and acetic acid (bottom).

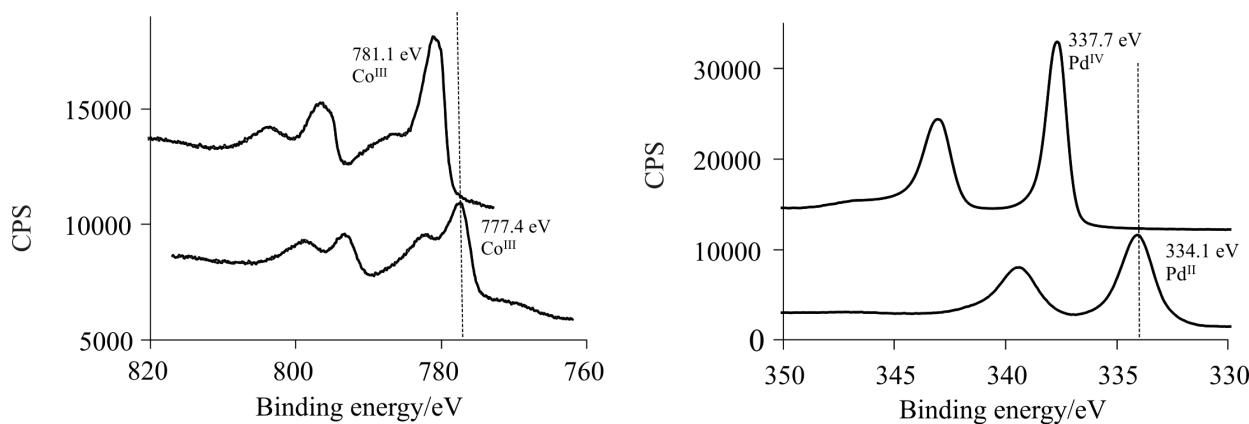


Figure 3 XPS spectra of cobalt (left), palladium (right) on γ -Al₂O₃, before (bottom) and after oxidation (top).

found for the Co in **4**. This could be indicative of Pd-promoted reduction of the Co as well as the broad shape of the peak shows the presence of highly dispersed Co-containing particles on the surface.²² This shows that the single source precursor approach yield good dispersion of the metals.

From the data obtained from the TPR, which shows that Pd in **4**, has a reduction maximum at 82 °C in a 10 mL min⁻¹ H₂/N₂ gas flow. It can be assumed that the Pd in **4** is fully reduced *in situ* at

80 °C and 3 MPa H₂, which implies that the active catalyst is Pd⁰Co^{III}/Al₂O₃, **5**.

In previous work **1** was used as a single source catalyst precursor to prepare the two dimensional model catalyst Pd^{IV}Co^{III}/Si(OH)/SiO₂/Si(100), which was found to work very well for aerobic oxidation of alcohols.¹⁸ The purpose of this study is to see if using **1** as a single source catalyst precursor could work as a dual catalyst. **5**, prepared from **1** and the physically mixed Pd⁰+Co^{III}/Al₂O₃, **6**, prepared from **2** and **3**, were applied as catalysts for hydrogenation of cinnamaldehyde, under solvent-free conditions at 3 MPa pressure of H₂ and temperature of 80 °C. Parallel control reactions catalyzed by Pd⁰/Al₂O₃, **7**, and Co^{III}/Al₂O₃, **8**, were also carried out.

For comparison, samples of the reduction reactions were taken after 24 h and were analyzed by means of ATR FTIR and H¹NMR, as an example the spectra obtained by **7** is shown in Fig. 5. On the ATR FTIR (Fig. 5, left), the disappearance of the CO stretching peak of the cinnamaldehyde (1668 cm⁻¹) and the appearance of the CO stretching of hydrocinnamaldehyde (1720 cm⁻¹) was followed. Conversion after 24 h (Table 1) was calculated by the ratio of the aldehyde peaks (see H¹ NMR in Fig. 5, right), cinnamaldehyde appears at 9.71 ppm as a doublet and hydrocinnamaldehyde appears at 9.83 ppm as a triplet.

The ATR FTIR graphs of all the tested catalysts after 24 h catalytic hydrogenation of cinnamaldehyde at 80 °C and 3 MPa H₂ pressure is shown in Fig. 6. It can be concluded that Co^{III}/Al₂O₃ is catalytically inactive towards hydrogenation since no conversion was found. After 24 h, **7** converted 82 % of cinnamaldehyde to hydrocinnamaldehyde and it was found that after 72 h there

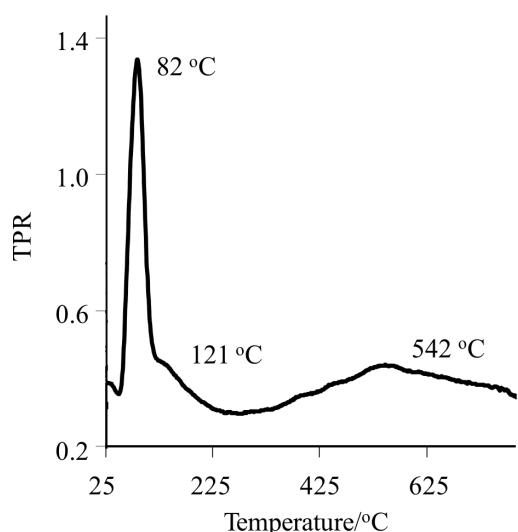


Figure 4 Temperature programmed reduction profile of the Pd^{IV}Co^{III}/ γ -Al₂O₃ catalyst.

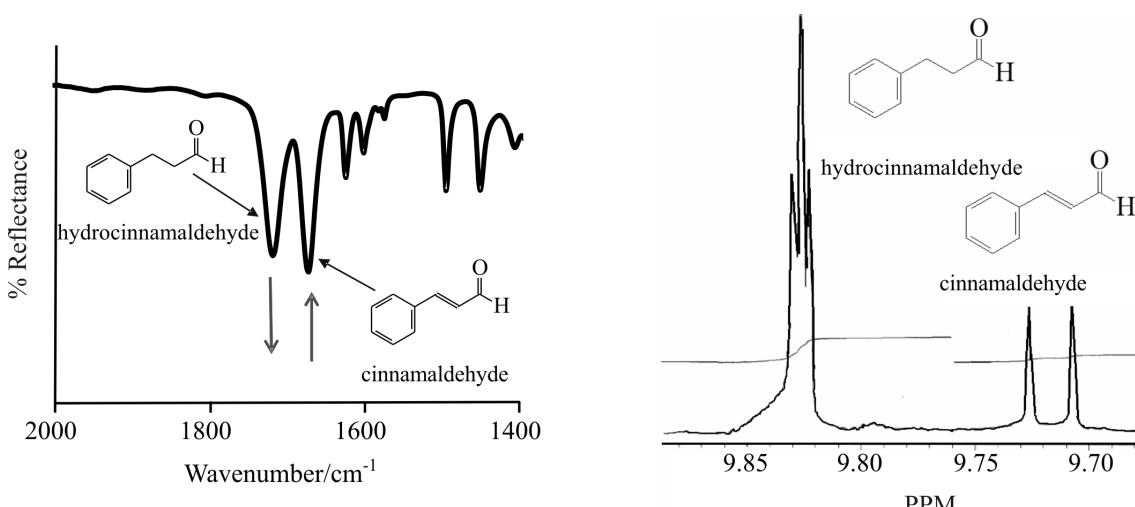


Figure 5 Partial ATR FTIR (left), showing the carbonyl region, and partial H^1 NMR (right), showing the region where the aldehyde peaks appear, of hydrogenation of cinnamaldehyde to hydrocinnamaldehyde after 24 h catalysis with 7.

was 100 % conversion, with no further conversion to phenylpropanol and a TOF = 38.7 h^{-1} . 6 showed a conversion of 58 % and a TOF = 27.5 h^{-1} after 24 h, which is considerably lower than was found for 7. This shows that the addition of Co^{III} to Pd^0 lowers its activity towards conversion of cinnamaldehyde to hydrocinnamaldehyde. This probably due to aggregation and segregation, with the Co^{III} only acting as a spectator and being positioned on the outside of the particle, thereby lowering the 'active' surface of the Pd, making the conversion slower. However, 5 showed that after 24 h no cinnamaldehyde was present with a TOF = 47.2 h^{-1} . A mixture of products was obtained, 35 % hydrocinnamaldehyde and 65 % phenylpropanol. Even after 72 h this ratio of hydrocinnamaldehyde to phenylpropanol did not change. This implies that after 24 h, the catalysis was complete or that the catalyst got poisoned. This result is in contrast to what was observed when a different support and physical mixture of Pt and Co is used, PtCo/ZrO results in 93.8 % conversion of cinnamaldehyde to cinnamylalcohol.²³ It was also shown that when Cu on silica support is promoted with Pd, the reaction is accelerated but the selectivity towards cinnamylalcohol is lost, similar to what we observed.²⁴

When a single source catalyst precursor is used, a more homogeneous dispersion of the two metals is obtained thus the addition of the Co^{III} to Pd^0 not only increase the conversion rate of cinnamaldehyde to hydrocinnamaldehyde but also facilitates the hydrogenation of hydrocinnamaldehyde to phenylpropanol. This confirms that the addition of an ionic species, in this case Co^{III} , to the metallic Pd catalyst activates the hydrogenation of

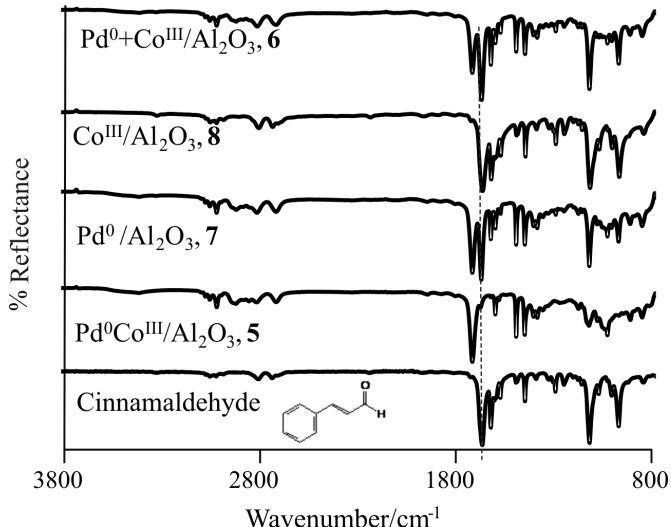


Figure 6 ATR-FTIR spectra of the hydrogenation of cinnamaldehyde after 24 h with different Al_2O_3 -supported catalysts.

the C=O bond, after hydrogenation of the C=C bond is complete.

Figure 7 shows the ATR FTIR graphs of the catalytic reaction with 5 at certain time intervals. It can be clearly seen how the CO stretching peak of the cinnamaldehyde (1668 cm^{-1}) disappears and the CO stretching hydrocinnamaldehyde (1720 cm^{-1}) appearance, which is accompanied by the formation of CH

Table 1 Results from the H^1 NMR data, showing the percentage conversion of the hydrogenation of cinnamaldehyde to hydrocinnamaldehyde and phenyl propanol with the different Al_2O_3 -supported catalysts.

Catalyst	% Conversion after 24 h catalysis		
Cinnamaldehyde			
$\text{Co}^{III}/\text{Al}_2\text{O}_3, 8$	100	0	0
$\text{Pd}^0/\text{Al}_2\text{O}_3, 7$	18	82	0
$\text{Pd}^0\text{Co}^{III}/\text{Al}_2\text{O}_3, 5$	0	35	65
$\text{Pd}^0+\text{Co}^{III}/\text{Al}_2\text{O}_3, 6$	42	58	0
		Hydrocinnamaldehyde	Phenyl propanol

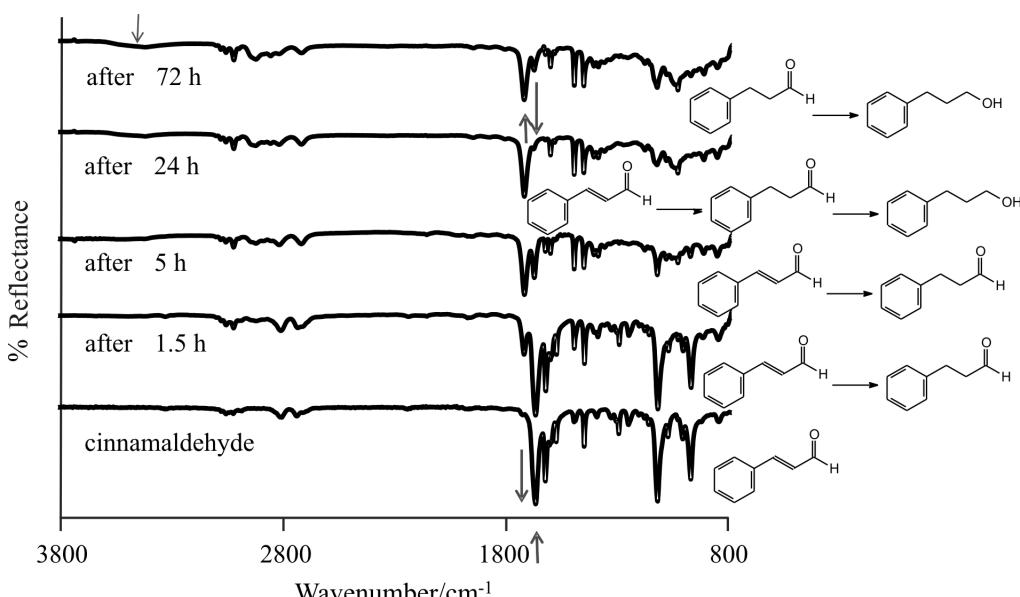


Figure 7 ATR-FTIR graph of hydrogenation of cinnamaldehyde with **5** at different time intervals.

stretching at 2919 cm^{-1} . In the last spectra after 72 h, the appearance of the OH stretching (3403 cm^{-1}) and the CO stretching peak (1676 cm^{-1}) of the phenylpropanol is observed.

Since catalyst **6**, which was prepared by physically mixing **2** and **3**, showed a decrease in catalytic hydrogenation of cinnamaldehyde and **5** catalyzed the reaction to 65 % phenylpropanol, it can be concluded that when using the organometallic precursor, **1**, a catalytic synergism is obtained due to a special interaction either between Pd^0 and Co^{III} or between the metals and the solid support.

Under the solvent free condition used for preliminary selective hydrogenation of the cinnamaldehyde to phenylpropanol, the catalyst needs regeneration by oxidation and *in situ* reduction of the Pd to reuse the catalyst.

4. Conclusion

The single source precursor $[\text{Pd}^{\text{II}}\text{Co}^{\text{II}}(\text{COOCH}_3)_4]\cdot\text{H}_2\text{O}\cdot2\text{CH}_3\text{COOH}$, **1**, was successfully used to impregnate nano $\gamma\text{-Al}_2\text{O}_3$ to form a bimetallic catalyst. The XPS of the oxidized catalyst showed that the metals oxidation states are Pd^{IV} and Co^{III} . TPR studies revealed that the Pd^{IV} get reduced *in situ* at $80\text{ }^\circ\text{C}$ and 3 MPa pressure of H_2 to yield an active catalyst with a form of $\text{Pd}^0\text{Co}^{\text{III}}/\text{Al}_2\text{O}_3$, **5**. The hydrogenation of cinnamaldehyde revealed that the catalyst obtained by physically mixing **2** and **3** to give $\text{Pd}^0+\text{Co}^{\text{III}}/\text{Al}_2\text{O}_3$, **6**, showed a lower conversion than the $\text{Pd}^0/\text{Al}_2\text{O}_3$, implying that the addition of Co^{III} to Pd^0 lowers its hydrogenation activity, most probably by lowering the active catalyst surface area. Catalyst **5**, prepared from the single source catalyst precursor **1**, revealed that hydrogenation of both $\text{C}=\text{C}$ and $\text{C}=\text{O}$ bond occurs and the product ratio obtained is 35 % hydrocinnamaldehyde and 65 % phenylpropanol.

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