

Review Article

Hydrogenation of Cinnamaldehyde by Aqueous Biphasic Ruthenium and Rhodium Catalysts: Mini-Review

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Abstract

Hydrogenation of aldehydes is a synthetically important reaction and has attracted much interest from the homogeneous catalysis community. The hydrogenation of cinnamaldehyde (*trans*-3-phenyl-2-propenal) supplies cinnamyl alcohol ((*E*)-3-phenylprop-2-en-1-ol), 3-phenyl-propanal and 3-phenyl-propanol. Selective hydrogenation of α,β -unsaturated aldehydes including catalyst recovery and product isolation are particularly challenging problems. Limited solubility of aldehydes in water allows the use of aqueous-organic biphasic systems with the use of water-soluble catalysts. Ruthenium and rhodium complexes have been used as aqueous biphasic catalysts precursor for the hydrogenation of cinnamaldehyde. This catalytic system favours benign solvents such as water due to being greener, inexpensive, non-toxic, non-flammable and environmentally sustainable. Rhodium and ruthenium complexes possessing carboxylic functional groups have been used for the hydrogenation of cinnamaldehyde due to their complete solubility property in the presence of additives such as KOH and triethylamine. Herein, this review article describes and explains the aqueous biphasic hydrogenation of cinnamaldehyde in the presence of rhodium and ruthenium catalysts and the mechanism involved therein.

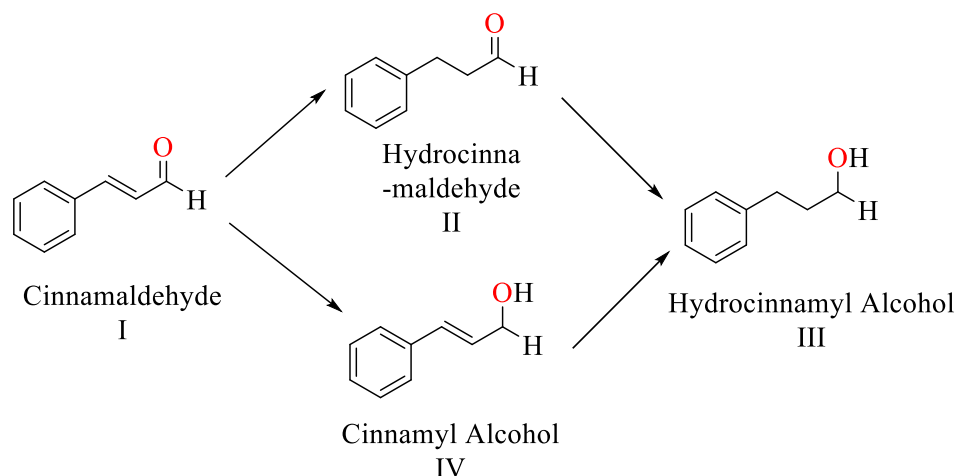
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Introduction

Selective hydrogenation of α,β -unsaturated carbonyl compounds, for example cinnamaldehyde into their corresponding unsaturated alcohol and saturated carbonyl compound is an important step in the industrial process for synthesis of fine chemicals, perfumes and pharmaceuticals[1-4]. Reduction of cinnamaldehyde can lead to the formation of three different products such as a saturated aldehyde (hydrocinnamaldehyde), an unsaturated alcohol (cinnamyl alcohol) and a saturated alcohol (hydrocinnamyl alcohol) (**scheme 1**)[5], [6]. It well established that the unsubstituted isolated C=C double bond undergoes easy hydrogenation than the isolated C=O aldehydic or ketonic group. Therefore, from thermodynamic point of view, the formation of saturated aldehyde and/or saturated alcohol is favored over the unsaturated alcohol. Hydrocinnamaldehyde has been used as a food additive in the flavouring industry and was recently claimed to be an important intermediate in the preparation of pharmaceuticals while hydrocinnamyl alcohol has been employed in the manufacture of perfumes[7-9].



Scheme 1 Products formed in hydrogenation of cinnamaldehyde

Limited solubility of aldehydes in water allows the use of aqueous–organic biphasic systems with the use of water-soluble catalysts. In such systems, the catalyst resides in the aqueous phase while the substrate is found in the organic phase so they can be separated upon completion of the reaction. Water soluble organometallic compounds of rhodium and ruthenium have been used as catalysts in various catalytic reactions such as hydrogenation and hydroformylation[10–13]. $[\text{RuCl}(\text{TPPMS})_2(\mu\text{-Cl})_2]$, $[\text{RuH}(\text{TPPMS})_2(\mu\text{-Cl})_2]$, $\text{OsH}_4(\text{TPPMS})_3$, $\text{OsHCl}(\text{CO})(\text{TPPMS})_2$ and $[\text{OsCl}(\text{TPPMS})_2(\mu\text{-Cl})_2]$ were investigated as catalyst for the hydrogenation of cinnamaldehyde under mild reaction conditions in aqueous biphasic systems; and the activity and selectivity of these catalysts were compared with those of homogeneous PPh_3 analogues [14]. It was reported that there is a clear advantage in using the aqueous biphasic mixtures over their analogous homogeneous solutions, since catalyst recovery and recycling are easy. Also, the regioselectivity towards the production of the α , β -unsaturated alcohol is considerably enhanced in the biphasic TPPMS and TPPTS systems, particularly in the case of Ru.

The selective hydrogenation of *trans*-cinnamaldehyde has been investigated with several types of multiphase reaction systems using Ru–phosphine complex catalysts[15] in the presence of high-pressure CO_2 . The selective formation of an unsaturated alcohol, cinnamyl alcohol, can be achieved in an organic solvent free two-phase system, which includes the liquid (*trans*-cinnamaldehyde, Ru complex) and gas (H_2 , CO_2) phases [15]. Total conversion and cinnamyl alcohol selectivity are enhanced with increasing CO_2 pressure. This enhancement is due to the dissolution of CO_2 molecules into the *trans*-cinnamaldehyde phase, which promotes the dissolution of H_2 and activates the reactivity of the carbonyl group of *trans*-cinnamaldehyde molecules, and a high concentration of *trans*-cinnamaldehyde in the reaction medium. The high cinnamyl alcohol selectivity can also be obtained in three- and two-phase reaction systems, which include a gas (H_2 , CO_2) phase, a liquid (water-dissolving Ru complex) phase, and/or another liquid phase (*trans*-cinnamaldehyde).

Water-soluble complexes $[\text{RuH}(\text{Ooc})(\text{mtppps})_3]$ ($\text{Ooc}=\text{octanoate}$, $\text{mtppps}=\text{monosulfonated triphenylphosphine}$) and $[\text{RuH}(\text{OAc})(\text{mtppps})_3]$ ($\text{OAc}=\text{acetate}$) were investigated for aqueous-organic biphasic hydrogenation of cinnamaldehyde as well as for hydrogenation of soybean liposomes under mild conditions [16]. The complexes showed pronounced selectivity (up to 75%) towards the hydrogenation of the $\text{C}=\text{O}$ function in the α,β -unsaturated aldehyde and the selectivity was influenced by the pH of the aqueous phase [17] and the hydrogen pressure. Phosphane-free porphyrin complexes with rhodium and ruthenium (**Figure 1**) were synthesized, characterized and used as catalysts for aqueous–organic biphasic hydrogenation of *trans*-cinnamaldehyde [18]. The catalysts were presumably homogeneous and could be recycled and reused without any significant difference in activity and selectivity. This is the first time that porphyrin–metal complexes have been used as catalysts for hydrogenation of α,β -unsaturated aldehydes.

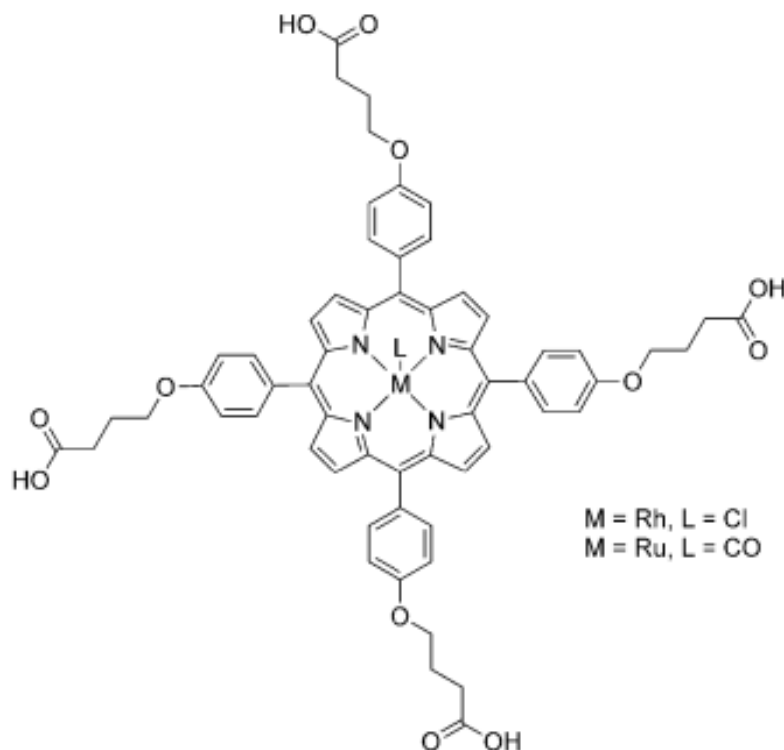


Figure 1 Phosphane -free porphyrin rhodium and ruthenium complexes as catalyst for aqueous organic biphasic hydrogenation of *trans*-cinnamaldehyde

The complexes $[\text{Rh}(\text{PhBP}_3)(\text{cod})]$ and $[\{\text{Ru}(\text{PhBP}_3)(\mu\text{-Cl})\}_2]$, containing the tripodal phosphanoborate ligand $[\text{PhB}(\text{CH}_2\text{PPh}_2)_3]^-$, were also found to be efficient catalysts for the selective hydrogenation of cinnamaldehyde to the corresponding allyl alcohol [19]. The ruthenium complex was reported to be the most efficient catalysts with TOF 527 h^{-1} and 97 % selectivity under mild reaction conditions (6.8 atm H_2 , 75 °C). The rhodium system also displayed good catalytic features in the hydrogenation of cinnamaldehyde (TOF 219 h^{-1}), particularly a high selectivity (81%) for this metal in the reduction of the C=O bond.

In the biphasic hydrogenation of cinnamaldehyde, the choice of the active metals is crucial to the selectivity to the desired product. The Ru-TPPTS catalyst is not only selective at C=O bonds but also at C=C bonds if certain parameters are controlled carefully. 96% selectivity to unsaturated alcohol and 97% selectivity to saturated aldehyde were achieved under optimum conditions by Nuithitikul and co-workers [20]. They also reported the biphasic hydrogenation by $\text{RhCl}(\text{TPPTS})_3$ catalyst and high selectivity towards C=C bond during cinnamaldehyde hydrogenation. In another study, hydrosoluble complexes $[\text{Rh}(\text{CO})(\text{Pz})(\text{L})_2]$ and $[\text{RuH}(\text{CO})(\text{CH}_3\text{CN})(\text{L})_3][\text{BF}_4]$ [$\text{L} = \text{TPPMS}$ (triphenylphosphinemonosulfonated) and TPPTS (triphenylphosphine-trisulfonated); $\text{Pz} = \text{Pz} = \text{pyrazolate}$] were evaluated in the selective hydrogenation of α,β -unsaturated aldehydes in biphasic media [21]. The Rh complexes were more active, required milder reaction conditions and preferentially reduced the C=C double bonds to form the corresponding saturated aldehydes; while the Ru ones, under harsher conditions, attacked the carbonyls to preferentially generate the unsaturated alcohols. Herein, this review discusses a number of important features in the hydrogenation of cinnamaldehyde by aqueous biphasic ruthenium and rhodium catalysts. These include the hydrogenation reaction conditions (the concentration of catalysts, mass transfer characteristics, the influence of pH and other catalytic additives) and the mechanistic studies for cinnamaldehyde hydrogenation.

Biphasic hydrogenation of cinnamaldehyde

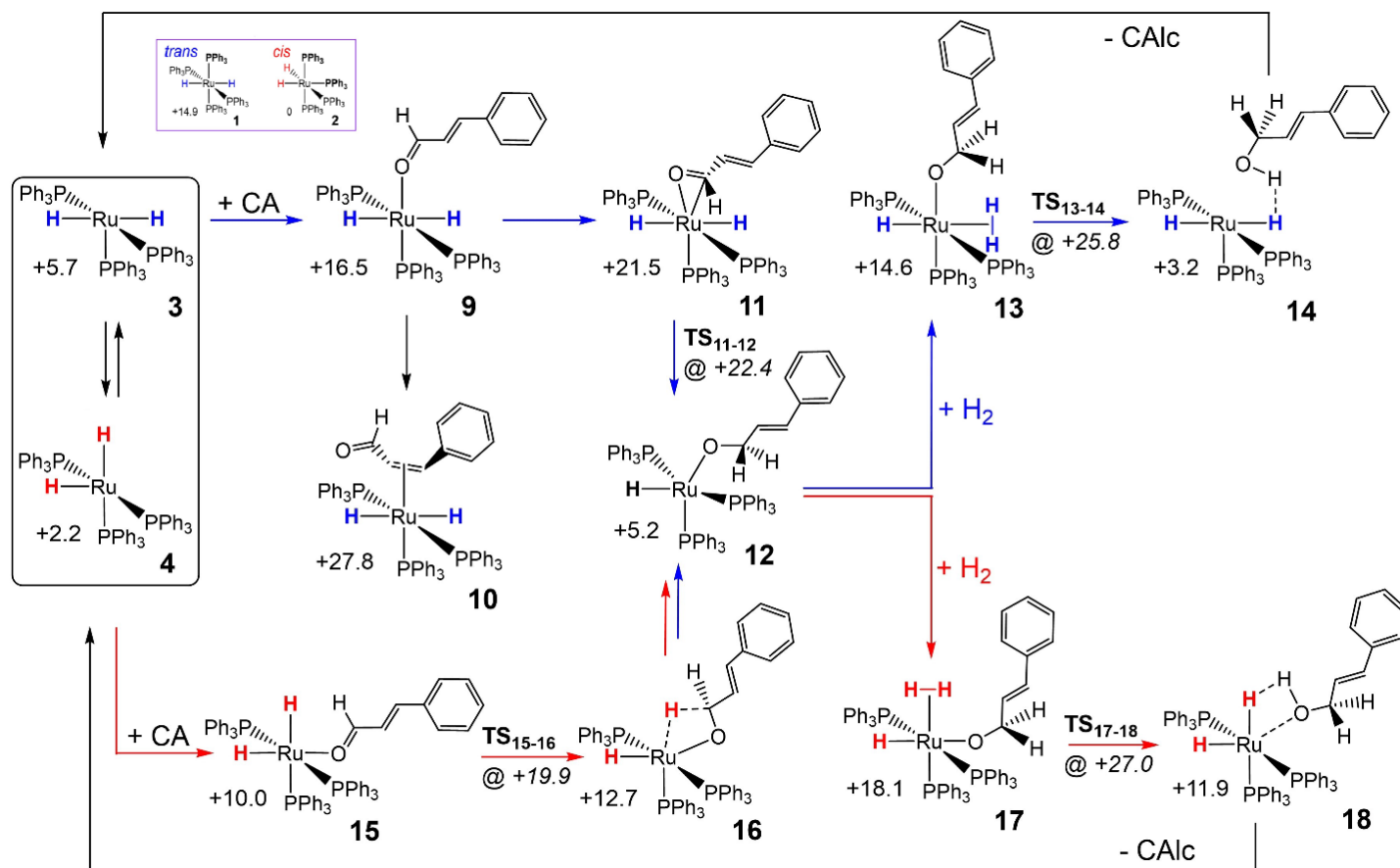
Water-soluble phosphine complex, $[\text{Rh}(\text{PTAH})(\text{PTA})_2\text{Cl}]\text{Cl}$, [PTA=1,3,5,-triaz-7-phosphaadamantane] was found to be an effective catalyst for the regioselective reduction of unsaturated aldehydes to saturated aldehydes [22]. The rate of hydrogenation of *trans*-cinnamaldehyde with sodium formate as reductant was studied with the same Rh(I) catalysts as a function of catalyst, substrate, and sodium formate concentration. The presence of an excess of PTA was found to inhibit hydrogenations completely and the reaction was also found to be partially inhibited by cyclo-octatetraene and Hg(0). Thus, leading to the conclusion that both heterogenous and homogenous mechanisms are operating in this system.

Unsaturated aldehydes were efficiently reduced by transfer hydrogenation from sodium formate in water–2-propanol mixtures using a water-soluble Ru(II)-tertiary phosphine catalyst by Szatmári and co-workers [23]. The reaction yielded unsaturated alcohols with complete selectivity and without hydrogenation or isomerization of C=C bonds of the substrates. Very high reaction rate was observed in the transfer hydrogenation of cinnamaldehyde already at 30 °C with turnover frequency of 160 h^{-1} and this increased to 3800 h^{-1} at 70 °C. Consequently, the method is applicable to the synthesis of unsaturated alcohols in case of heat sensitive or highly volatile aldehydes, too. A study by Joó and coworkers reported that the selectivity in the hydrogenation of cinnamaldehyde for reaction at C=C or C=O can be completely inverted by changing the pH value [24].

The mass transfer analysis of the biphasic system is quite complicated as there are many interfaces involved, especially when one of the reactants is gaseous (e.g. hydrogenation). The selective aqueous hydrogenation of cinnamaldehyde in the presence of $\text{RhCl}(\text{TPPTS})_3$ aqueous solution [TPPTS (tris(*M*-sulphonatophenyl)phosphine)] and NaOH solution to control pH control by NaOH solution was investigated under a biphasic (water/toluene) system in a batch reactor [25]. It was reported that the hydrogenation more usually occurred at C=C bonds, giving hydrocinnamaldehyde with selectivity of 99.9%. Both the kinetic and mass transfer aspects were also evaluated, and the results implied that the biphasic system was under mass transfer control. In addition, supported aqueous phase catalysts of the $\text{RhCl}(\text{TPPTS})_3$ solution supported on fine silica were prepared and tested for the selective hydrogenation of cinnamaldehyde under similar conditions for comparison and they gave a very high selectivity to hydrocinnamaldehyde which was an advantage, although their activity was lower than the biphasic catalysts. The mass transfer characteristic in supported aqueous phase catalysis was also evaluated using a first-order film model. The gas–liquid mass transfer is evidently affected by the presence of a second liquid phase. It has been reported that the liquid–liquid mass transfer rate of all reactants was assumed to be rapid compared with the gas–liquid mass transfer, therefore the liquid–liquid equilibrium is established [26], [27]. However, this assumption needs to be examined when a development of the biphasic system reaches a manufacturing scale, since the liquid–liquid mass transfer may be limiting in large scale reactions as may the gas–liquid mass transfer.

Mechanism for biphasic hydrogenation of cinnamaldehyde

The mechanism for the hydrogenation of cinnamaldehyde starts by investigating and establishing the resting state of the catalyst. Extensive studies using ruthenium and rhodium complexes as catalyst indicate that metal hydrides are the most likely catalyst for the cinnamaldehyde hydrogenation reaction. Metal hydride catalysts formation depend on the external H_2 pressure, pH of the reactants, nature of solvent used for the reaction and ligand truncation. The hydrogenation of cinnamaldehyde as model reaction was investigated to gain theoretical insight about whether the water soluble ruthenium trans-dihydride complexes, *trans*-[Ru(II)H₂P₃L] (P=PPh₃; monosulfonated PPh₃ (*mtp*ppms); L=H₂O or P), are, indeed, feasible catalytic species as suggested on the basis of experimental investigations [28]. According to Fehér and co-workers, *trans*-dihydrides provide two viable competing reaction channels with the key feature of the mechanisms being inclusion of explicit solvent (water) molecules, which allows the separation of substrate hydrogenation from the rate determining catalyst regeneration, where the H-H activation occurs [28]. It was found that the former determines the selectivity towards carbonyl hydrogenation through the more favorable hydride transfer to the carbonyl carbon. **Scheme 2** shows a *trans*-dihydride based associative pathway for the hydrogenation of cinnamaldehyde with the first step being cinnamaldehyde coordination through the C=O or the C=C bonds to yield intermediates at +16.5 kcalmol⁻¹ (**9**) and +27.8 kcalmol⁻¹ (**10**), respectively[28]. The associative mechanism reveals that the *cis*- and *trans*-[RuH₂P₃] complexes offer very similar energetics on pathways which involve cinnamaldehyde coordination followed by hydride migration, then proton transfer from a coordinated hydrogen or water molecule. Connecting these results to experimental factors implies that the removal of the alcohol product, thus separating the production and regeneration cycles or using phosphine in excess also shifts the system further towards C=O hydrogenation. To understand this catalyst even better, there is need for additional targeted experiments to be performed together with a theoretical study about the formation of stable *trans*-dihydride complexes because the calculations indicate that the existence of stable *trans*-dihydride species is not always required for them to be key catalytic intermediates or transition states.



Conclusion

Selective biphasic hydrogenation of cinnamaldehyde can be made successful by the nature of the metal. The metal precursor for the hydrogenation process can reduce cinnamaldehyde either to the corresponding cinnamyl alcohol or to the saturated hydrocinnamaldehyde. Biphasic water-soluble catalysts have been found to be effective for the regioselective reduction of unsaturated aldehydes to saturated aldehydes. The catalyst could be recycled by simple phase separation and reused without any significant change in activity and selectivity.

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