Kinetics of liquid-phase hydrogenation of \( n \)-valeraldehyde to \( n \)-amyl alcohol over a Ru/Al\(_2\)O\(_3\) catalyst

Prakash D. Vaidya, Vijaykumar V. Mahajani

Chemical Engineering Division, Institute of Chemical Technology, University of Mumbai, Matunga, Mumbai 400019, India

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Abstract

The kinetics of liquid-phase hydrogenation of \( n \)-valeraldehyde to \( n \)-amyl alcohol over a 5\% Ru/Al\(_2\)O\(_3\) catalyst was studied in the chemical control regime in the range of temperatures 323–348 K and pressures 0.69–2.76 MPa, using 2-propanol as the solvent. The selectivity to \( n \)-amyl alcohol was 100\%. The addition of water led to a marked increase in the rate of reaction. The catalyst could be reused thrice without any loss in activity. The initial rates increased linearly with the catalyst loading at 348 K in the range of catalyst concentrations 0.26–1.29 kg m\(^{-3}\). A simple power law model was used for analysis of the reaction kinetic data. A single-site Langmuir–Hinshelwood-type model suggesting dissociative adsorption of hydrogen and surface reaction as the rate-controlling step provided the best fit of the experimental data.

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Keywords: \( n \)-Valeraldehyde; \( n \)-Amyl alcohol; Catalysis; Kinetics; Mass transfer; Reaction engineering

1. Introduction

\( n \)-Butenes from C\(_4\) fraction present a cheaper sustainable source for specialty chemicals. \( n \)-Butenes can be hydroformylated to produce \( n \)-valeraldehyde, which on hydrogenation results in \( n \)-amyl alcohol. This route has excellent SHE (Safety, Health and Environment) compliances compared to the other synthetic route of chlorination of pentanes followed by alkaline hydrolysis of chloropentanes to produce \( n \)-amyl alcohol. Amyl alcohol is a good solvent for fats, oils and resins. Esters of amyl alcohol are used as perfumes and extractants.

The hydrogenation of \( n \)-valeraldehyde is carried out in the liquid phase using mostly Ni, Co or Mo sulfides supported on alumina as catalysts, in the range of temperatures 323–523 K and pressures 0.5–25 MPa. Earlier studies on heterogeneous catalytic liquid-phase hydrogenation of \( n \)-valeraldehyde are few and were carried out using either Raney Ni or noble metal-based catalysts such as Rh and Ru (Bonnier et al., 1987; Kaneda et al., 1992; Lau and Cheng, 1993). The Ru catalyst system (in a water-soluble form) is highly effective for the hydrogenation of organic carbonyl compounds in a biphasic medium (Lau and Cheng, 1993). Butene-1 can be easily obtained from C\(_4\) stream after removal of \( iso \)-butylene. Butene-1 can then be hydroformylated (reaction with CO and H\(_2\)) using a biphasic catalyst system containing a water-soluble catalyst, namely, Rh complex with trisodium salt of tri (\( m \)-sulfophenyl phosphine) catalyst (Kuntz, 1987). This system yields a very high normal/\( iso \) ratio. This means that the reaction mass consists of mainly \( n \)-valeraldehyde and \( iso \)-valeraldehyde as an impurity. There can be a small amount of water also if the phase separation after catalyst washing is not proper. Of course, the possibility of heavies at small ppm level cannot be ruled out. From a process intensification point of view, it would be a good idea to hydrogenate this stream straightaway by eliminating energy-intensive distillation to obtain pure \( n \)-valeraldehyde. With this view in mind, as a first step toward process development, it was thought desirable to study the
kinetics of hydrogenation of $n$-valeraldehyde to $n$-amyl alcohol over supported Ru catalyst. Our experience is that a heterogeneous Ru catalyst works better in the presence of water. It is also expected that Ru will enable hydrogenation to be carried out at milder reaction conditions than those with other catalysts. The catalyst based on Ru is expected to be cheaper than Pd and Pt as both are costlier than Ru. An in-depth study on catalytic hydrogenation of $n$-valeraldehyde to $n$-amyl alcohol over a Ru catalyst is not reported in the literature. It is expected that the results presented here would provide an insight into the reaction mechanism and aid process design and pilot plant studies for the production of valuable amyl alcohol. Some experiments in the presence of water are also reported. The scope of the present investigation excludes the effect of very small ($\leq 5\%$) quantities of iso-valeraldehyde and heavies formed during hydroformylation. These issues could be addressed separately.

2. Experimental

2.1. Materials

$n$-Valeraldehyde used in all experiments was of Fluka make and was purchased from a local vendor. Ruthenium trichloride trihydrate (pure), used as a metal precursor, was procured from SISCO Research Laboratories Pvt. Ltd., Mumbai. Alumina and titania supports having a high surface area were obtained from Degussa, Germany. Activated charcoal, also used as a support during catalyst preparation, was purchased from a local vendor. Sodium hydroxide, used as a precipitant in catalyst preparation, was purchased from a local vendor. Sodium hydroxide, used as a precipitant in catalyst preparation, was of analytical reagent grade and was purchased from S. D. Fine Chemicals, Mumbai, India. Hydrogen from a cylinder with a minimum stated purity of 99.9\% was obtained from Industrial Oxygen Company Ltd., India, and was used for experimentation as well as catalyst reduction. Analytical reagent grade 2-propanol was obtained from a local vendor and used as the reaction medium.

2.2. Catalyst preparation

Finely divided alumina support was contacted with an aqueous solution of ruthenium trichloride trihydrate ($\text{RuCl}_3$, $3\text{H}_2\text{O}$) in deionized water. The metal precursor was added in a predetermined amount to prepare a 5\% Ru/$\text{Al}_2\text{O}_3$ catalyst. The slurry was stirred for 6 h at 303 K. Sodium hydroxide, used as a precipitant, was then added. The slurry was then stirred again and heated to 333 K for 1 h. After settling, the top aqueous layer was separated from the precipitate below. Deionized water was added to this precipitate and the solution was allowed to digest at 353 K for 1 h. Excess of formaldehyde and an additional amount of sodium hydroxide were then added to this mixture to ensure that reduction was complete and heating at 353 K was continued for another hour. This was followed by cooling, filtration, washing and drying. A silver nitrate solution was used to ascertain the absence of Cl$^-$ ions in washings from the prepared catalyst. The 5\% Ru/TiO$_2$ and 5\% Ru/C catalysts were prepared in a similar way (with respective supports added in appropriate amounts) and used as such.

The alumina-supported Ru catalyst was pretreated by further drying at 473 K for 1 h in nitrogen flow in a temperature-controlled furnace to remove the residual moisture. Reduction was then carried out at 648 K at atmospheric pressure by replacing nitrogen with pure hydrogen. Smaller size catalyst particles were obtained by sieving through the appropriate size mesh. The particle size range used for these studies was 25–60 $\mu$m while the mean diameter was 46.6 $\mu$m. The particle size range was determined by using a particle size analyzer (Coulter LS 230). A typical scanning electron micrograph of this catalyst is depicted in Fig. 1 to have an idea of surface topography at a glance. It is seen that the catalyst particles are not spherical. The catalyst characterization was done by using a Micromeritics ASAP 2010 analyzer by nitrogen adsorption. The catalyst had a BET surface area of 598 m$^2$ g$^{-1}$, a pore volume of 0.17 cm$^3$ g$^{-1}$ and an average pore diameter of 10.1 nm.

2.3. Experimental setup

A schematic diagram of the experimental setup is shown in Fig. 2. All experiments were conducted in a Hastealloy C-276 Parr high-pressure reactor of capacity 0.1 dm$^3$. The reactor having a diameter of 50 mm (impeller diameter 35 mm) was equipped with an electrically heated jacket, a turbine agitator and a variable-speed magnetic drive ensuring zero leak. The temperature and the speed of agitation were controlled by means of a Parr 4842 controller. The gas inlet, gas release valve, cooling water feed line, pressure gauge and rupture disk were situated on top of the reaction vessel. The liquid sample line and the thermocouple well were immersed in the reaction mixture. A chilled water condenser was fitted on the sample valve exit line to avoid flashing of the sample. The entire assembly was leak-proof.
2.4. Experimental procedure

The reactor was first charged with 0.05 dm$^3$ of the reaction mixture and the catalyst. It was purged with nitrogen to ensure that the entire assembly was leak-proof under nitrogen pressure. The residual nitrogen left over near atmospheric pressure also ensured inert atmosphere. Then hydrogen was used to purge out nitrogen. All the lines were closed, the speed of agitation was adjusted to a pre-determined value, and the reaction temperature was set. The contents were heated to the desired temperature and a sample was withdrawn. This was considered ‘zero’ time for the reaction. The conversion during the time of achieving the desired temperature was very less (<2%) and therefore was neglected while calculating initial rates. Hydrogen from the cylinder was then sparged into the liquid phase directly beneath the impeller at the desired partial pressure of hydrogen. The amount of hydrogen charged was far in excess than that theoretically required. Samples were withdrawn periodically after sufficient flushing of the sample line. The fall in pressure was monitored by a pressure gauge. Additional hydrogen was charged from the cylinder through a manually operated control valve to make up for that consumed during the reaction, thus maintaining a constant total pressure (the entire system was in semi-batch mode). The reaction was allowed to proceed for a prescribed time after which the autoclave was allowed to cool. Samples of the remaining reaction mixture were analyzed thereafter.

2.5. Product analysis

Gas chromatography was used to analyze the reaction mixture. A GC 1000 unit (Chemito Instruments) equipped with a flame ionization detector and a 2.5 m long SE 30 column was used for this purpose. In all experiments, $n$-amyl alcohol was the only product formed during hydrogenation. The reproducibility of the results was checked and the error in all experimental measurements was less than 2%. A mass balance of the reactant consumed and the product formed showed an agreement to an extent of about 96% as per stoichiometry and this coincided well with GC analysis.

3. Results and discussion

In any catalytic process, the catalyst element and support interaction play an important role. It was, therefore, thought desirable to screen readily available catalysts with us, namely, Ru/Al$_2$O$_3$, Ru/TiO$_2$ and Ru/C for this hydrogenation reaction. We restricted our scope to metal support interaction by observing conversion as a function of time only. The catalyst morphological studies to support this interaction are excluded. As seen in Fig. 3, Ru/Al$_2$O$_3$ exhibited the highest activity and hence it was thought desirable to study the hydrogenation of valeraldehyde with Ru/Al$_2$O$_3$ catalyst.

3.1. Mass transfer limitations

The heterogeneously catalyzed hydrogenation of valeraldehyde is a three-phase (gas–liquid–solid catalyzed) reaction system, in which a number of steps occur in series. The solute gas hydrogen is diffused through the gas phase to the gas–liquid interface and then from the interface to the bulk liquid. Dissolved hydrogen and valeraldehyde present in the liquid phase are then transferred to the external surface of
The effect of speed of agitation on the rate of reaction was studied from the experimental rate data. All further experiments were conducted with two particle sizes in the range 25–60 μm. The extent of resistance offered to the liquid-phase mass transfer and the transfer of dissolved hydrogen and valeraldehyde in the liquid phase to the external surface of the catalytical particle depends on the intensity of turbulence in the liquid, which, in turn, is governed by the speed of agitation. The effect of speed of agitation on the rate of reaction was studied in the range 15–23 rps at 348 K, 2.76 MPa hydrogen partial pressure and a catalyst loading of 1.29 kmol m⁻³. The feed valeraldehyde concentration, viz. 0.3 kmol m⁻³, and the reaction time were the same in all experiments. It was observed that the rate of reaction was independent of the impeller speed in the range studied, thereby indicating the absence of these diffusional resistances. On the other hand, at 373 K, the speed of agitation had a pronounced effect on the initial rate of hydrogenation even at speeds as high as 20 rps.

The classical approach of reporting kinetic data in terms of the turnover frequency (TOF) defined as

\[
\text{TOF} = \frac{\text{no. of molecules reacted}}{\text{(active centers)} \times \text{time}}
\]

would have been the ideal choice. However, gaining knowledge of the active centers on the catalyst surface is a rather difficult task necessitating highly sophisticated techniques. Chemisorption of H₂ (or CO) would have given some idea. Our SEM analysis of the morphology of the surface showed that the catalyst particles are not spherical in shape but of irregular shape. It was, therefore, decided to express rates as kmol kg⁻¹ cat⁻¹ min⁻¹. After all, the active centers are proportional to the catalyst weight. From a process engineering point of view, in the absence of exact knowledge of active centers, this simpler approach would be more comprehensive.

The effect of catalyst loading on the initial rates was studied in the range of catalyst concentrations 0.26–1.29 kg m⁻³, at 348 K and 2.76 MPa hydrogen partial pressure. These results are shown in Fig. 4. The initial rates increased linearly with the catalyst loading in the range studied. The effects of various reaction parameters such as hydrogen partial pressure and feed valeraldehyde concentration on the initial rate were studied at 323, 333 and 348 K and are described below.

### 3.2. Reaction kinetic data

The rate of reaction was evaluated by fitting the valeraldehyde concentration vs. time plot to a polynomial followed by differentiation and calculation at various time intervals. The extent of resistance referred to the liquid-phase mass transfer and the transfer of dissolved hydrogen and valeraldehyde at the gas–liquid interface is instantaneously saturated with hydrogen as the gas phase is almost pure hydrogen (owing to the low vapor pressure of the solvent under reaction conditions). The gas–liquid interface is instantaneously saturated with hydrogen. The gas-phase mass transfer resistance can be neglected as the gas phase is almost pure hydrogen (owing to the low vapor pressure of the solvent under reaction conditions). The classical approach of reporting kinetic data in terms of turnover frequency (TOF) defined as

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#### 3.2.1. Effect of hydrogen partial pressure

The effect of hydrogen partial pressure on initial rates was studied in the range of hydrogen partial pressures 0.69–2.76 MPa at an initial valeraldehyde concentration of 0.3 kmol m⁻³. The results are shown in Fig. 5. These results indicated that the order with respect to hydrogen was far less than unity.

#### 3.2.2. Effect of initial valeraldehyde concentration

Experiments were conducted at varying feed valeraldehyde concentrations in the range 0.075–0.3 kmol m⁻³, at 2.76 MPa hydrogen partial pressure. The dependence of the initial rates on feed valeraldehyde concentration is presented in Fig. 6.

![Fig. 3. Effect of various catalysts on hydrogenation of n-valeraldehyde (323 K, H₂ pressure 1.38 MPa, initial n-valeraldehyde concentration 0.3 kmol m⁻³, catalyst loading 1.29 kg m⁻³) (□, 5% Ru/Al₂O₃, ■, 5% Ru/TiO₂, ●, 5% Ru/C).](image)

![Fig. 4. Effect of catalyst loading on initial rates of reaction (348 K, H₂ pressure 2.76 MPa, initial n-valeraldehyde concentration 0.3 kmol m⁻³).](image)
The solubility of hydrogen in 2-propanol at the given parameters represented as model to the experimental data. The rate of reaction was obtained from temperature (H$_2$ pressure 2.76 MPa, catalyst loading 1 kg m$^{-3}$) (at 323 K, 333 K, 348 K).

Fig. 6. Effect of initial n-valeraldehyde concentration on initial rates at various temperatures (H$_2$ pressure 2.76 MPa, catalyst loading 1.29 kg m$^{-3}$) (at 323 K, 333 K, 348 K).

3.3. Power law model

The reaction kinetics were modeled by fitting a power law model to the experimental data. The rate of reaction was represented as

$$ r = k[A]^m[B]^n, \quad (1) $$

where [A] = (H$_A$P$_A$). A and B denote hydrogen and valeraldehyde, respectively, and H$_A$ is the solubility parameter. The solubility of hydrogen in 2-propanol at the given partial pressure was used (Shaw, 1987). The values of $m$ and $n$ were found using regression analysis. The resulting rate expression was

$$ r = 69.9 \exp(-2587/T)[A]^{0.44}[B]^{0.46}, \quad (2) $$

where $T$ is in kelvin. The activation energy obtained from this expression was 21.5 kJ mol$^{-1}$. This indirectly confirms the absence of diffusional (mass transfer) resistances. The power law model is always useful for identifying controlling regime (mass transfer or kinetics) while designing a commercial reactor. Heterogeneous models based on reaction mechanisms were proposed for a further in-depth study on the observed reaction kinetics.

3.4. Langmuir–Hinshelwood kinetics

Langmuir–Hinshelwood-type models for single- and dual-site mechanisms were proposed. The estimated parameters for models proposing non-competitive adsorption of valeraldehyde and hydrogen were negative and hence rejected. Among all single-site mechanisms, rate expressions were derived for dissociative as well as non-dissociative adsorption of hydrogen. Statistical analysis of the experimental data for molecular adsorption of hydrogen also proved to be unsatisfactory because of negative model parameters and hence non-dissociative adsorption of hydrogen was ruled out. Further, models suggesting dissociative adsorption of hydrogen were derived for the case of adsorption of valeraldehyde controlling (model I), adsorption of hydrogen controlling (model II) and surface reaction controlling (model III). Only for these models (I, II and III), all parameters were positive. The residual sum of squares (RSS) and variance for these models at various temperatures are presented in Table 1. A comparison of the RSS or the variance indicates that model III based on single-site surface reaction-controlling mechanism gives the best fit to the experimental data as compared to models I and II. This model III could be represented as

$$ r = \frac{k_3 K_A K_B [A]^2 [B]}{[1 + (K_A[A])]^{1/2} + (K_B[B])^3}. \quad (3) $$

Experiments performed at 348 K showed that addition of the product n-amyl alcohol (10% w/w of n-valeraldehyde) to the reaction mixture in the beginning had no effect on the rate of reaction and hence the product term in the denominator in Eq. (3) was omitted. The kinetic data at various temperatures and valeraldehyde concentrations were correlated to this expression using MathCad. Table 2 shows the values of various parameters at different temperatures for this model. The parity plot giving both the initial rate data as well as the actual rate data at different reactant concentrations (during the course of reaction) is presented in Fig. 7. It is seen that the model validates well over the entire range. The activation energy for the surface reaction step was calculated from the temperature dependence of $k_3$ and was found to be 38.5 kJ mol$^{-1}$. The heats of adsorption of hydrogen and valeraldehyde were found from temperature dependence of $K_A$ and $K_B$ using the van’t Hoff equation. These values were 48.6 and 29.1 kJ mol$^{-1}$, respectively.

3.5. General considerations

n-Valeraldehyde prepared by low-pressure oxo synthesis might contain a small amount of water. It was, therefore, thought worthwhile to study the effect of addition of water on the rate of reaction in the presence of the supported Ru catalyst. Experiments carried out at 323 K showed a marked enhancement in the rates of reaction when 2-propanol containing water (10% w/w) was used as the reaction medium. These results depicted in Fig. 8 are of considerable significance in the production of amyl alcohol.

The catalyst was reused thrice at 348 K to see any poisoning of the same. The poisoning is expected to reduce active centers. The conversion vs. time data for different recycles of the catalyst showed no change in the rates and therefore,
Table 1
Residual sum of squares (RSS) and variance for the models proposed

<table>
<thead>
<tr>
<th>Temperature (K)</th>
<th>Model I</th>
<th>Model II</th>
<th>Model III</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>( k_1[B] )</td>
<td>( k_2[A] )</td>
<td>( k_3K_AK_B[A][B] )</td>
</tr>
<tr>
<td>RSS</td>
<td>Variance</td>
<td>RSS</td>
<td>Variance</td>
</tr>
<tr>
<td>323</td>
<td>9.0 ( \times 10^{-6} )</td>
<td>1.6 ( \times 10^{-5} )</td>
<td>5.0 ( \times 10^{-6} )</td>
</tr>
<tr>
<td>333</td>
<td>1.9 ( \times 10^{-5} )</td>
<td>3.2 ( \times 10^{-5} )</td>
<td>6.5 ( \times 10^{-6} )</td>
</tr>
<tr>
<td>348</td>
<td>2.4 ( \times 10^{-5} )</td>
<td>4.9 ( \times 10^{-5} )</td>
<td>8.5 ( \times 10^{-6} )</td>
</tr>
</tbody>
</table>

Table 2
Rate parameters with 95% confidence intervals for the L–H model III (Eq. (3))

<table>
<thead>
<tr>
<th>Temperature (K)</th>
<th>( k_3 )</th>
<th>( K_A )</th>
<th>( K_B )</th>
</tr>
</thead>
<tbody>
<tr>
<td>323</td>
<td>0.11 ( \pm 0.02 )</td>
<td>48.1 ( \pm 8.7 )</td>
<td>2.4 ( \pm 1.1 )</td>
</tr>
<tr>
<td>333</td>
<td>0.18 ( \pm 0.05 )</td>
<td>28.2 ( \pm 4.1 )</td>
<td>1.7 ( \pm 0.7 )</td>
</tr>
<tr>
<td>348</td>
<td>0.31 ( \pm 0.11 )</td>
<td>13.1 ( \pm 2.2 )</td>
<td>1.1 ( \pm 0.3 )</td>
</tr>
</tbody>
</table>

Fig. 7. Comparison of predicted rates (Eq. (3)) with experimental rates at various temperatures (feed n-valeraldehyde concentration 0.075–0.3 kmol m⁻³, 95% confidence intervals for slope 1.02 \( \pm 0.004 \)).

Fig. 8. Hydrogenation of n-valeraldehyde in 2-propanol containing water (10% w/w) (323 K, \( H_2 \) pressure 2.76 MPa, initial n-valeraldehyde concentration 0.3 kmol m⁻³, catalyst loading 1.29 kg m⁻³) (\( \square \), without water, \( \square \), with water).

we inferred that there was no change in the catalyst morphology and that the number of active centers remained the same even after three recycles. In other words, there was no loss of activity even after three recycles.

4. Conclusions

The kinetics of liquid-phase catalytic hydrogenation of n-valeraldehyde to n-amyl alcohol were studied in a stirred three-phase slurry reactor between 323 and 348 K using a hydrogen partial pressure range of 0.69–2.76 MPa, with a 5% Ru/Al₂O₃ catalyst. 2-Propanol was used as the reaction medium. All mass transfer resistances (gas–liquid, liquid–solid and intraparticle diffusion) were unimportant under the reaction conditions studied, thereby ensuring a kinetically controlled regime. The reaction kinetic data were modeled using the power law rate expression. The order of the reaction with respect to n-valeraldehyde was 0.46 and that of hydrogen was 0.44. A Langmuir–Hinshelwood model suggesting competitive adsorption of n-valeraldehyde and hydrogen was proposed for interpretation of the reaction kinetics. The catalyst activity remained constant even after three recycles, thereby confirming that the catalyst was highly stable under the conditions employed for hydrogenation. The rate of reaction was enhanced in the presence of water.

Notation

A hydrogen
B n-valeraldehyde
[A] concentration of hydrogen in the bulk liquid phase, kmol m⁻³
[B] concentration of n-valeraldehyde in the liquid phase, kmol m⁻³
\( H_A \) Henry’s constant for hydrogen, kmol m⁻³ MPa⁻¹
\( k \) reaction rate constant in Eq. (1), (m³)\( n+m \) kmol⁻¹ m⁻¹ kg cat⁻¹ min⁻¹
\( k_3 \) surface reaction rate constant in Eq. (3), kmol kg cat⁻¹ min⁻¹
\( K_A \) adsorption equilibrium constant for A, m³ kmol⁻¹
\( K_B \) adsorption equilibrium constant for B, m³ kmol⁻¹
\( K_P \) adsorption equilibrium constant for P, m³ kmol⁻¹
\( m \) order with respect to hydrogen
\( n \) order with respect to \( n \)-valeraldehyde

\( P \) \( n \)-amyl alcoh

\([P]\) concentration of \( n \)-amyl alcohol in the liquid phase, \( \text{kmol m}^{-3} \)

\( P_A \) partial pressure of hydrogen, MPa

\( r \) overall rate of reaction, \( \text{kmol kg cat.}^{-1} \text{min}^{-1} \)

\( T \) temperature, K

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**References**


