

Temperature-dependent Study of Isobutanol Decomposition

by Jonathan E. Mitchell and Ivan C. Lee

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Sensors and Electron Devices Directorate, ARL

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14. ABSTRACT The purpose of this work is to identify surface intermediates during the catalytic oxidation of isobutanol in a Fourier transform infrared (FTIR) reaction chamber at various temperatures and equivalence ratios (ϕ). This was achieved by collecting infrared spectra of surface species and locating peaks that would characterize the presence of these intermediates and products, such as carbon dioxide (CO₂), olefins, and aldehydes. The reaction was studied using γ-Al₂O₃, Rh/Al₂O₃, and Pd/Al₂O₃ powders as catalysts at temperatures from 100–500 °C over three equivalence ratios: 0.1718, 1.4248, and 3.501. By comparing the ratio of the intensity of the CH peak representative of isobutanol to the intensity of the C=O peak representative of aldehydes, it is possible to elucidate a trend in the activation of aldehyde formation from isobutanol. The results show that increasing temperature promotes aldehyde formation on the surface of each catalyst. In addition, it is shown that palladium (Pd) activates the formation of aldehydes and CO₂ at a lower temperature than a rhodium (Rh) catalyst.					
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1. Introduction

As our culture improves, the push to find alternative fuel sources to conventional petrol becomes increasingly more fervent. New legislations and pressure is being forced on the fuel industry to reduce America's dependence on fossil fuels for energy consumption. It has become more and more desirable to obtain a fuel that can not only meet safety and environmental standards, but provide more power for less than current fuel sources. One attractive alternative is isobutanol, which has been proven to burn cleaner and, with its higher energy density, more efficiently than ethanol (table 1). Isobutanol can be used as a gasoline blendstock or converts readily to isobutylene, a precursor to a variety of transportation fuel products. Isobutanol's flexibility makes it valuable to both the producer and the consumer (1).

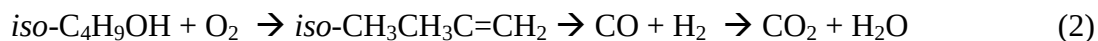
Table 1. Energy densities of various fuels.

Fuel	Energy Density (MJ/L)
Ethanol	19.6
Isobutanol	29.2
Gasoline	32

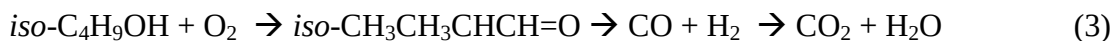
With such a promising alternative fuel available, it becomes important to study all aspects of its combustion process, including its reaction pathways and surface intermediates. When isobutanol combusts, it reacts as follows:



However, isobutanol does not always combust directly into carbon dioxide (CO₂). It often reacts by first taking on an intermediate pathway, either a dehydration pathway (equation 2) or a dehydrogenation pathway (equation 3). In the dehydration pathway, the isobutanol loses its hydroxyl bond entirely and becomes as an olefin (also known as an alkene), a carbon chain with a double bond.



In the dehydrogenation pathway, the isobutanol loses the hydrogen bond on the hydroxyl group and becomes an aldehyde.



These intermediates can also become side products if the fuel does not combust cleanly. One of the factors affecting this is the equivalence ratio of the stoichiometric amount of oxygen needed for the reaction to the actual amount of oxygen being fed into the reaction, or ϕ . Having a small ϕ gives a high O₂ input to the reaction, which tends to result in more complete combustion, while

medium or high ϕ result in partial oxidation or incomplete oxidation (total olefin production), respectively (2).

In order to best understand these reaction pathways and the factors that affect them, it is critical to observe them as they occur. Because these intermediates would not remain long enough to study in an outside environment due to their structural instability, reproducing the reaction in a reaction chamber contained inside an Fourier transform infrared (FTIR) spectrometer is ideal since it is capable of picking up the vibrations from the C=O stretch of the aldehyde or C=C stretch of the olefin should they be present. To facilitate this setup, it is necessary to use a catalyst powder as a surface on which the isobutanol can react. Because the partial oxidation of most alcohols is exothermic, it is essential to use high-surface area catalysts to increase, or if possible, maximize heat transfer. Alumina catalysts are useful and easily obtainable high-surface area catalysts that can be used in a number of ways. The best is to coat it with a metal surface to maximize their ability to transfer heat. A notable choice is rhodium (Rh), which has been proven to greatly increase the efficiency of combustion (3).

In this study, we examine the effects of temperature, catalysts, and equivalence ratios on the combustion of isobutanol. These factors are varied to find the reaction pathway followed by isobutanol under varied conditions, as well as determine how easily the fuel can come achieve total combustion.

2. Procedures

2.1 Materials and Reagents

Isobutanol was purchased from Sigma Aldrich with a purity exceeding 99%. All precursors used to make the catalyst powders were purchased through Sigma Aldrich. Spectra were taken with a Vertex 70 FTIR spectrometer from Bruker Optics and mass spectrometer. The measurements for the experiments were conducted at each temperature: 100, 200, 300, 400, and 500 °C.

2.2 Calibration of Isobutanol Flow

The flow of the isobutanol through the system was calibrated by weighing a condenser filled with 20 ml of isobutanol before passing argon through it at a rate of 200 sccm for 20 min. The condenser with isobutanol was then reweighed to see how many grams of isobutanol had flowed through the reactor per minute. With this data, the three ϕ values for the temperature-dependent study were then calculated.

2.3 Experimental Setup

As the reaction occurs and progresses inside the reaction chamber, the isobutanol oxidizes on the surface of the catalyst in the chamber, producing intermediates and products for the FTIR spectrometer to scan. This method has been used successful for a number of experiments for the

purposes of characterizing products for smoke testing and others (5–10). The gas products produced vent from the reaction chamber and are picked up by a mass spectrometer, which reads the weights of the molecules in the gasses and interprets as particular compounds. Together, this setup allows in situ characterization for isobutanol otherwise impossible for its partial oxidation.

2.4 Temperature Dependent Study

The constant-temperature reactions were carried out in a Praying Mantis reaction chamber, and the spectra taken with a Vertex 70 FTIR. The reaction chamber contained one of three different catalysts: γ -alumina (Al_2O_3), Rh/ Al_2O_3 , and palladium (Pd)/ Al_2O_3 . The pre-treated catalyst powder was added to the sample cup in the chamber until the cup was full. Once the powder was prepared in the chamber, the temperature control box was switched on and set to the desired temperature. Argon and oxygen were passed through the reaction chamber to take the background spectrum. After the background was taken, argon was allowed to bubble through the condenser filled with isobutanol at 200 sccm and pass into the reaction chamber, along with the pure argon and oxygen gasses. The reaction was allowed to run for 30 min before the FTIR ran a spectrum of the reaction. The mass spectrometer took samples of the exhaust gas constantly during the reaction. The reactions were run at 100, 200, 300, 400, and 500 °C consecutively for each of the three ϕ values (0.7187, 1.4248, and 3.501) with mass flow controllers settings described in table 2.

Table 2. Mass flow controller settings.

Phi	Argon (bubbled)	Argon	Oxygen
0.7187	200	71.8	28.2
1.4248	200	85.8	14.2
3.501	200	94.2	5.8

2.5 Temperature-programmed Desorption/Oxidation Study

The temperature-programmed desorption (TPD) and temperature-programmed oxidation (TPO) reactions were carried out in a Praying Mantis reaction chamber, and the spectra taken with a Vertex 70 FTIR. The reaction chamber contained one of three different catalysts: γ - Al_2O_3 , Rh/ Al_2O_3 , and Pd/ Al_2O_3 . The pre-treated catalyst powder was added to the sample cup in the chamber until the cup was full. Once the powder was prepared in the chamber, the temperature control box was switched on and set to 30 °C. Argon was bubbled through isobutanol at 200 sccm for 5 min to saturate the catalyst in the reaction chamber. The isobutanol line was then closed, and then argon for the TPD or oxygen for the TPO was allowed to flow through the chamber at 300 sccm. A background spectrum was then taken. The temperature control box was then turned on and set to ramp from 30 to 500 °C at 10 °C per minute. The FTIR was programmed to take spectra every 30 s until a total of 154 spectra had been taken over the course of approximately 90 min. The total 154 spectra were then collected and put into a three-dimensional (3-D) graphic to show the increase of peak intensity as temperature increased.

3. Results and Discussion

3.1 γ -Al₂O₃

Isobutanol was first run through the reaction chamber filled with γ -Al₂O₃ (figure 1). Spectra were analyzed for notable peak activity in the area of the CH band at 3000 cm⁻¹, representative of isobutanol presence, and C=O bands at 1700 cm⁻¹, representative of aldehyde presence. Once these peaks were located, the ratio of the intensities was compared to determine the relative amount of aldehyde being produced in the combustion. At $\phi = 0.7187$, the reaction showed little to no C=O peak activity at 100 °C, but suddenly increased at 200 °C. Both temperatures maintained a steady CH band, which is expected due to aldehydes possessing a CH bond like isobutanol. At 300 °C, the amount of C=O stayed roughly the same, but was accompanied by a new peak at 2300 cm⁻¹, representative of the presence of CO₂ in the environment of the reaction. This suggests that once this temperature was reached, the reaction obtained enough heat to move toward complete combustion of the isobutanol. Along with this increase in CO₂, there was a steady drop in the intensity of the CH peak of isobutanol. This trend continued over 400 and 500 °C. For the second ϕ , 1.4248, the reaction showed little to no C=O peak activity at 100 °C, but suddenly increased at 200 °C. At 300 °C, the amount of C=O stayed roughly the same, but was accompanied by a small amount of CO₂ as the CH peak decreased. This became more noticeable at 400 °C before making a dramatic spike at 500 °C. The third ϕ , 3.501, showed no notable C=O activity at 100 °C, but suddenly increased at 200 °C. At 300 °C, the amount of C=O stayed roughly the same. At 400 °C, small traces of CO₂ became visible, with a slight decrease in CH presence. This trend continued into 500 °C.

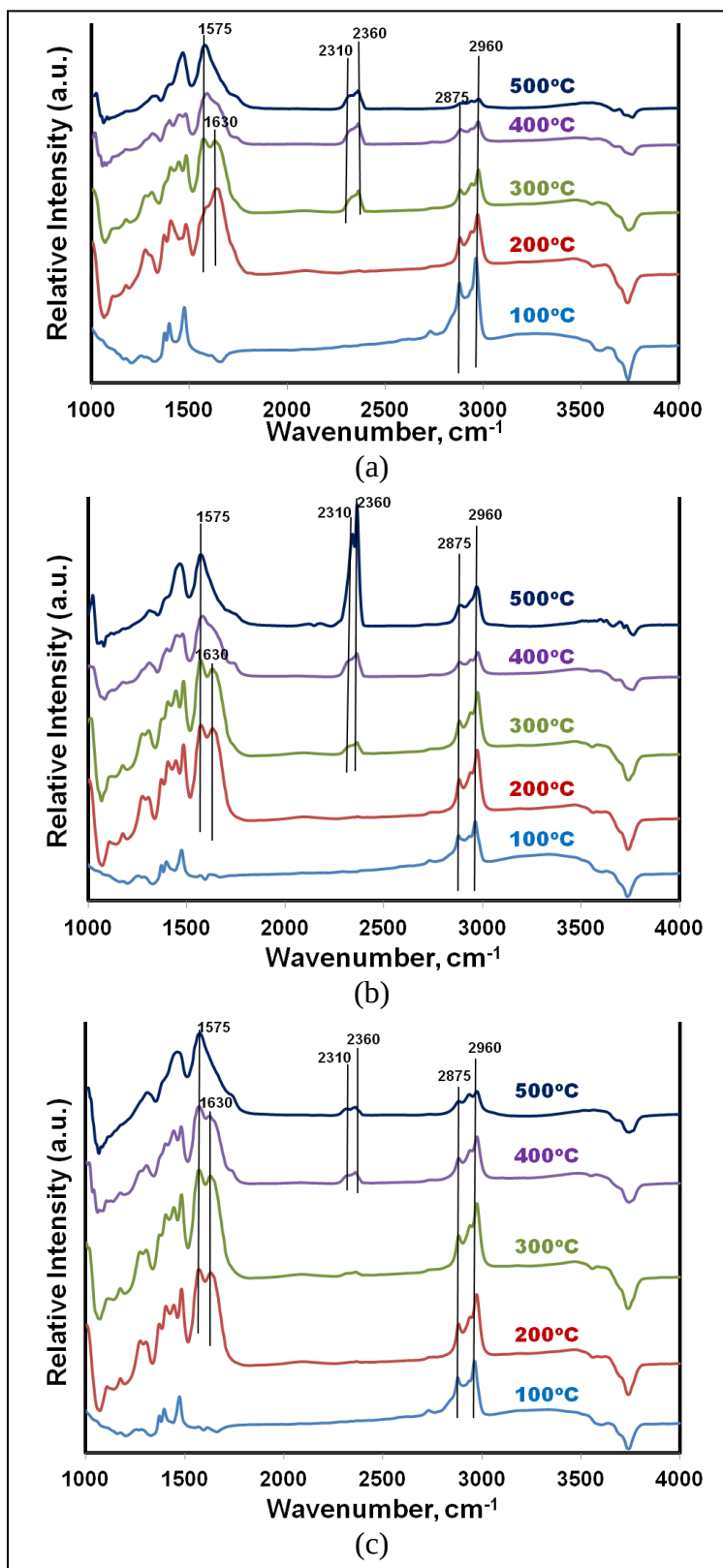


Figure 1. In-situ FTIR spectrum of isobutanol combustion on γ -Al₂O₃ catalyst from 100 to 500 °C at (a) $\phi = 0.719$, (b) $\phi = 1.425$, and (c) $\phi = 3.105$.

The TPD (figure 2) showed a steady linear increase in C=O activity matching a steady decrease in CH activity from the initial conditions as the temperature increased until 500 °C, at which point the C=O activity leveled off as the temperature remained steady. The TPO (figure 3) showed a much steeper, nonlinear increase in C=O activity before peaking at 450 °C, at which point the C=O activity dropped slightly before leveling off at 500 °C. In both reactions, a steady linear increase in free surface OH groups (3745cm^{-1}) accompanied these other activities before leveling off at 500 °C (11).

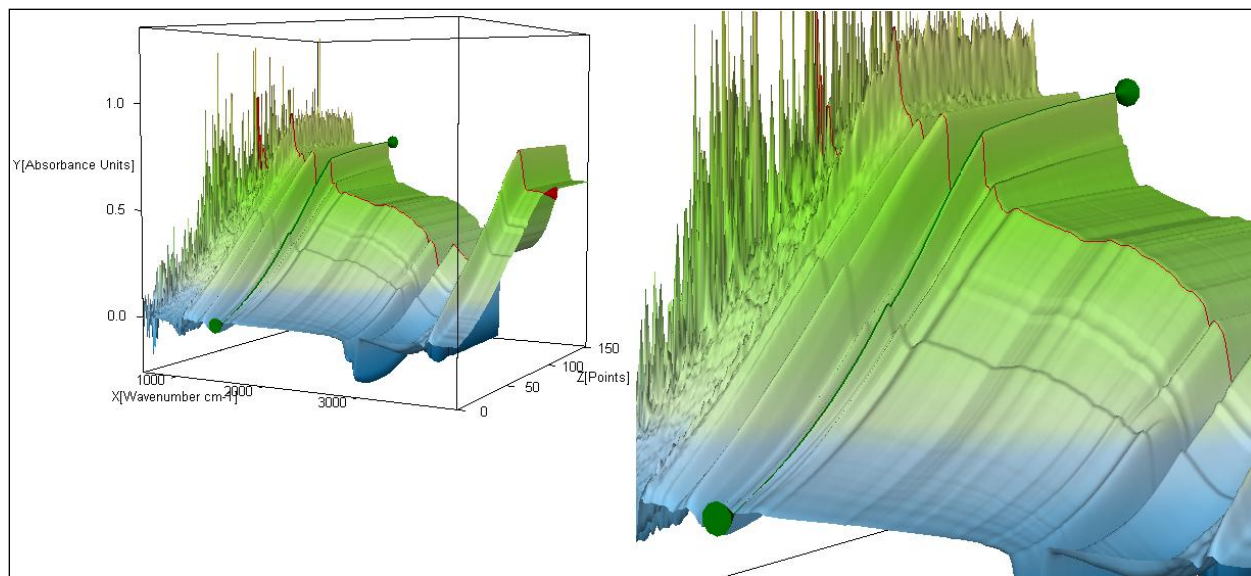


Figure 2. In-situ FTIR spectrum of the TPD of isobutanol on $\gamma\text{-Al}_2\text{O}_3$.

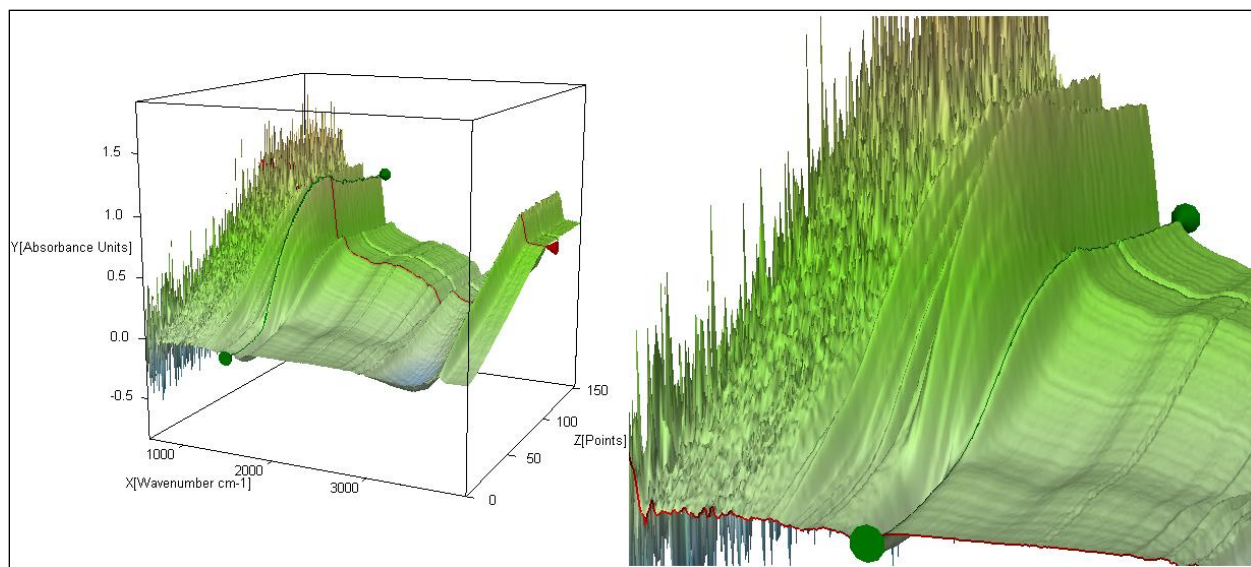


Figure 3. In-situ FTIR spectrum of the TPO of isobutanol on $\gamma\text{-Al}_2\text{O}_3$.

3.2 Rh/Al₂O₃

Isobutanol was next run through the reaction chamber filled with Rh/Al₂O₃ (figure 4). Spectra were analyzed for notable peak activity in the area of the CH band at 3000 cm⁻¹, representative of isobutanol presence, and C=O bands between 1600 and 1700 cm⁻¹, representative of aldehyde presence. Once these peaks were located, the ratio of the intensities was compared to determine the relative amount of aldehyde being produced in the combustion. At the first ϕ , 0.7187, the reaction showed slow increase in aldehyde production from 100 to 200 °C. At 300 °C, however, there was a sharp increase in aldehyde presence, accompanied by a notable decrease in the 3000 cm⁻¹ CH peak for isobutanol. While the isobutanol presence decreased significantly, a CO₂ peak appeared. The peak increased steadily at 400 and 500 °C. For the second ϕ , 1.4248, the reaction showed a similar slow progression of aldehyde activity until 400 °C, at which point the aldehyde and CO₂ peak intensities rapidly increased while the isobutanol peak dropped significantly. The activity then leveled off at 500 °C. The third ϕ , 3.501, showed a sudden increase in aldehyde activity at 200 °C, which slowly increased until 500 °C. From 300 to 500 °C, CO₂ slowly became visible as the isobutanol peak gradually decreased.

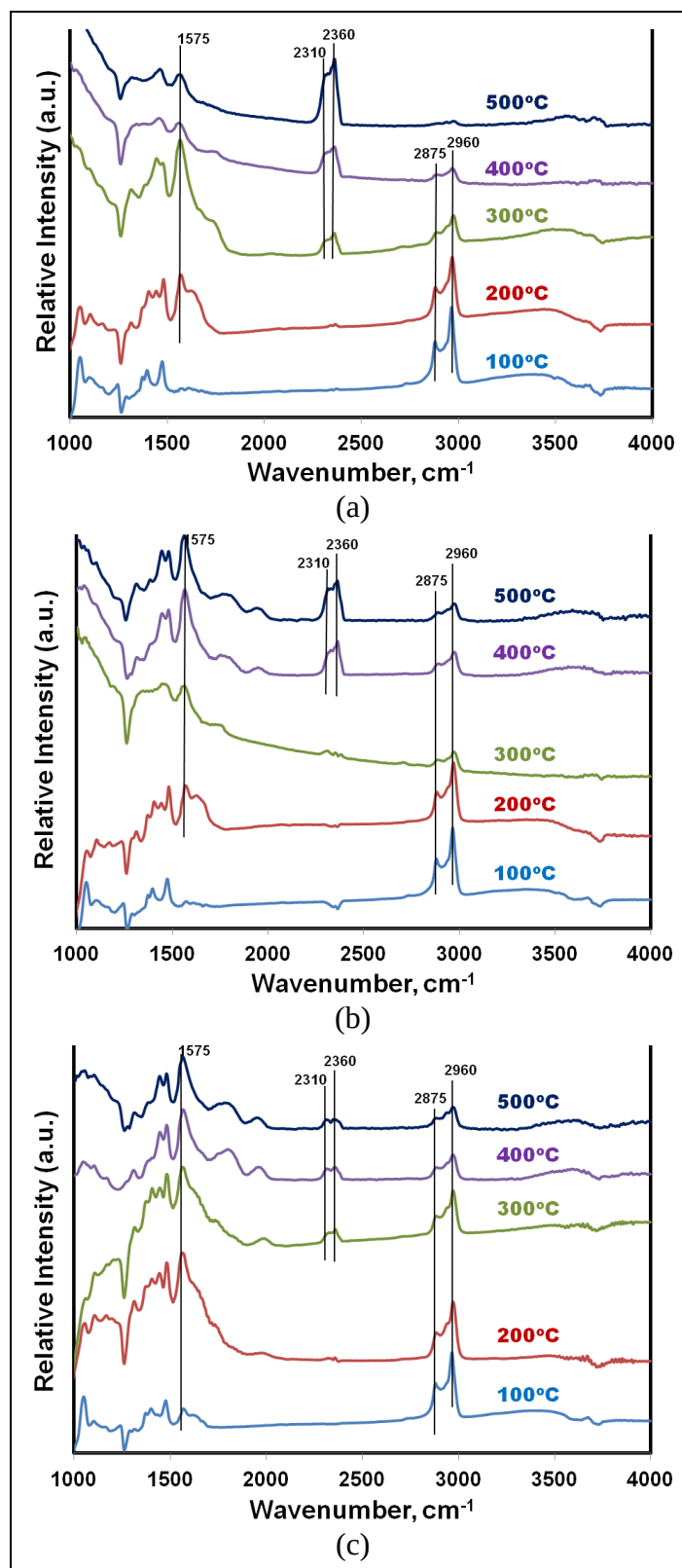


Figure 4. In-situ FTIR spectrum of isobutanol combustion on Rh/Al₂O₃ catalyst from 100 to 500 °C at (a) $\phi = 0.719$, (b) $\phi = 1.425$, and (c) $\phi = 3.105$.

The TPD (figure 5) showed a steady nonlinear increase in C=O activity matching a steady decrease in CH activity from the initial conditions as the temperature increased, but quickly lost any noticeable peak activity around 145 °C. Likewise, the TPO (figure 6) showed a much steeper, linear increase in C=O activity before quickly losing any notable peak activity around 255 °C. This suggests that the isobutanol desorbs from the surface quite rapidly as temperature increases, but takes more time to oxidize.

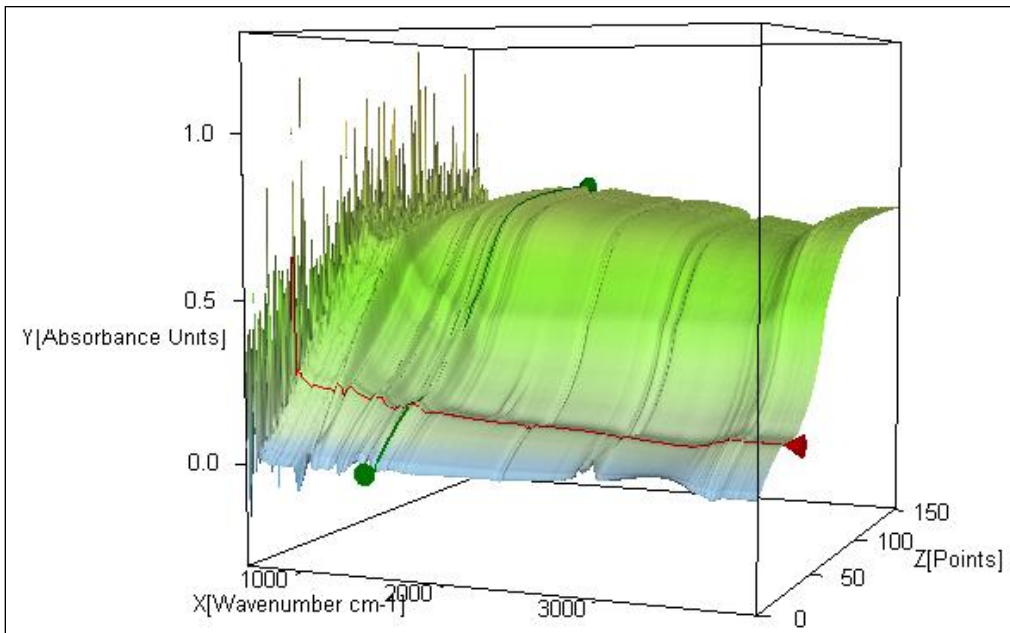


Figure 5. In-situ FTIR spectrum of the TPD of isobutanol on Rh/Al₂O₃.

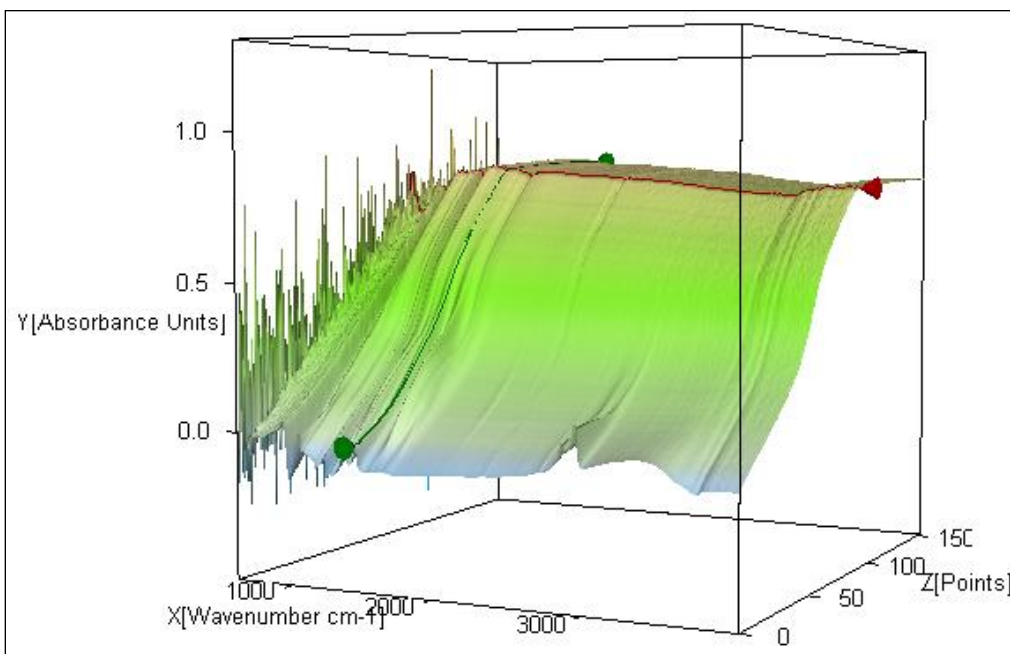


Figure 6. In-situ FTIR spectrum of the TPO of isobutanol on Rh/Al₂O₃.

3.3 Pd/Al₂O₃

Lastly, isobutanol was run through the reaction chamber filled with Pd/Al₂O₃ (figure 7). Spectra were analyzed for notable peak activity in the area of the CH band at 3000 cm⁻¹, representative of isobutanol presence, and C=O bands between 1600 and 1700 cm⁻¹, representative of aldehyde presence. Once these peaks were located, the ratio of the intensities was compared to determine the relative amount of aldehyde being produced in the combustion. At the first ϕ , 0.7187, the reaction showed a noticeable increase in aldehyde production starting at 100 °C. By 200 °C, CO₂ production was already visible at 2300 cm⁻¹, and the 3000 cm⁻¹ CH peak for isobutanol had already significantly decreased. The CO₂ production increased as the temperature rose, accompanied by a slow decrease in aldehyde production. For the second ϕ , 1.4248, the reaction showed a similar progression of aldehyde activity, with CO₂ production beginning at 200 °C and increasing until 500 °C, accompanied by a significant drop in isobutanol presence. The third ϕ , 3.501, the reaction showed a similar progression of aldehyde activity, with CO₂ production beginning at 200 °C and increasing until 500 °C, accompanied by a significant drop in isobutanol presence. However, the amount of CO₂ produced was notably less compared to the previous two equivalence ratios.

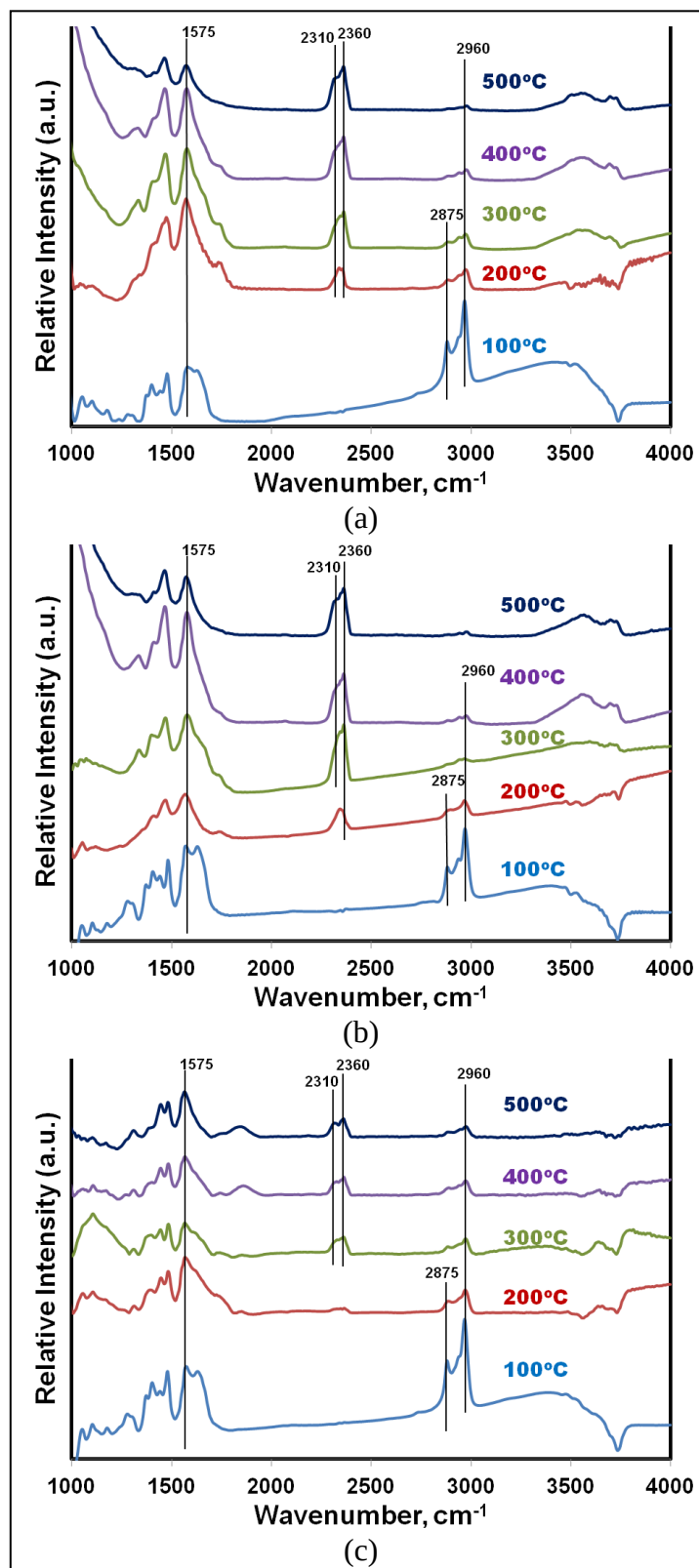


Figure 7. In-situ FTIR spectrum of isobutanol combustion on Pd/Al₂O₃ catalyst from 100 to 500 °C at (a) $\phi = 0.719$, (b) $\phi = 1.425$, and (c) $\phi = 3.105$.

The TPD (figure 8) showed a steady nonlinear increase in C=O activity matching a steady decrease in CH activity from the initial conditions as the temperature increased, but quickly lost any noticeable peak activity at 180 °C. Likewise, the TPO (figure 9) showed a much steeper, linear increase in C=O activity before quickly losing any notable peak activity at 280 °C. This suggests that the isobutanol desorbs from the surface fairly quickly as temperature increases, but takes more time to oxidize.

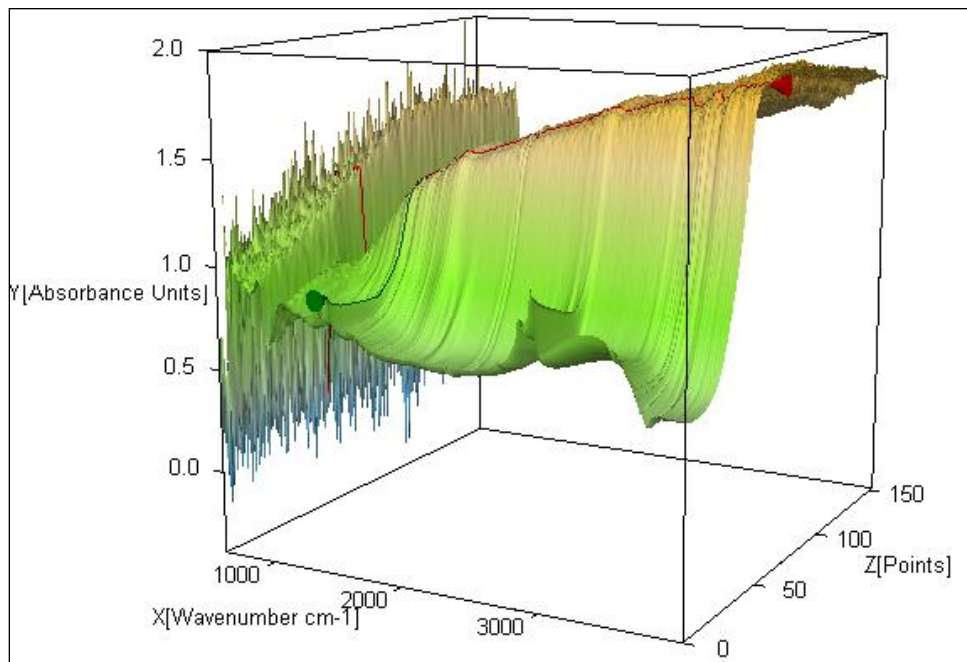


Figure 8. In-situ FTIR spectrum of the TPD of isobutanol on Pd/Al₂O₃.

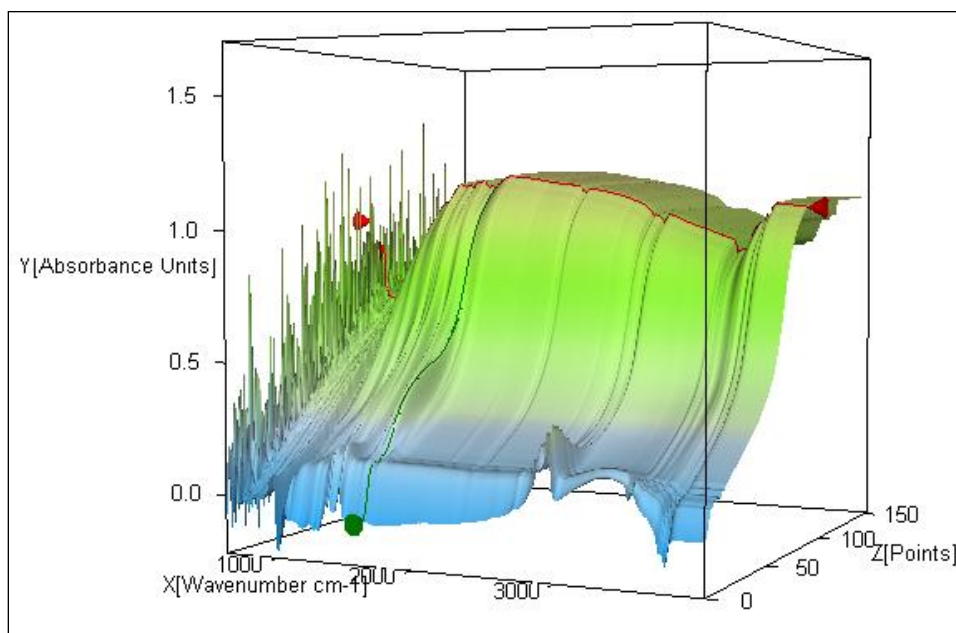


Figure 9. In-situ FTIR spectrum of the TPO of isobutanol on Pd/Al₂O₃.

3.4 Comparison of Catalyst Compositions

From the data obtained in these reactions, we can see an important relation between temperature, equivalence ratio, and catalyst identity. Since we know that combustion of isobutanol is an exothermic reaction, it becomes obvious that the higher the temperature of the reaction, the further toward total combustion it will come. From the catalysts, we can see that the higher surface area of γ -Al₂O₃ helps to promote the aldehyde intermediate, but is not sufficient for producing CO₂ at most equivalence ratios. However, an addition of transition metal on such a catalyst, such as Rh/Al₂O₃, will greatly increase the catalyst's ability to promote total combustion at higher temperatures. At different equivalence ratios, we can see that there is a significant contribution of dehydrogenation at $\phi = 3.501$ and, to a lesser degree, at $\phi = 1.425$. We can also see that smaller equivalence ratios, like $\phi = 0.719$, are more capable of producing higher concentrations of CO₂.

4. Conclusions

Overall, Pd/Al₂O₃ appears to activate at a lower temperature than either of the other catalysts to bring the reaction of isobutanol to total combustion. Rh/Al₂O₃, while effective, was still only partly successful at achieving complete combustion, while γ -Al₂O₃ was only able to produce surface intermediates until higher temperatures, at which point only trace amounts of CO₂ seemed to be produced.

With this information, we can more clearly understand the reaction mechanism of isobutanol during its combustion and how factors such as temperature, equivalence ratio, and catalyst type affect the pathways it takes and how completely it combusts. With this knowledge, we can work toward greater thermal efficiency for isobutanol combustion and further its availability as a valuable alternative fuel source.

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List of Symbols, Abbreviations, and Acronyms

3-D	three-dimensional
Al ₂ O ₃	alumina
CO ₂	carbon dioxide
FTIR	Fourier transform infrared
Pd	palladium
Rh	rhodium
TPD	temperature-programmed desorption
TPO	temperature-programmed oxidation

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