

Continuous Electrochemical Synthesis of 2-Octanone from 1-Octene

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Abstract

An electrochemical continuous flow system was created to replicate a synthesis of 2-octanone from 1-octene originally carried out in a batch process. Our results showed that the desired product was formed, and trends for how catalyst precursor concentration affects the reaction were observed, which will be important for future work.

Introduction and Background

Continuous electrochemical synthesis of desired products is important to industrial applications of organic reactions. Selectivity in electrochemical synthesis reactions and reliable product formation are easily demonstrated in batch processes but are less common in continuous flow processes. Formation of a ketone (2-octanone) from an alkene (1-octene) is one such important reaction.

Objective

The objective of this experiment was to produce 2-octanone from a 1-octene solution while minimizing unwanted side products using a continuous flow system.

Methods

- A 25 cm² cell was used with carbon flow field blocks, Teflon gaskets, catalyst-coated carbon cloth GDE's, and a Nafion™ 115 membrane.
- The cathode GDE had 1 mg cm⁻² Platinum catalyst and the anode had 0.1 mg cm⁻² Palladium catalyst.
- A 0.1M 1-octene solution was pumped through the anode side at a flow rate of 0.1 ml/min and air was flowed through the cathode side at a flow rate of 0.4 L/min.
- A power supply unit was used to apply a set current to the cell.

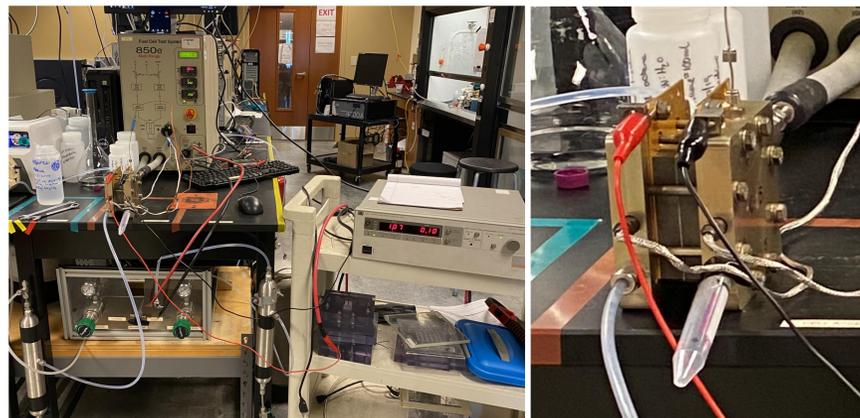


Figure 1: An overview of the whole apparatus, including power supply and fuel cell test station used for temperature control and cathode fuel. Figure 2: A close up of the cell itself ready for operation.

Results

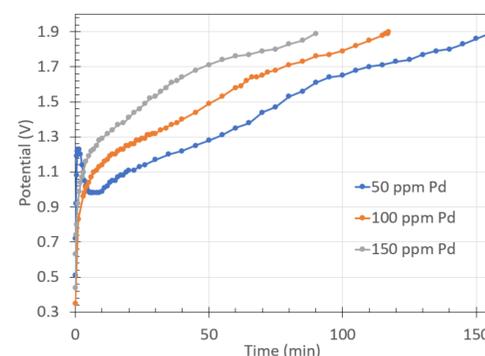


Figure 3: Potential over time during 100 mA runs for 3 catalyst precursor concentrations (50 ppm, blue; 100 ppm, orange; 150 ppm, gray). Pd loading was 0.1 mg cm⁻².

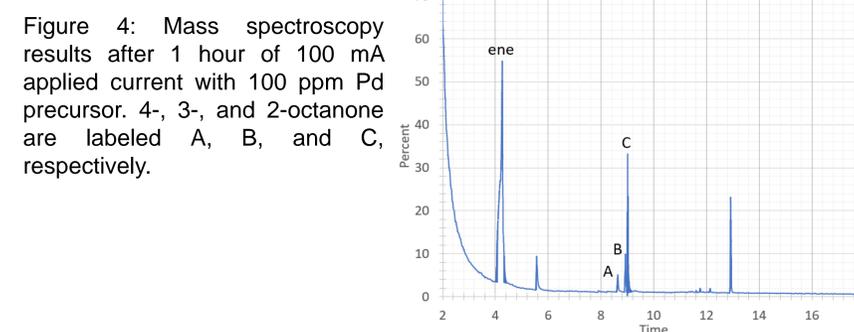


Figure 4: Mass spectroscopy results after 1 hour of 100 mA applied current with 100 ppm Pd precursor. 4-, 3-, and 2-octanone are labeled A, B, and C, respectively.

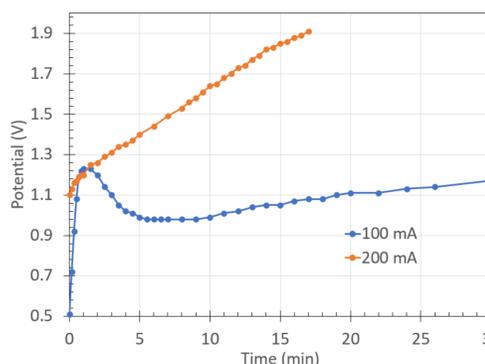


Figure 5: Comparison of the potential over time for 50 ppm catalyst runs at applied currents of 100 mA (blue line) and 200 mA (red line).

Discussion

- Trends for how voltage and catalyst ppm interact over time have been determined, as shown in Figure 3. In general, the voltage increases faster as the ppm increases, which means the reaction is happening quicker.
- The mass spectroscopy results (Figure 4) shows 2-octanone, the desired product, was formed, demonstrating the efficacy of the system.
- It was also observed that as we increased the applied current the reaction occurred much faster, as shown in Figure 5.

Conclusions

- We have demonstrated that the batch process can be translated into a continuous flow process.
- Reproducible trends for how the precursor catalyst affects the system have been observed.
- Single-pass conversion is adequate, and we observe numerous products, including 2-, 3-, and 4-octanone, octaldehyde, and an unidentified product with m/z = 171.

Future Plans

Moving forward, we want to be able to select the product created by manipulating the system parameters, demonstrate the complete conversion by multiple passes through the electrolyzer, and determine the stability and durability of the Pd catalyst over long run times.

Acknowledgements

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