

## Introduction

Fuel cells that rely on alternative fuel sources to hydrocarbons are promising clean and sustainable energy technologies. In particular, ethylene glycol (EG) has received considerable interest as an alternative fuel choice for a range of mobile, stationary and portable cells. The use of EG boasts promising energy capacities, high boiling points, and remarkable efficacy in electric power conversion.

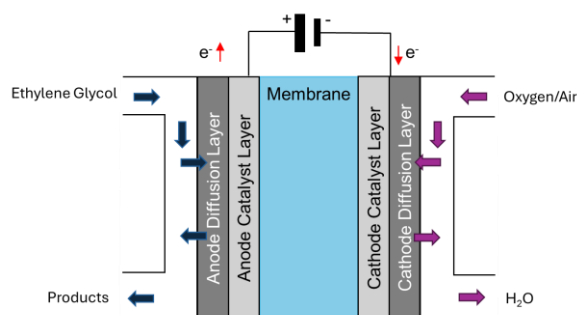


Figure 1. Ethylene glycol fuel cell schematic.

EG-based fuel cells are commonly analysed in situ using electrochemical measurements, but such techniques are limited by the lack of information acquired about reaction intermediates and by extension the reaction pathways. The inclusion of FTIR spectroscopy with electrochemical measurements allows for a simple, yet powerful method of providing real-time molecular characterisation of fuel cells. FTIR spectroscopy allows for a quantitative means of assessing the kinetics of specific molecular transformations, aiding simultaneously acquired electrochemical data.

Here, an Edinburgh Instruments IR5 FTIR Spectrometer coupled with an EC-ATR connected to an electrochemical workstation is used to characterise EG oxidation electrochemical reactions *in situ*.

## Experimental

The oxidation of EG in a 1M KOH solution was analysed using an IR5, equipped with a standard DLaTGS detector for general measurements. However, in-situ electrochemical reactions require higher sensitivity; thus, the second detector port was fitted with an MCT-A detector, offering enhanced sensitivity and faster response times crucial for tracking reaction intermediates. For the electrochemical reaction, the IR5 was coupled to an electrochemical workstation including three electrodes: nickel-plated carbon as the working electrode; Ag/AgCl as the reference electrode and Pt wire as the counter electrode.

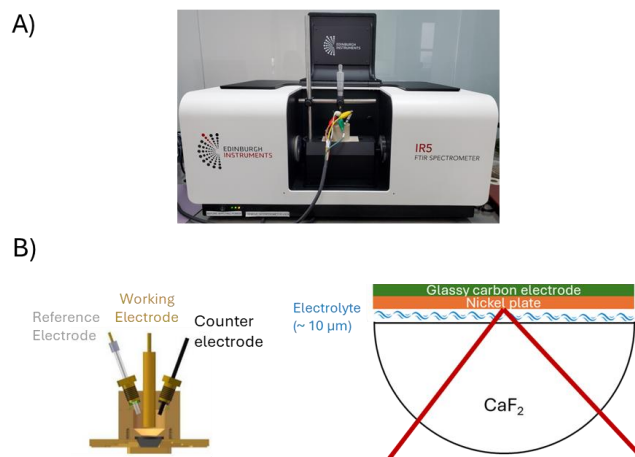


Figure 2. A) Experimental setup using an Edinburgh Instruments FTIR IR5 coupled with B) an EC-ATR. Ag/AgCl reference electrode (RE), nickel-plated carbon working electrode (WE) and Pt wire counter electrode (CE).

## Results and Discussion

In an EG-based fuel cell, oxidation takes place at the anode. The possible reaction pathways are shown in Figure 3.

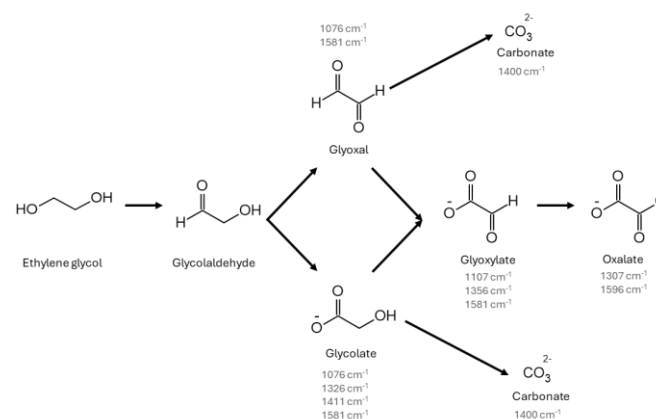
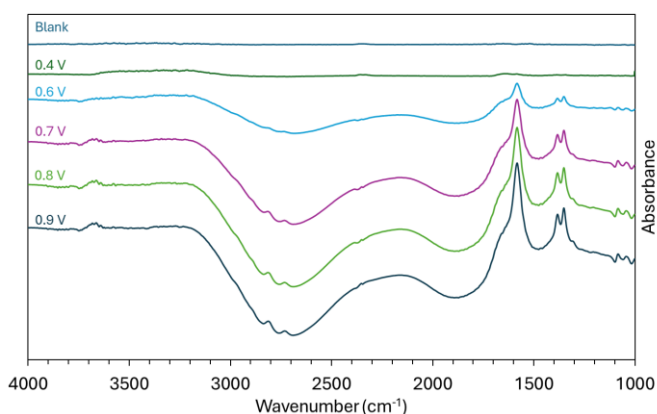


Figure 3. Ethylene glycol reaction pathway oxidation pathway.

Molecules	Asymmetric (COO <sup>-</sup> ) (cm <sup>-1</sup> )	Symmetric (COO <sup>-</sup> ) (cm <sup>-1</sup> )	Other bands (cm <sup>-1</sup> )
<b>Carbonate</b>			1390 - 1410
<b>Formate</b>	1581	1350 1381	
<b>Acetate</b>	1550	1411	
<b>Glyoxal</b>	1581		1076
<b>Glycolate</b>	1581	1326 1411	1076
<b>Glyoxylate</b>	1581	1365	1107
<b>Oxalate</b>	1596	1307	

**Table 1.** Ethylene glycol expected frequencies (cm<sup>-1</sup>) of various candidate intermediate species formed in electrooxidations.<sup>1</sup>

The reaction intermediates and products generated at the anode under operation can be detected using FTIR. In the electrochemical cell, the voltage was iteratively increased, and the composition of the EG/KOH solution was analysed. FTIR spectra acquired at each voltage are shown in Figure 4. A blank was recorded with no voltage, and subsequent samples with increased voltage were divided by this measurement to show the reaction products generated. Vibrational modes were more apparent on IR spectra after applying a voltage range from 0.4 - 0.9 V. Bands appear in the regions of 1000-1100 cm<sup>-1</sup> from C-O stretching, 1390-1410 cm<sup>-1</sup> from carbonate (CO<sub>3</sub><sup>2-</sup>), 1583 cm<sup>-1</sup> for the carboxylate (COO<sup>-</sup>) stretch and 3400–3700 cm<sup>-1</sup> from O-H stretch.



**Figure 4.** FTIR spectra of ethylene glycol in 1 M KOH at varying voltages.

## Conclusion

EG oxidation was successfully monitored using an IR5 configured with a highly sensitive MCT detector and electrochemistry setup. The use of FTIR characterisation coupled with electrochemical setups has great potential to provide invaluable information on intermediate products and kinetics of specific molecular transformations particularly useful in fuel cell development.

## References

1. Chang, S. C et al., Applications of Real-Time FTIR Spectroscopy to the Elucidation of Complex Electroorganic Pathways: Electrooxidation of Ethylene Glycol on Gold, Platinum, and Nickel in Alkaline Solution. *J. Am. Chem. Soc.* 1991, 113 (25), 9506–9513.