Master’s Thesis Engineering Technology

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Fabrication and Implementation of an Operando Gas Analysis Setup for Pouch Cell Batteries, and Its Application in the Characterization of Gas Evolution

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INTRODUCTION

The transition to renewable energy has increased the need for efficient storage solutions, with lithium-ion batteries playing a key role. However, their broad use introduces challenges, particularly **gas formation** during operation, which affects safety and performance, especially in **pouch cells (Figure 1)**.

Existing gas analysis techniques require manual extraction, resulting in inconsistent measurements, frequent leakages, and do not allow operando measurements. This master thesis aims to develop an automated setup that enables precise and reliable real-time gas analysis of pouch cell batteries.

The **goals** of this master thesis are:

- Developing an **automated, airtight setup** for real-time gas analysis.
- Enabling **operando** measurements without manual handling.
- Ensuring system reliability for studying battery degradation and safety.

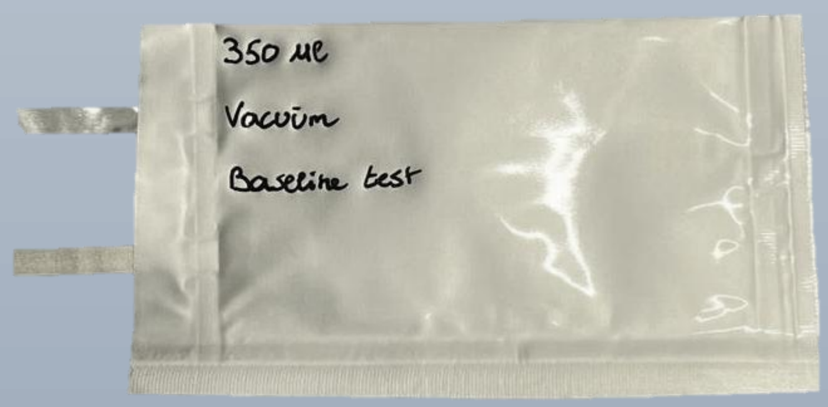

Figure 1: Pouch Cell

DESIGN

A custom, airtight test cell was developed to enable real-time gas extraction and analysis from lithium-ion pouch cells during cycling. The system enables operando gas measurements while ensuring airtightness and reliability. Figure 2 shows the setup configuration.

1. Airtight Battery Enclosure
An airtight mechanical enclosure was designed to contain the pouch cell. One corner of the pouch is opened to allow gas release, while the rest remains sealed preventing leakage of electrolyte. The enclosure directs all evolved gases through a controlled outlet, using gas-tight fittings.

2. Automated Gas Flow Control
A gas line is built using chemically resistant tubing and implements solenoid valves to control gas flow automatically. These valves enable precise start and stop control of the sampling process, allowing for reproducible and automated operando analysis.

3. Electronics and Valve Control
The solenoid valves operate on 24V DC, supplied by a custom-built circuit. An Arduino Nano controls the valve switching through programmed signals, enabling integration with battery cycling routines and removing the need for manual operation.

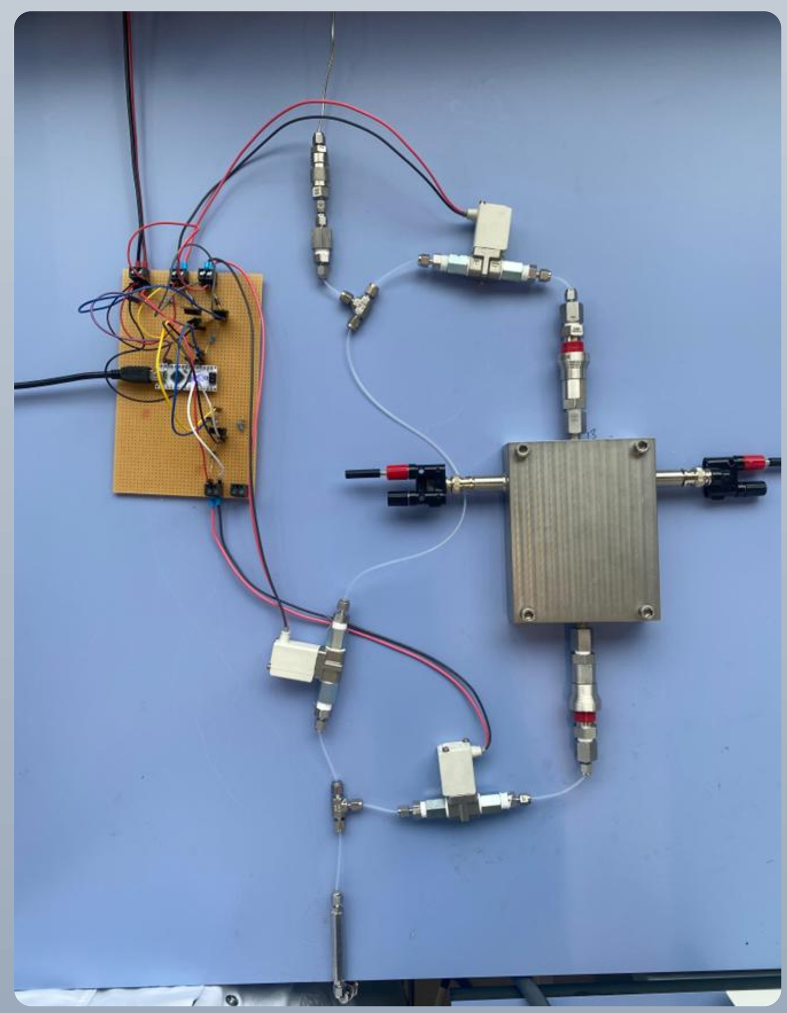

Figure 2: Gas Analysis Setup for Pouch Cell Batteries

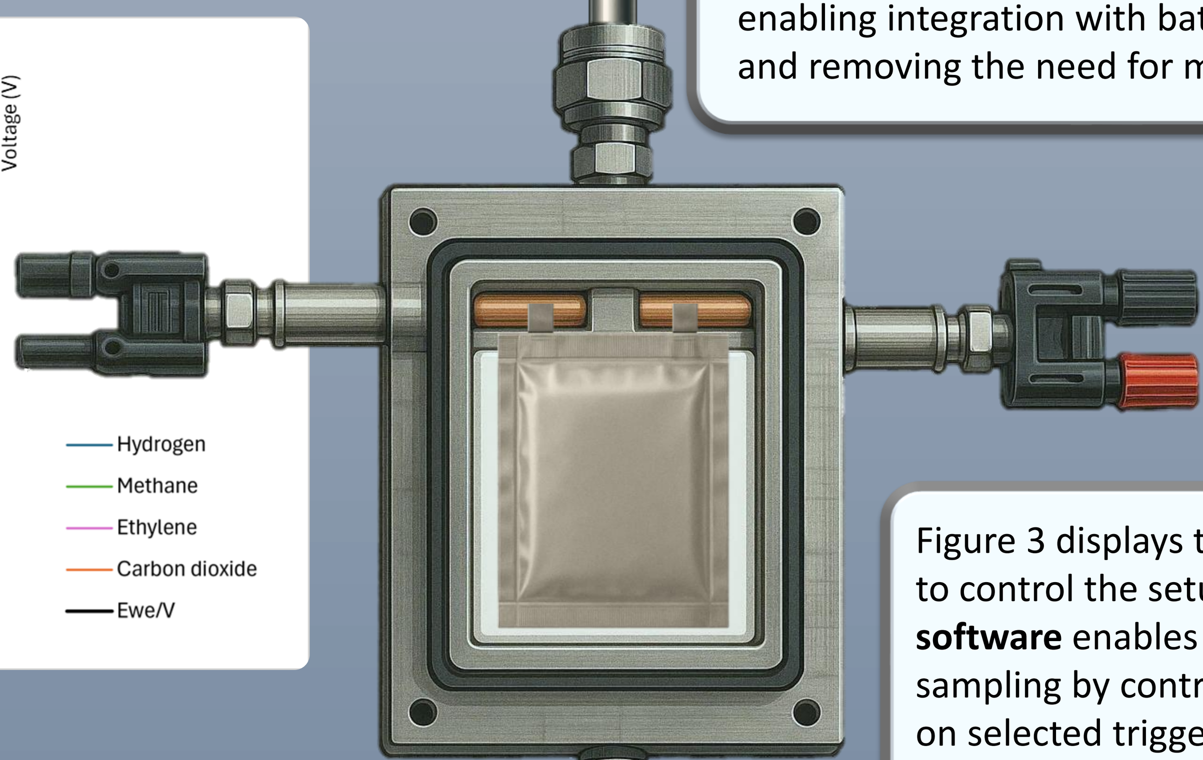

Figure 3 displays the software interface to control the setup. This **custom-made software** enables fully automated gas sampling by controlling each line based on selected trigger types, such as time, voltage, or cycling events, ensuring precise and flexible operando analysis.

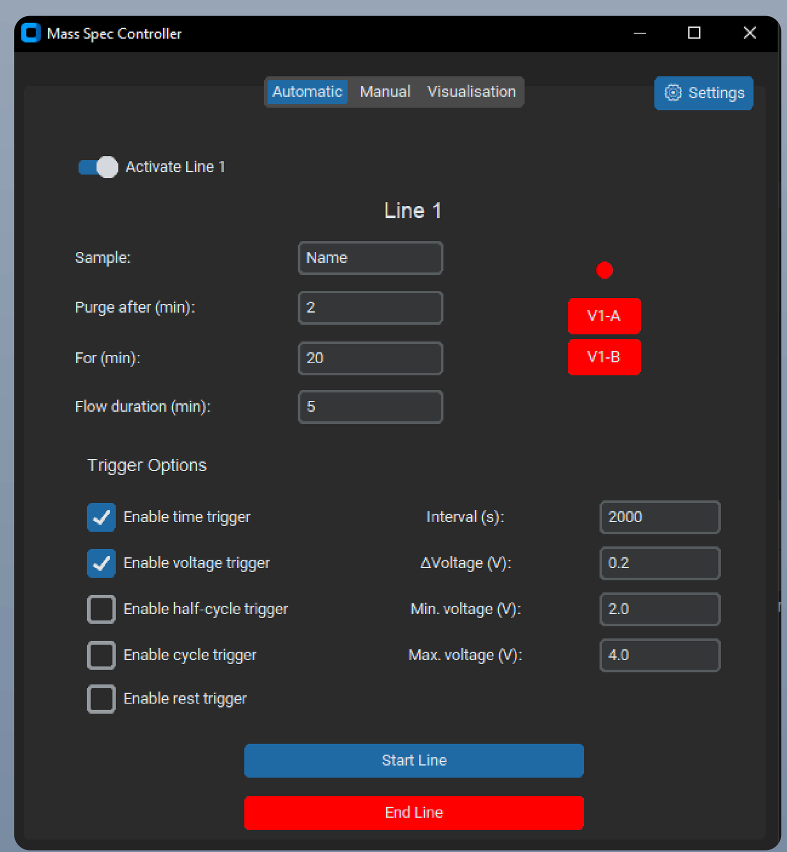

Figure 3: Software Interface

RESULTS

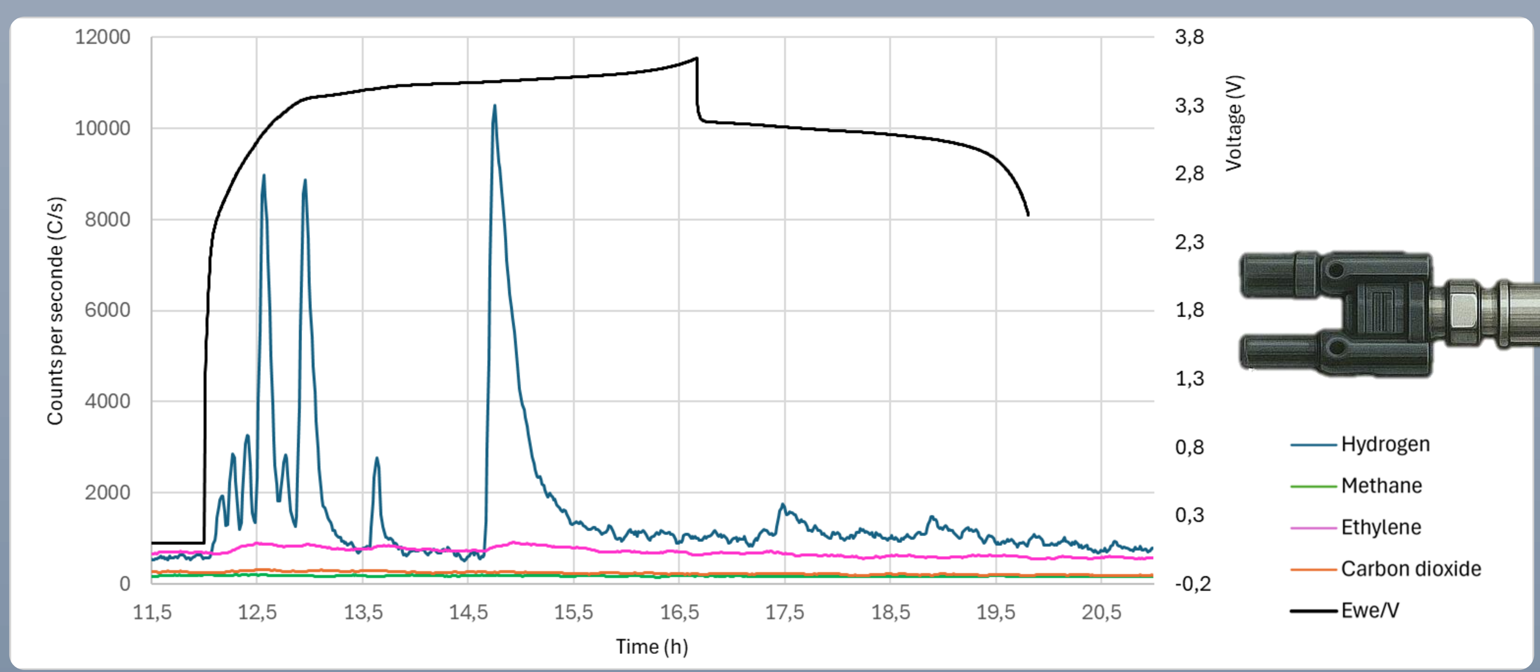

Figure 4: Gas production and cycling analysis

The mass spectrometry and voltage data shown in Figure 4, reveal distinct gas evolution events that correlate with the cycling process. Notably, **hydrogen** and **ethylene** are the most prominent species detected, indicating processes such as electrolyte decomposition and solid electrolyte interphase (SEI) formation. **Carbon dioxide** and **methane** are also present in lower but consistent quantities, typically associated with solvent breakdown and anode degradation [1].

The voltage profile, shows approximately a stable charge-discharge cycle, with characteristic plateaus around **3.5 V** during charging and **2.0 V** during discharging. The consistent curve shapes indicate good electrochemical stability throughout the measurement period. Moreover, this confirms **stable electrical connection** within the cell, which was a crucial requirement for reliable measurements.

During the measurement, an average of **800 counts per second (c/s)** of **air** is registered, as shown on Figure 5. When compared to the standard argon reference signal of 1.11×10^6 c/s, the air signal represents **0.072%** of the total gas quantity. This low relative intensity confirms the airtightness of the system and low background contamination.

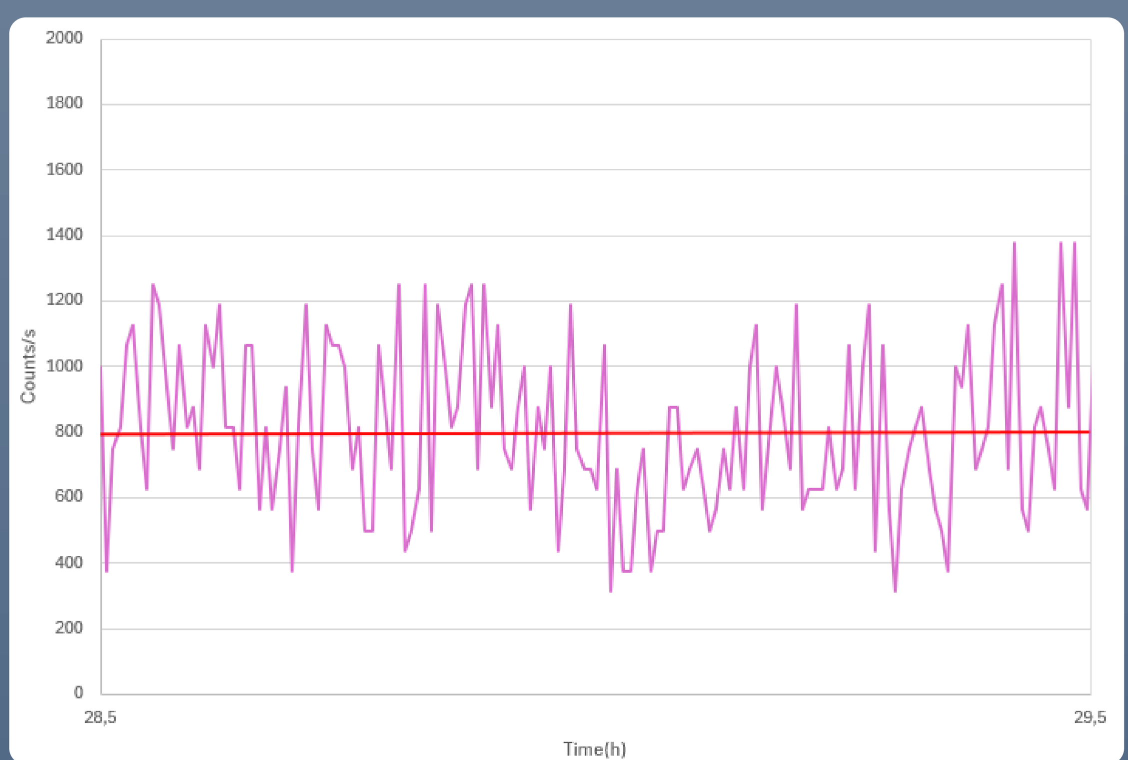

Figure 5: Air infiltration analysis

CONCLUSION

In this master thesis, a **fully automated** and **airtight** setup was successfully developed for **operando gas analysis** of pouch cell batteries, meeting all predefined objectives. A custom mechanical enclosure ensures airtight containment of the pouch cell, enabling controlled gas extraction during cycling. A dedicated gas flow line with solenoid valves was implemented and controlled via a **self-designed electronic circuit** based on an Arduino Nano. In addition, the system is operated through **custom-made software** featuring a graphical user interface (GUI) and built-in trigger logic based on time, voltage, and cycling events.

The system consistently maintained a **stable electrical connection** to the cell, as demonstrated by repeatable charge-discharge profiles with clear voltage plateaus. Airtightness was validated by the low air-to-argon signal ratio of **0.072%**, indicating minimal leakage and high measurement fidelity. This were crucial requirements for achieving reliable gas analysis during operation.

The mass spectrometry results confirmed the presence of expected gas species such as **ethylene**, **hydrogen**, **carbon dioxide** and **methane**. These findings demonstrate the system's effectiveness in capturing meaningful degradation signals and confirm its value as a robust platform for future operando diagnostics in lithium-ion battery research.

Supervisors / Co-supervisors / Advisors: Prof. dr. ir. Momo Safari
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[1] J. S. Edge *et al.*, “Lithium ion battery degradation: what you need to know,” *Phys. Chem. Chem. Phys.*, vol. 23, no. 14, pp. 8200–8221, 2021, doi: 10.1039/d1cp00359c.

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