RUB

RUHR-UNIVERSITÄT BOCHUM

Pressurized Thermo-electrochemical Reactor

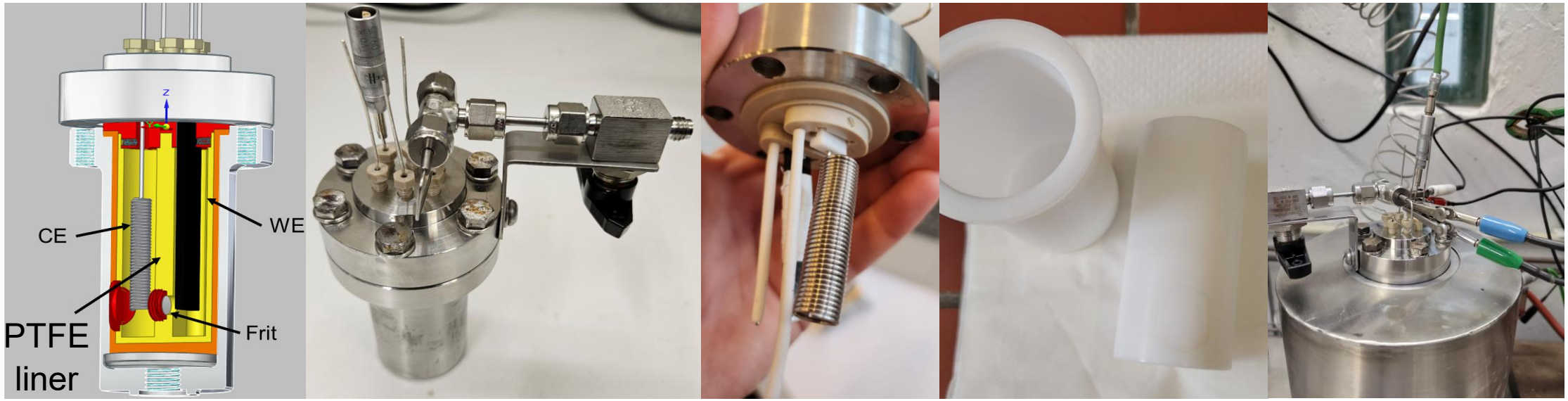
Catalina Leiva-Leroy/ Cluster meeting 16.07.2026

CRC/TRR 247
HETEROGENEOUS OXIDATION
CATALYSIS IN THE LIQUID PHASE

LEHRSTUHL FÜR TECHNISCHE CHEMIE RUHR-UNI BOCHUM

Combining thermal and electrocatalysis

Pressurized thermo-electrochemical reactor operated under galvanostatic conditions



WE: Co_3O_4 ink drop-coated on glassy carbon electrodes (loading: 1 mg cm^{-2}, area: 2.1 cm^{-2})
CE: Platinum coil **RE:** Pt wire
Separator: Zirfon Pearl UTP 500
Electrolyte: 1 M KOH + 1 M EG (Anode)
1 M KOH (Cathode)

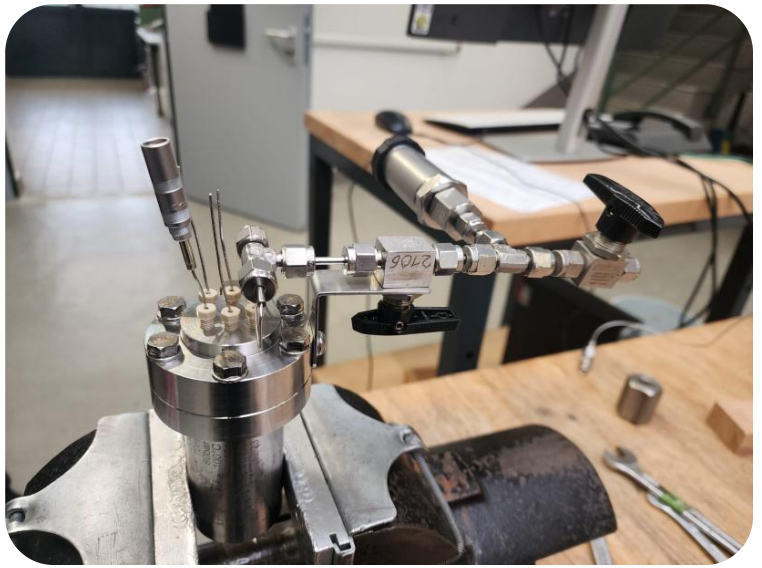
Experiments with the pressurized thermoelectric reactor
Chronopotentiometric: 30 mA (2h), 5 mA (12 h)
Reaction conditions: 30, 50, 70, 90 °C temperature
15 bar O_2 Pressure

A. Koul, C. Leiva-Leroy, M.L. Krebs, J.P. Fandré, A. Shekhawat, H. Tüysuz, F. Schüth, M. Muhler, W. Schuhmann, **"Electrothermal Oxidation of Ethylene Glycol over Co_3O_4"** 2026 *Angewandte Chemie*, e1818551.

Workflow

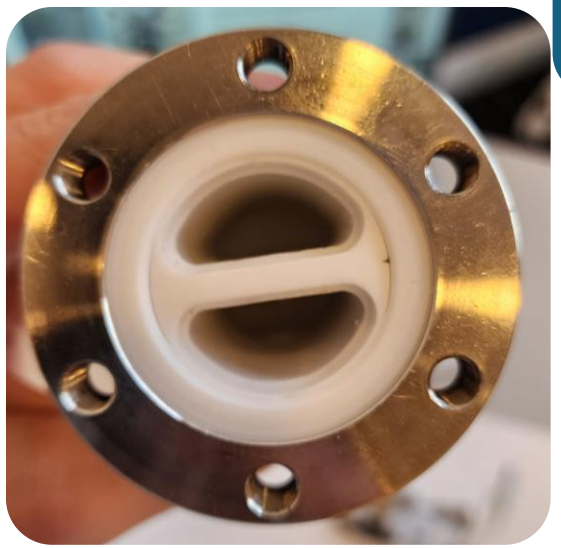
Preparation for measurements

- WE with drop-coated Co_3O_4.
- Reaction solution with 1 M EG (anode) and 1 M KOH (anode and cathode).
- Add small stirrers.
- Tight the screws between 15-20 Nm (torque wrench).
- Pressurization with monitoring.



Before reaction measurements

- Open-circuit voltage.
- Uncompensated resistance.
- Three CV 0.3–1.0 V vs P-RE at 50 mV s^{-1}.



Reaction

- Chronopotentiometry at:
- 30 mA for 2 h and 5 mA for 12 h (overnight).
- 15 bar O_2, up to 90 bar.
- 30 °C to 90 °C.

Analysis

- Recover the entire anode and cathode solution (separately).
- Acidify the solution and measure in HPLC.

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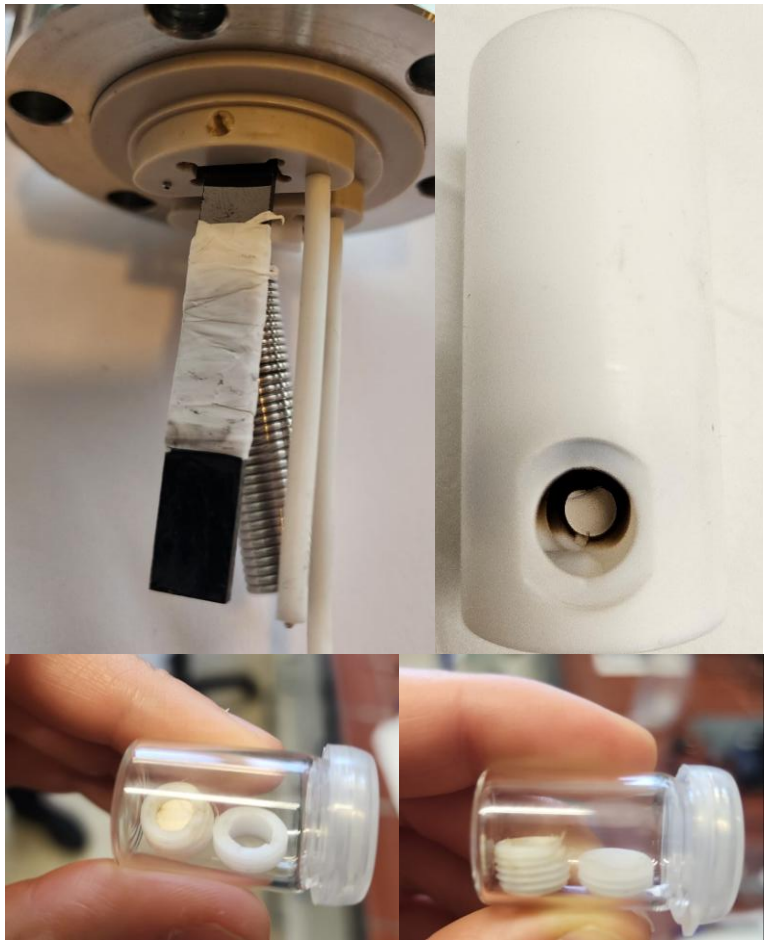
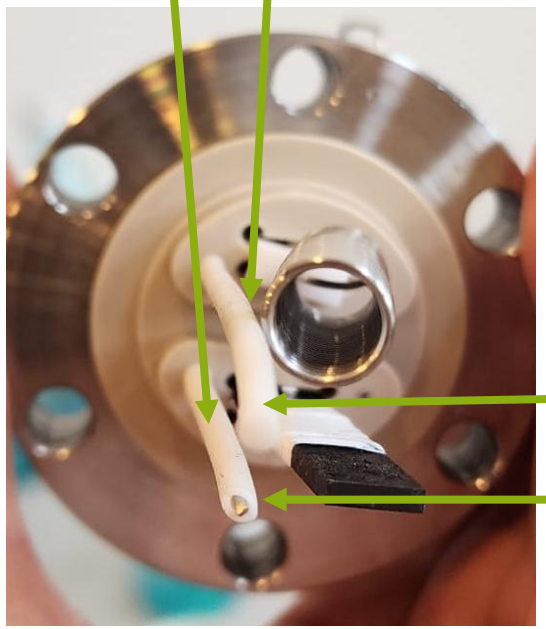
Extra details

- The working electrode was prepared by **drop-coating Co_3O_4 catalyst ink** (loading: 1 mg cm^{-2}) onto both sides of a **glassy carbon plate**, giving a total exposed catalyst area **of 2 cm^2**. The catalyst ink was prepared by dissolving 2 mg of catalyst in 96 µL ethanol and 4 µL Nafion 117.
- Anode and cathode **compartments were separated** by a Zirfon Perl UTF 500 diaphragm, this will be **changed to Nafion**.
- The closing of the **6 screws** has to be done by **tighten** in a crossing way and step by step (15-20 Nm).



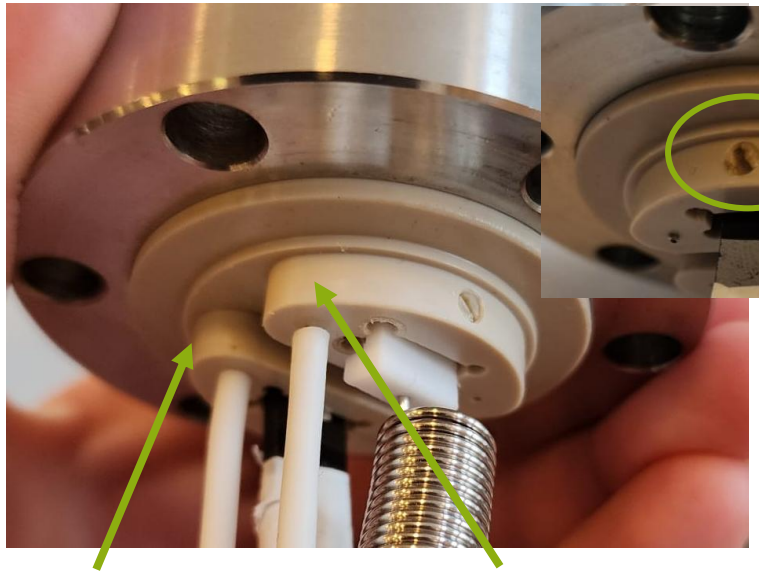
Our version of the reactor



Teflon cover

Thermocouple

Pseudo-reference electrode



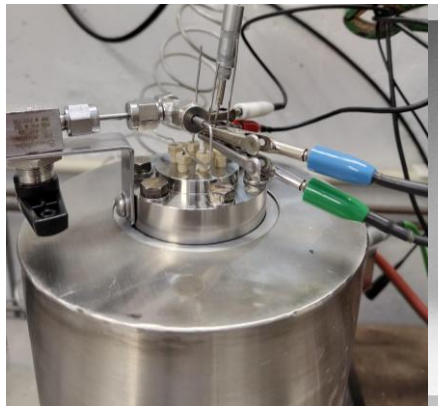
Screws will be added to fix the **Teflon covers** of the thermocouple and pseudo reference electrode.

The separator will be moved up 3 mm to **avoid stirring problems**.

Longer screw to remove the Teflon liner.

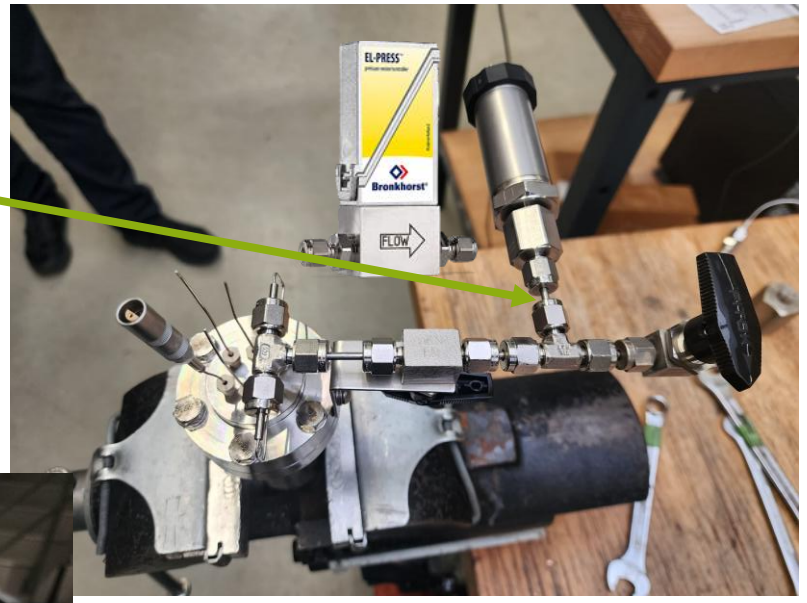
Our version of the reactor

Heating block

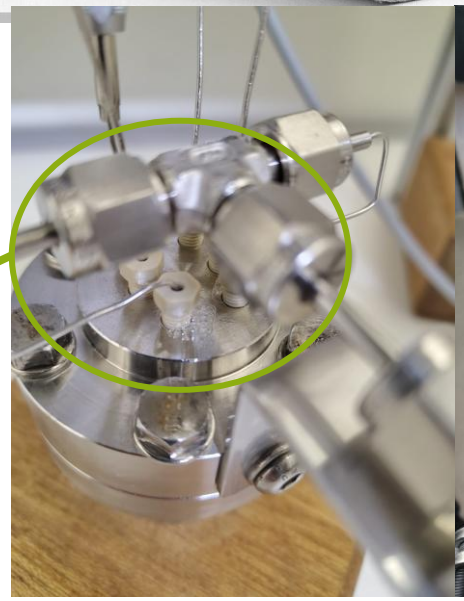


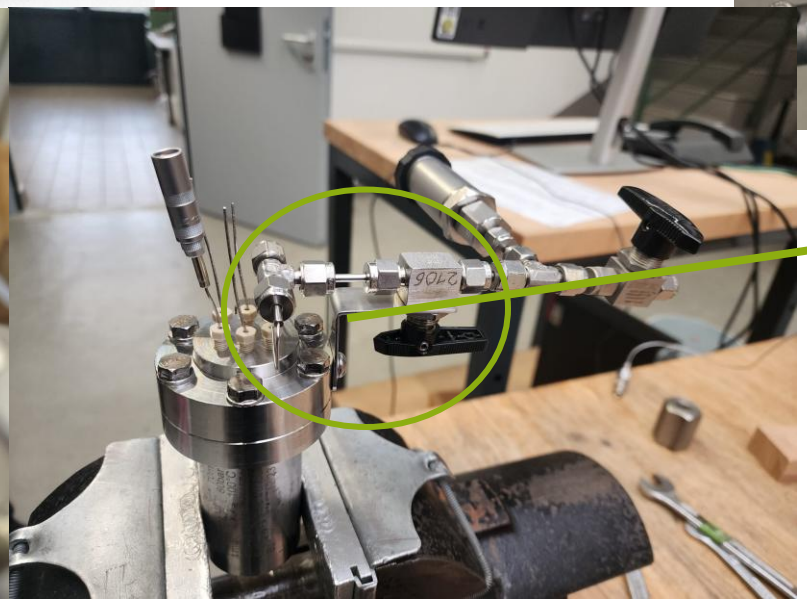
Heating jacket

Digital pressure meter, instead of a pressure gauge.



Leaks in the **capillary connections**





The pressure inlet connection will be **reinforced to avoid leak problems.**

https://www.horst.de/en/produkte/heating-jackets/hfh/hfb-350-c

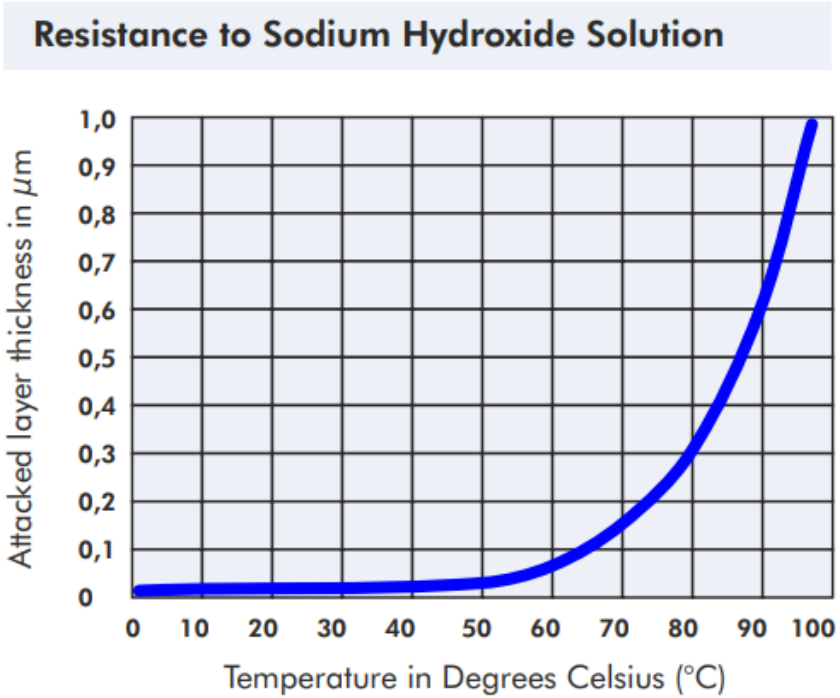
Future work on Büchi glass reactor

Material: Borosilicate glass 3.3, Teflon, PFA, FEP

Operation limitations:

- Temperature <150°C.
- Pressure (including heating) <10 bar.
- Alkaline conditions need to be studied to not damage the reactor.

This reactor could be modified for thermo-electrochemical and use a proper reference electrode.



The graphic shows the surface attack on borosilicate glass 3.3 by 4%weight Sodium Hydroxide Solution with pH 14 (1,0 mol/l NaOH) after one hour as a function of temperature.

https://www.buchiglas.com/en/pressure-reactors-stirred-autoclaves/miniclaveinert.html
https://www.robuglas.com/fileadmin/dateien/Bilder/Service/Downloads/Technische_Daten_im_Detail/laugenbestaend_vitrapor_d.pdf

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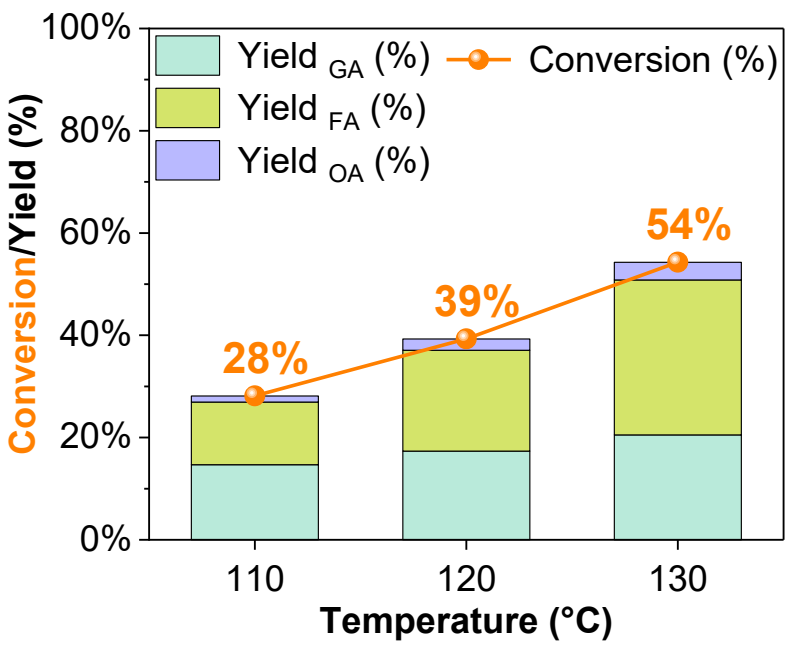
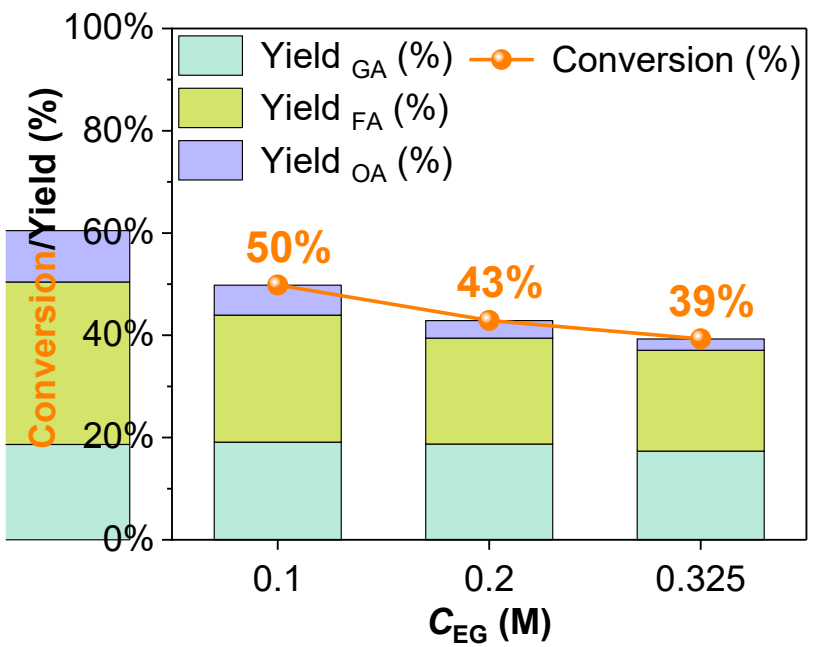
Previous experiments

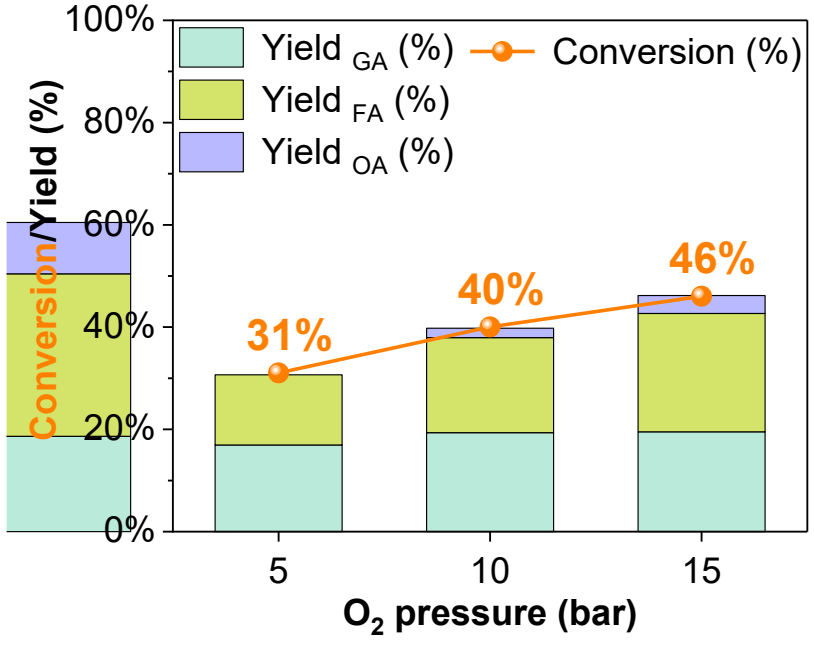
Kinetic study using **50 mg of spray-flame synthesized catalyst in 50 mL solution.**



Experiments performed at **0.65 M KOH, 0.325 M EG, 10 bar O_2**, 600 rpm for 6 h.



Experiments performed at **0.65 M KOH, 10 bar O_2, 120 °C,** 600 rpm for 6 h.



Experiments performed at **0.65 M KOH, 0.325 M EG, 120 °C,** 600 rpm for 6 h.

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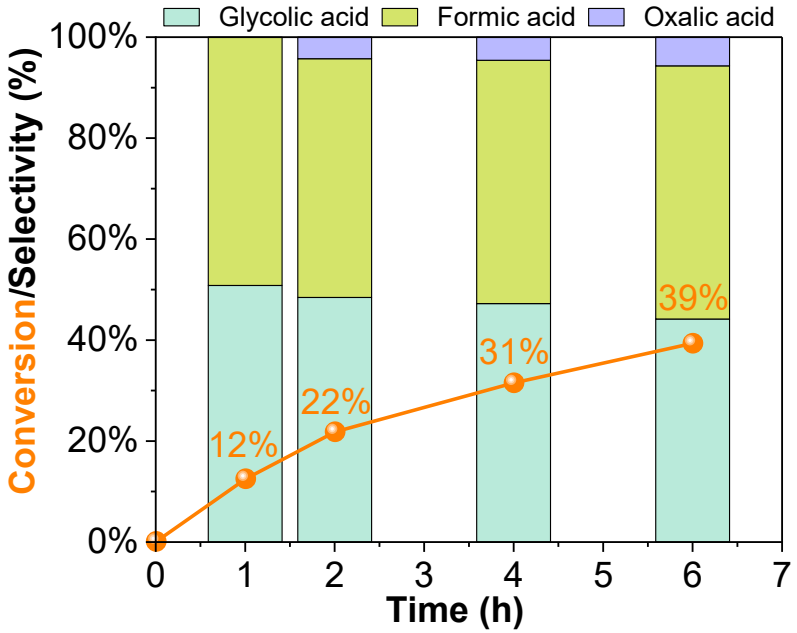
Previous experiments

Kinetic study using **50 mg of spray-flame synthesized catalyst in 50 mL solution.**



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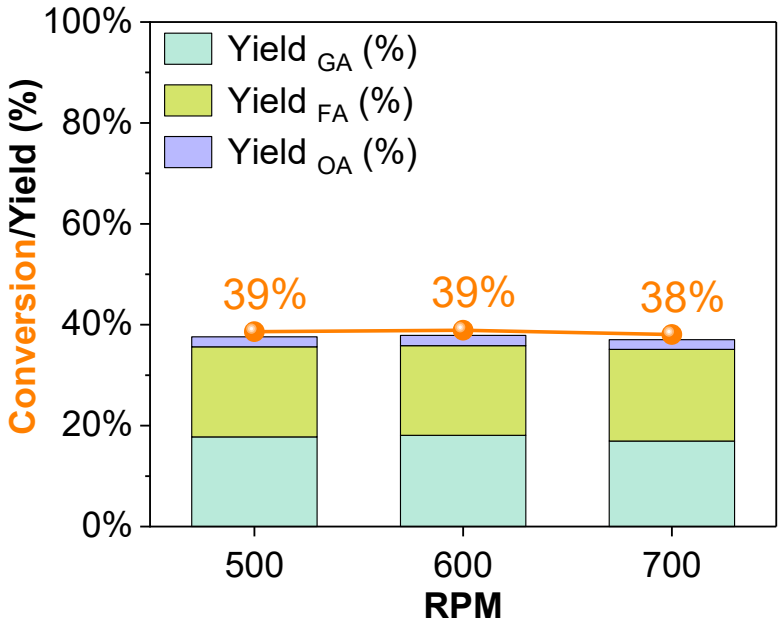
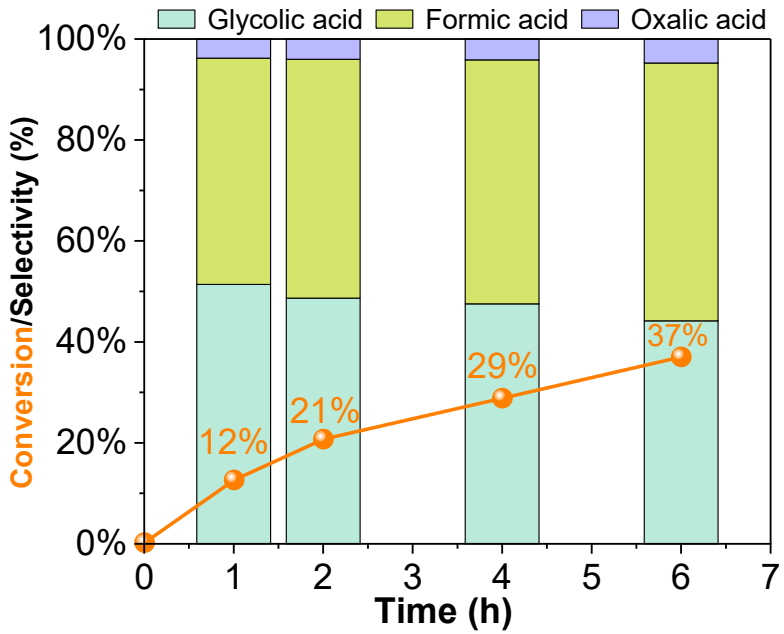


Experiments performed at **0.65 M KOH, 0.325 M EG, 10 bar O_2, 120 °C,** for 6 h.



Experiments performed at **0.65 M KOH, 0.65 M EG, 10 bar O_2, 120 °C,** for 6 h.

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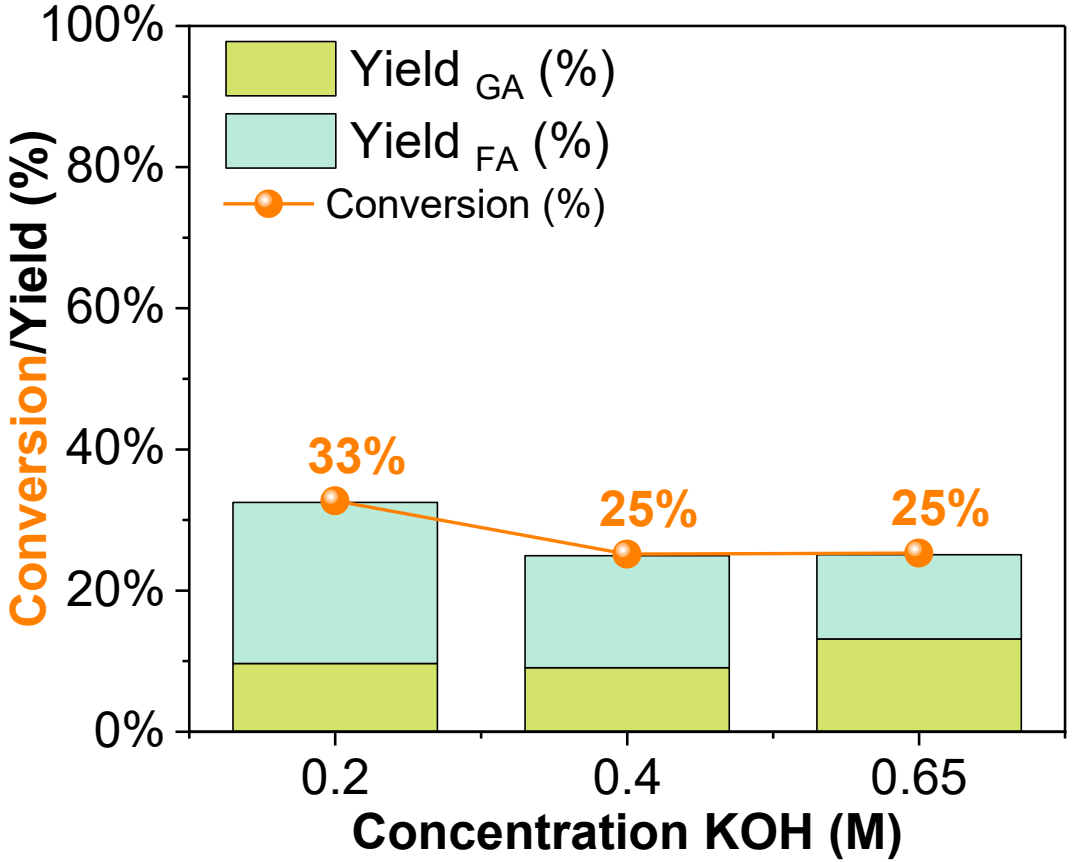
Previous experiments

Kinetic study using **50 mg of hard-templating synthesized catalyst in 50 mL solution.**



Experiments performed at, **10 bar O_2, 120 °C, 2:1=KOH:EG,** 600 rpm for 6 h.

Future work on Büchi glass reactor

Considering the kinetic study shown, the catalytic performance depends on:

- Elevated temperatures, up to 150°C.
- Lower EG concentration.
- Highly alkaline conditions (pH~13.5), that also results in higher KOH:EG ratio.
- Initial applied pressure before the heating, limited depending on the reaction temperature.
- Stirring speed applied to the reaction in the glass reactor.
- Reaction time, minimum of 6 h.
- Catalyst mass can be increased in the glass reactor, using the MCOSR sample (calcinated Co_3O_4 from Merck).

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Thank you for your attention!

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