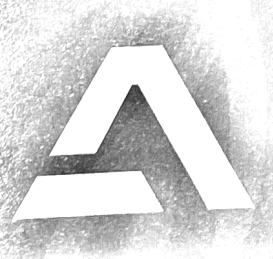
Book 3

Talal



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ME 149

Impact of Cations on Kolbe electro-oxidation

Acetic acid+ Sodium Acetate

Date	Experiment No	Current density mA/cm²	Total Molarity	Molarity acetic acid	Molarity Na-acetate	pH before	pH after	Volume ml	Electrolysis time min
		10	1M			3			
		10	1M			5			
		10	1M			9			
		10	1M			12			
5-7-2022	1	25	1M			3		30ml	90
		25	1M			5			
		25	1M			9			
		25	1M			12			
		50	1M			3			
		50	1M			5			
		50	1M			9			
		50	1M			12			

Acetic acid+ Potassium Acetate

		10	1M			3			
		10	1M			5			
		10	1M			9			
		10	1M			12			
		25	1M			3			
		25	1M			5			
		25	1M			9			
		25	1M			12			
		50	1M			3			
		50	1M			5			
		50	1M			9			
		50	1M			12			

Acetic acid+ Calcium Acetate

		10	1M			3			
		10	1M			5			
		10	1M			9			
		10	1M			12			
		25	1M			3			
		25	1M			5			
		25	1M			9			
		25	1M			12			
		50	1M			3			
		50	1M			5			
		50	1M			9			
		50	1M			12			

Important points

Date

Experiment No

Current density mA/cm²	Total Molarity	Molarity acetic acid	Molarity Na-acetate	pH before	pH after	Volume ml	Ele time min
Acetic acid+ Lithium acetate							
10	1M			3			
10	1M			5			
10	1M			9			
25	1M			12			
25	1M			3			
25	1M			5			
25	1M			9			
50	1M			12			
50	1M			3			
50	1M			5			
50	1M			9			
50	1M			12			
Acetic acid+ Cesium acetate							
10	1M			3			
10	1M			5			
10	1M			9			
10	1M			12			
25	1M			3			
25	1M			5			
25	1M			9			
25	1M			12			
50	1M			3			
50	1M			5			
50	1M			9			
50	1M			12			

Important Points

20220705 Impact of Cations full (main folder GC EC)

He flow in check = 31.5 ml/min (MFC = 30 ml/min)

red marker numbering are experiment number for GC data excel file

20220705 >> 1M Naacetate >> 20220705-pH3_25mA/cm² ①

Working electrode: Pt foil (double sided): area: 7x7mm □
= 49mm²
= 0.98cm² (double sided)
≈ 1 cm²

CE: Pt-ti

RE: Ag/AgCl

Electrolyte: 1M acetic acid + Na-acetate pH3 → 30 ml

volume of electrolyte: 30 ml

→ O_2 content was not decreasing: indicating problem of leakage:

All leakage test: soap, He detector: He detectors work best for minute leakage

→ Main leakage was the glass beaker of 50 ml alibaba reactor, not the cap.

Ⓐ Leakage problem was solved by the afternoon of 20220706. So, the directory folder and data of this experiment will remain the same 20220705_pH3_25mA/cm²

01_Purge: He 31.5 ml/min : purge time 45min

O_2 after purge 0.0305

02_CVs (0-3 V_{RHE}) - from inject 11 GC; CV start

50 mV/s, η=2

100 mV/s, η=2

150 mV/s, η=2

03_LSV: 100 mV/s -(0-3 V_{RHE})

04_CI: 208.876 Ω

05_ZIR: Ω

06_CC: mA/cm² : mA ⇒ V

t: 90 min

Control amplifier overload.

06_CC again: inject no 23 start 25 mA/cm² t: 90 min

V: 8.1 V

Overload again at inject no 24

GC injection 25

overload: till the C.E

through mesh

packed

0.5ml 0.5M H_2SO_4 to increase conductivity

System again 0.5ml H_2SO_4: 184.311 Ω GC injection: 30 - 47

Added 04_C2 after H_2SO_4 (0.5ml) $t = 90$ min

06_CC again at 0.5M H_2SO_4 (0.5ml) $I = 25$ mA, $E_{we} = 6.638$ V

25 mA/cm², $E_{oc} = 841$ mV

O_2 = 0.0299 before Kolbe : inject: 29

07_CV: 50 mV/s, 100 mV/s, 150 mV/s

$\eta_c = 2$ $\eta_c = 1$ $\eta_c = 1$

08_LSV: 0-3 V_{RHE} ⇒ −0.375 − 2.625 $V_{Ag/AgCl}$

09_C2 = 180.364 Ω (channel 2) 100 mV/s : 0-3 V_{RHE}

10_Z2R = 146.881 Ω (channel 1)

11_C2 = 176.169 (channel 1)

pH after = 7.7

F.E eth = 0%.

20220705 ⇒ 20220705-pH3-50mA/sq.cm. 2

01_C1 = 197.629 Ω

02_CC: 25 mA/cm² ⇒ 50 mA ⇒ amplifier overload:

20220706 >> 1M kacetate >> 20220706_pH 3_25mA/cm² 3

01 - purge He : 30 mins. 30 ml ↓ electrolyte

02 - CV (0-3 V_{RHE})

50 mV/s = 2

100 mV/s = 2

150 mV/s = 2

03_LSV : 100 mV/s : −0.375 − 2.6275 $V_{Ag/AgCl}$

04_C1 = 283 Ω

06_CC_25 mA/sq.cm : GC injection 7 to injection 24. : O_2 0.0290 (inj 6)

PH after: 2.31 (liquid sample collected) FE ethane 0%.

20220705 >> 1M Na acetate >> 20220707_pH5_25mA/cm²

01_purge He 31.5 ml/min 20ml electrolyte

02_CV (0-3 V_{RHE}) −0.4926 to 2.5074 | E_{OC} 351 mV

@ 50 mV/s $\eta_c = 1$, @ 100 mV/s $\eta_c = 1$, @ 150 mV/s $\eta_c = 1$

03_LSV (0-3 V_{RHE}) −0.4926 to 2.5074

@ 100 mV/s

04_C1 = 26.649 Ω (channel 2)

05_C1 = 23.526 Ω (channel 1)

06_Z2R = 17.647 Ω (without stirring)

07_Z2R = 16.692 Ω (with stirring)

08_CC @ 25 mA/cm² ⇒ 25 mA O_2 0.0351 (inject 11)

analysis inject 12 - 29

09_CV (0-3 V_{RHE}) −0.4926 to 2.5074 FE ethane 89.01%

@ 50 mV/s $\eta_c = 1$ @ 100 mV/s $\eta_c = 1$ @ 150 mV/s $\eta_c = 1$

10_LSV (0-3 V_{RHE})

11_C1 = 22.939 Ω (channel 2) pH: 4.8 liquid sample collected.

20220705 >> 1M K acetate >> 20220707_pH5_25mA/cm² 5

01_purge He 31.5 ml/min 20ml electrolyte

02_CV (0-3 V_{RHE}) −0.4926 to 2.5074 | E_{OC} 441.5

@ 50 mV/s, $\eta_c = 1$, @ 100 mV/s $\eta_c = 1$ @ 150 mV/s $\eta_c = 1$

03_LSV (0-3 V_{RHE}) −0.4926 to 2.5074 @ 100 mV/s

04_C1 = 16.21 Ω (channel 2)

05_CC 25mA/cm² ⇒ 25mA injection 4 - 21
(O_2 injection 3 = 0.0291

06_CV (0-3 VRHE)
50 mV/s nc=1, 100 mV/s nc=1, 150 mV/s nc=2

08_LSV @ 100 mV/s 0-3 VRHE

08_CI = 2059 Ω

(liquid sample forgot)

FEetoac = 93.21 %

20220705 >> 1M Kacetat pH5 >> 20220707_pH5_10mA/cm² (6)
Electrolyte volume = 20 ml
gas flow 31.5

01_ purge He

02_CV (0-3 VRHE)
50 mV/s nc=1, 100 mV/s nc=1, 150 mV/s nc=1

03_LSV @ 100 mV/s : 0-3 VRHE

04_ CI = 22.982 Ω Channel 2.

05_ CC @ 10 mA/cm² or 10 mA for 90 mins
injection 15 - 32, (O_2 before = 0.0285 injection)

06_CV (0-3 VRHE)
50 mV/s nc=1, 100 mV/s nc=1, 150 mV/s nc=2

07_LSV @ 100 mV/s 0-3 VRHE

08_CI = 19.101 Ω

(liquid sample taken)

pH after = 4.84

FE

20220705 >> 1M Nacetat pH5 >> Electro... EOx = 3.96 m (7)

01_ purge He

02_CV (0-3 VRHE) ⇒ -0.493 to 2.507 VAg/AgCl
50 mV/s nc=1, 100 mV/s nc=1, 150 mV/s nc=1
stopped after because I'm wrong: should be current density. 10 instead

03_LSV @ 100 mV/s 0-3 VRHE

04_CI = 30.864 Ω Channel 2

04_CI with stirring = 25 Ω

05_CC @ 15 mA/cm² or 15 mA for 90 min
from inj: 14 to 31, (O_2 before = 0.0334 injection 13)

06_CV (0-3 VRHE) ⇒ -0.493 to 2.507 VAg/AgCl
@ 50 mV/s nc=1, 100 mV/s nc=1, 150 mV/s nc=1
stopped.

07_LSV: @ 100 mV/s 0-3 VRHE

08_CI = 21.4.

20220705 >> 1M Nacetat pH5 >> 20220708_pH5_10mA/cm² >> rep (8)
20ml electrolyt

01_ purge He: (31 ml/min)

02_CV (0-3 VRHE) ⇒ -0.493 to 2.507 VAg/AgCl
50 mV/s nc=1, 100 mV/s nc=1, 150 mV/s nc=1

03_LSV @ 100 mV/s : 0-3 VRHE

04_CI = 21.633 channel 2

05_CC @ 10 mA/cm² or 10 mA for 90 min
from inj 13 to 30, (O_2 before = 0.0320, inj = 12)

06_ CV (0 - 3 VRHE)
50 mV/s nc = 1, 100 mV/s nc = 2, 150 mV/s nc = 3

07_ LSV (0 - 3 VRHE) @ 100 mV/s

08_ CI - 21.473 Ω

20220705 >> 1M Liacetate pH 5 >> 20220708_pH 5_ 25mA/cm²

Electrolyte volume 20ml (9)

01 - purge He 32.8ml/min

02_ CV (0 - 3 VRHE) => - 0.493 to 2.507 VAg/AgCl
50 mV/s nc = 1, 100 mV/s nc = 2, 150 mV/s nc 3

03_ LSV @ 100 mV/s => (0 - 3 VRHE)

04_ CI : 26.530 Ω

05_CC = 25 mA/cm² ≈ 25 mA 90 min
inj 7: 0.0303 Ov from injection 8 to 85

06 CI : 30.362 Ω

07_ CV (0 - 3 VRHE) => - 0.493 to 2.507 VAg/AgCl
50 mV/s nc = 1, 100 mV/s nc = 1, 150 mV/s nc = 1

08_ LSV @ 100 mV/s (0 - 3 VRHE)

(always flush the cell with 40ml/min for quick flushing) (10)

20220705 >> 1M Liacetate pH 5 >> 20220709_pH 5_ 10mA/cm²

electrolyte Vol = 20 ml

01- He purge He flow (actual) 31ml/min

02_ CV (0 - 3 VRHE) => 0.493 to 2.507 VAg/AgCl
50 mV/s nc = 1, 100 mV/s nc = 1, 150 mV/s nc = 1

03_ LSV @ 100 mV/s (0 - 3 VRHE)

04_ CI = 30.597 Ω

05_CC = 10 mA/cm² => 10 mA for 90 min (0, before 0312: inj: 12)
from injection 13 to 30.

06_CI_24.205 Ω

07_ CV (0 - 3 VRHE) => 0.493 to 2.507 VAg/AgCl
50 mV/s, nc = 1, 100 mV/s nc = 1, 150 mV/s nc = 1

08_ LSV (0 - 3 VRHE)
100 mV/s

oscillations in CV
↳ unstable Eue
↳ Eoc
cause: bubble in RE.
Solution: remove the bubble by finger striking

(11)

20220705 >> 1MCe acetate pH 5 >> 20220709_pH 5_ 25mA/cm²

· Electrolyte volume 20 ml

01_ He purge He flow (actual) = 31.2 ml/min

02_ CV (0 - 3 VRHE) => 0.493 to 2.507 VAg/AgCl
50 mV/s nc = 1, 100 mV/s nc = 1, 150 mV/s nc = 1

03_LSV @ 100 mV/s (0 - 3 VRHE)

04_CI : 16.023 Ω

05_CC: 25 mA/cm² ⇒ 25 mA (O₂ before 0.0319: inject 10)
from 11 to 28

07_CI: 16.508.

08_CV: (0-3 VRHE) ⇒ -0.493 to 2.507 VAg/AgCl
50 mV/s nc=1, 100 mV/s nc=1, 150 mV/s nc=1

09_LSV: (0-3 VRHE) at 100 mV/s (to repeat again because i forgot if i added acetic acid)

20220705 ⇒ 1M Ce acetate pH5 ⇒ 20220709_pH5_25mA/cm²_rep (12)
flow: 3 l/ml

01 He purge

02 CV (0-3 VRHE) ⇒ -0.493 to 2.507 VAg/AgCl
50 mV/s nc=1, 100 mV/s nc=1, 150 mV/s nc=1

03 LSV @ 100 mV/s = 0-3 VRHE

04 CI = 19.660 Ω

05_CC: 25 mA/cm² : 25 mA. (O₂ before 0.0326, inj 6)
from injection 7 to 24.

06_CI: 12.48 Ω

07_CV (0-3 VRHE) ⇒ -0.493 to 2.507 VAg/AgCl
50 mV/s nc=1, 100 mV/s nc=1, 150 mV/s nc=1

08_LSV @ 100 mV/s (0-3 VRHE)

20220705 ⇒ 1M Ce acetate pH5 ⇒ 20220

01_He purge

02_CV: (0-3 VRHE) ⇒ -0.493 to 2.507 Ag/AgCl
50 mV/s nc=1, 100 mV/s nc=1, 150 mV/s nc=1

03_LSV @ 100 mV/s = 0-3 VRHE

04_CI = 18.128 Ω

05_CC: 10 mA/cm² or 10 mA (O₂ before=0.327: Injection=4)
for 90 minutes from 5 to 22.

06_CI 15.502 Ω (compensation needed)

07_CV: (0-3 VRHE) ⇒ -0.493 to 2.507 Ag/AgCl
50 mV/s nc=1, 100 mV/s nc=1, 150 mV/s nc=1

08_LSV @ 100 mV/s = 0-3 VRHE

>> 1M Kacetate pH9 >> 20220718_pH9_25mA/cm² 14
flow = 31.4 ml/min
Volume electrolyte = 20ml

01_ He purge

02_ CV : (0-3 VRHE) = -0.729 to 2.271 VAg/AgCl
@ 50 mV/s nc=1 , 100 mV/s nc=1 , 150 mV/s nc=1

03_ LSV: (0-3 VRHE) : -0.729 to 2.271 VAg/AgCl
@ 100 mV/s

04_ CI: 14.435 Ω at channel 2

05_ CC : @ 25 mA/cm² = 25 mA (O₂ before 0.327 : inj= 11)
t: 90 min : from injection 12 to 29

06_ CI : 12.518 Ω at channel 2

07_ LSV: (0-3 VRHE) >> -0.729 to 2.271 VAg/AgCl
@ 50 mV/s nc=1 , 100 mV/s nc=2 , 150 mV/s nc=1

08_ LSV: (0-3 VRHE) at 100 mV/s

Liquid samples collected :
GC HS (vial HS 215) vial 201-202
Folder >> Ainoa Maged Talal >> 20220719 >> 01-48-Rep1 23

pH after 9.59

Rep
17-01-48
17-39-57
18 25 29

20220705 >> 1M Kacetate pH12 >> 20220718_pH12_25mA/cm²
He flow = 31.4 ml/m
volume = 20 ml

01_ He purge 15

02_ CV: (0-3 VRHE) >> -0.905 to 2.094
@ 50 mV/s nc=1 , 100 mV/s nc=1 , 150 mV/s nc=1

03_ LSV (0-3 VRHE) >> -0.905 to 2.904
@ 100 mV/s

04_ CI : 12.496 Ω at channel 2

05_ CC : 25 mA/cm² >> 25 mA (O₂ before 0.0086 inj: 11)
for 90 min from(injection 12 to 29)
Stopped due to leakage

20220705 >> 1M Kacetate pH12 >> 20220718_pH12_25mA/cm²_Rep2
He flow : 31.4 ml/min
volume = 20ml

01_ He purge 16

02_ CV : 0-3 VRHE >> -0.905 to 2.094
@ 50 mV/s nc=1, @ 100 mV/s nc=1 , 150 mV/s nc=1

03_ LSV : 0-3 VRHE >> -0.905 to 2.094 @ 100 mV/s

04_ CI : 11.817 Ω

05_ CC : 25 mA/cm² >> 25 mA (O₂ before=0.319 inject: 10
for 90 min >> from inject 11 to 28

07_CI : 11.205 Ω

08_ CV 0-3 VRHE >> -0.905 to 2.094 VAg/AgCl
@ 50 mV/s nc=1 , 100 mV/s nc=1 , 150 mV/s nc=1

09_ LSV: 0-3 VRHE >> -0.905 to 2.094 VAg/AgCl @ 100 mV/s

pH after: 9.21

GC HS samples: folder >> Ainoa Maged talal >> 20220719 >>
vial 202. ?

Rep
19-11-05_Rep1
195605 Rep2
195632 Rep3

20220705>>1M Nacetate +NaOH>>20220719_PH12_25mA/cm²
He flow: 31.7 ml/min
volume: 20 ml

17

01_purge Helium

02_CV : 0-3VRHE ⇒ -0.905 to 2.094
nc=1 @ 50mV/s, 100mV/s, 150mV/s

03_LSV: (0-3VRHE) ⇒ -0.905 to 2.094
@ 100 mV/s

04_CI = 12.941 Ω channel2

05_CC : 25mA/cm² ⇒ 25mA (O₂ before: 00328, inj 20)
for 90 min from inj 11 - inj 28

06_CI: 12.862 Ω

07_CV 0-3VRHE ⇒ -0.905 to 2.094
nc=1 @ 60mV/s, 100mV/s, 150mV/s

08_LSV 0-3VRHE ⇒ -0.905 to 2.094

samples taken
pH after =
loc:
Ti7 vial 205
Ainoa Margot Talab>> 20220720

20220705>>1M Nacetate +NaOH>>20220719_PH9_25mA/cm²
He flow = 31.7 ml/min
volume = 20 ml

18

01_purge Helium

02_CV 0-3VRHE ⇒ -0.729 to 2.271 VAg/AgCl
@ 50 mV/s, 100mV/s, 150mV/s nc=1

03_LSV : 0-3 VRHE @ 100 mV/s

04_CI = 17.290 Ω channel2

06 CC: 25mA/cm² ⇒ 25mA (O₂ before: 00306, inj no 13)
from 90 min from inj 14 to inj 32

07_CI: 14.349 Ω

08_CV : @ 100, 150, 50 mV/s, nc=1
from (0-3VRHE) ⇒ -0.905 to 2.094

09_LSV: 0-3VRHE
pH after:
T18 vial 216
loc: Ainoa Margot Talab>>20220720

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20220705>>1M Ceacetate +CeOH>>20220719_PH9_25mA/cm²
He flow = 31.9 ml/min
volume = 20 ml

01_purge Helium

02_CV: (0-3VRHE) ⇒ -0.729 to 2.271 VAg/AgCl
@ 50 mV/s, 100 mV/s, 150 mV/s nc=1

03_LSV : 0-3VRHE @ 100 mV/s
-0.729 to 2.271 VAg/AgCl

04_CI: 8.801 Ω channel2

05_CC: 25mA/cm² ⇒ 25mA (O₂ before 0.0334, injection = 2)
t: 90 min from injection 3 to inject. 20

06_CV: 0-3VRHE
@ 50, 100, 150 mV/s nc=1 -0.729 to 2.271 VAg/AgCl

07_LSV: 0-3VRHE
@100 mV/s

08_CI : 10.31 Ω

Liquid samples collected.
pH after.
T19 Vial no 217

loc: Ainoa Margot Talab >> 20220720

20220705 >> 1M Nacetate >> 20220719_pH9_25mA/cm² 5ep

Due to high FE than 4,ce: i want to repeat it again to see how it goes

01_ He purge He flow: 31.9

02_CC @ 25mA/gcm ⇒ 25mA (O₂ before 0.004 inj 5)
from inj 6 to 167 (14hr 10min)
no liquid samples: same sample from previous experiment

03_CV: 0-3 VRHE : -0.729V - 2.271 VAg/AgCl
nc = 1 @ 50, 100 mV, 150 mV/s

04_LSV: 0-3 VRHE : -0.729 V - 2.271 VAg/AgCl
nc = 1 @ 100 mV/s

05_CI: 12.946 Ω

Liquids ample taken : pH after

liq analysis ① ② ③

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2022 0705 >> 1M Nacetate >> 20220720_pH12_25mA/cm²
+CeOH

flow 32 ml/min

volume 20ml

01_ He purge

02_CV (0-3 VRHE) : -0.905 - 2.094 VAg/AgCl
nc 1 @ 50, 100, 150 mV/s

03_LSV : 0-3 VRHE : -0.905 - 2.094 VAg/AgCl
@ 100 mV/s (1.5 minutes rest time before LSV

04_CI: 10.647 Ω

05_CC: 25mA/cm² ⇒ 25mA (O₂ before 0.0318; inj 7)
t = 90 min from injection 8 to 25

06_CV: 0-3 VRHE : -0.905 to 2.094
nc = 1 @ 50 mV/s, 100 mV/s, 150 mV/s

07_LSV @ 100 mV/s (0-3 VRHE) : -0.905 to 2.094

08_CI: 9.934 Ω

Vial: 12-13-14-15 Liquid samples taken pH after

22

20220705 >> 1M Lioacetate + LiOH >> 20220720_pH9_25mA/cm
flow = 32ml/min
volume = 20ml

01_ He purge 30ml/min

02_CV: 0-3 VRHE from -0.729 to 2.271 VAg/AgCl
nc = 1 @ 50, 100, 150 mV/s

03_LSV: 0-3 VRHE from -0.729 to 2.271 VAg/AgCl
@ 100 mV/s

04_CI: 18.667 Ω channel 2

05_CC: 25mA/cm² ⇒ 25mA (O₂ before: 0.0318; inj: 7)
90 min time from inj 8 to 25

06_CI: 20.249 Ω

07_CV (0-3 VRHE): from -0.729 to 2.271 VAg/AgCl
nc = 1 @ 50, 100, 150 mV/s

08_LSV (0-3 VRHE) from -0.729 to 2.271
@ 100 mV/s

Liquid samples taken pH after

20220705 >> 1M Lactate >> 20220720_pH12_ 25mA sq/cm
He flow = 32 ml/min
volume 20 ml

01_ He purge

02_ CV = 0-3 VRHE : -0.905 to 2.049 VAg/AgCl
nc = 1 @ 50, 100, 150 mV/s

03_LSV = 0-3 VRHE : -0.905 to 2.049 VAg/AgCl

04_CI = 18.165 Ω

05_CC: 25 mA sq cm ⇒ 25 mA (O2 before 0.031, injection 5)
from 90 min from injection: 6 to 23

06_CV : 0-3 VRHE : -0.905 to 2.049 VAg/AgCl
nc 1 : 50, 100, 150 mV/s

07_LSV: 0-3 VRHE : -0.905 to 2.049 VAg/AgCl

08_CI : 19.368 Ω channel 2

Liquid samples taken, pH after.

20220721 (Pyrolysis oil analysis)

Samples preparation to GC-MS

Sugar acid fraction
↳ aqueous phase wt% water 50 oil 50
weight : 200mg 200mg

Phenolic fraction
↳ aqueous phase water 50 oil 50
200mg 200mg

Hydrogenated oil distilled phase
↳ aqueous phase water 50 oil 50
200mg 200mg

pH of aq phase

(for final step: dilution needed 20 parts acetone, 1 part oil aqueous phase)
↓
HPLC grade

filtered with 0.2 µm

sample ready in vials on 20220726 and given to GC MS / HPLC lab

GC-HS Liquid samples

Liquid analysis result

	Area 1	Methanol Area 2	Area 3	Methyl acetate	Others
T_1	NO			NO	
T_2	—			—	
T_3	476[illegible]	1695		5849	7060
T_4	[illegible]568	8722		52 [illegible]	2133 [illegible]
T_5	[illegible]52	NO		[illegible]	NO
T_6	5995	2852		15833	16393
T_7	—			—	
T_8	1567	10762		7832	7660
T_9	261	8[illegible]87		3746	3164
T_{10}	9006	[illegible]09751		450	2683
T_{11}	10119	10848		6630	9561
T_{12}	9796	9168		4834	5680
T_{13}	8728	2412		6869	6865
T_{14}	19747	19460	18701		44 / 699
T_{15}	—			—	—
T_{16}	19015	18547	19151		133379 / 126117 / 130827

End: [illegible]

	Area 1	Methanol Area 2	Area 3	Methyl acetate		
T_{17}	21101	20844	21255		18942	11483 / 13198
T_{18}	19838	18954	16496		28363	218598 / 197087
T_{19}	18378	23203	20592		67910	76937 / 74761
T_{20}	144067 / 139020	149287 / 137559	142523 / 137746	407 / 400 / 2737	5055	3204 / 3289
T_{21}	27845	24286	20597	390 / 781	115900	104473 / 89512
T_{22}	15249	12266	11707		26956	17048 / 20006
T_{23}	14554	14015	14755		5754	4663 / 7124
T_{24}	20386	18406	20606			
T_{25}	22904	24710	23305			
T_{26}	23956	28002		417 / 4299		
T_{27}	28166	25249				
T_{28}						
T_{29}						
T_{30}						
T_{31}						
T_{32}						
T_{33}						

T24

20220705 >> 1M Nacetate pH9 >> 20220722_pH9_25mA/cm²_replig
He flow = 31.6
Volume = 20 ml

* Doing this experiment because i saw ethanol in liquid product, the ethanol could be from cleaning because i used ethanol for cleaning before starting of experiment and rinsing thoroughly with water.
→ from GC Headspace, with discussion with Margot we came to conclusion that GC is very sensitive to ethanol, even with concentration of 0.1M we see a very sharp peak.
→ for this sample, we need to do quick injection soon after the experiment

01_ He purge

02_ CV: (0-3VRHE) → : -0.729 to 2.271 VAg/AgCl
nc = 1 @ 50 mV/s, 100 mV/s, 150 mV/s

03_ LSV (0-3VRHE) → -0.729 to 2.271

04_ CI = 13.821 Ω

05_ CC: 25mA/cm² → 25mA (I0_before = 0.335, injection 9)
from injection 10- for 90 min

06_ CV : (0-3VRHE) → -0.729 to 2.271 VAg/AgCl
nc = 1 @ 50 mV/s, 100 mV/s, 150 mV/s

07_ LSV (0-3VRHE) → -0.729 to 2.271 VAg/AgCl

08_ CI : 13.285 Ω

liquid sample instant analysis

Ethanol peak gone in liquid analysis

20220705 >> 1M Nacetate pH 12 >> 20220722_pH12_25mA/cm²_replig
He flow = 31.6 ml/min
Volume = 20 ml

01_ He purge

02_ CV : (0-3VRHE) → -0.905 to 2.094 VAg/AgCl
nc = 1 @ 100 mV/s, 50 mV/s, 150 mV/s

03_ LSV (0-3VRHE) → -0.905 to 2.094 VAg/AgCl

04_ CI = 12.684 Ω

05_CC = 25mA/cm² → 25mA I0_before 0.0303: inj 2)
for 90 mins → from injection number 3 to

06_ CV (0-3VRHE) → -0.905 to 2.094 VAg/AgCl
nc = 1 @ 50, 100, 150 mV/s

07_ LSV (0-3VRHE) → -0.905 to 2.094 VAg/AgCl

08_ CI: 11.947 Ω

T26

20220705 >> 1M Nacetate pH9 >> 20220722_pH9_25mA/cm²_replig
He flow = 31.6 ml/min
Volume = 20 ml

01_ He purge

02_ CV : 0-3VRHE → -0.729 to 2.271 VAg/AgCl
nc = 1 @ 50 mV, 100, 150 mV/s

03_ LSV (0-3VRHE) → -0.905 to 2.094 VAg/AgCl

04_ CI = 14.02 Ω

05_CC : 25mA/cm² → 25mA I0_before : 0.0337 injection 10)
90 mins from injection 11

07_CV : (0-3 VRHE) ⇒ -0.729 to 2.271 Vag/AgCl
@ 50, 100, 150 mV/s nc=1

08_LSV : (0-3 VRHE) ⇒ -0.729 to 2.271 Vag/AgCl
@ 100 mV/s

09_CI : 11.82 Ω

20220705 ⇒ 1MKacetate pH12 ⇒ 20220722_pH12_25mA_exp1y (TL)
volume = 20 ml
He flow = 31.6 ml/mn

01_He purge

02_CV : (0-3 VRHE) ⇒ -0.905 to 2.094 Vag/AgCl
nc=1 @ 50, 100, 150 mV/s

03_LSV: (0-3 VRHE) ⇒ -0.905 to 2.094 Vag/AgCl

04_CI: 11.295 Ω

05_CC : @ 25 mA/cm² ⇒ 25 mA (O₂ before = 3 inj0089)
for 90 mins ⇒ from inj 4 to

06_CV : 0-3 VRHE ⇒ -0.905 to 2.094
nc=1 @ 50, 100, 150 mV/s

07_LSV : (0-3 VRHE)

08_CI : 11.28 Ω

20220726 - 29072022

goal :

BDD influence : surface termination on current density of Kolbe oxidation

⇒ surface termination impact on electrocatalyst stability

⇒ attachment of different nanoparticles

⇒ SEM images
⇒ Raman
⇒ XRD
⇒ CV
⇒ Impedance
⇒ Stability cycles
⇒ Constant current / potential
⇒ Contact angle

⇒ Self absorb monolayer of carboxylic acid

⇒ reductive Kolbe with Ni, Ag, SS

Separation of carboxylic acids by means of electrodialysis

⇒ Oxidation of levoglucosan.
in H_2SO_4 on Pt and on BDD
50 mM

20220726

Levoglucosan oxidation

01_ purge Helium

02_ CV blank (0.5M H_2SO_4) pH(0)
0-2VRHE from -0.1976 to 1.8024 Vag/AgCl
@ 50, 100, 150 mV/s
(or dif pH solution is in 1.22 Vag/AgCl)

03_ LSV blank (0.5M H_2SO_4)
0-2VRHE
@ ~~50, 100, 150~~ 100 mV/s
(It is always better to rest step of 5 min before LSV to reduce OW because LSV starts from OCV)

04_ CI. 3.968Ω

05_ CV levo (50mM levo + 0.5M H_2SO_4)
0-2VRHE from -0.1976 to 1.8024 Vag/AgCl
@ 50, 100, 150 mV/s, nc = 2

06_ LSV levo (0.5M H_2SO_4 + 50mM levo)
0-2VRHE @ 100 mV/s

07_ CI. 4.233

Current density	inj 1	inj 2	inj 3
10 mA/cm²			
20 mA/cm²			
50 mA/cm²			
100 mA/cm²			

10_CC levo
10 - 25 - 50 - 100 150 200 mA/cm², 5 mins each

11_ 20mV/s LSV levo

20220728

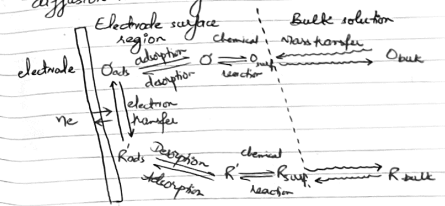
prepare stock 10mM, 20mM, 30mM, 50mM levoglucosan

prepare: 0.25 M H_2SO_4 & adjust pH to

prepare acetic acid + acetate stock

Why do we need RDE

"diffusion in electrolyte can limit the reaction"



Veniamin Levich invented RDE

forced convection take the electrolyte from below the electrolyte to the electrode surface

Rotation changes concentration profile

vs I at different RPM

Levich equation

$$i_{lc} = 0.62\, n F A D_O^{2/3} \omega^{1/2} \nu^{-1/6} C_O^*$$

Koutecky Levich

$$\frac{1}{i} = \frac{1}{i_K} + \frac{1}{i_{lc}}$$

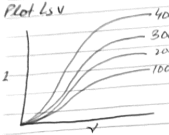
i_K: limiting factor by reaction on surface of electrode as f(V)

i_{lc}: limit factor caused by rotating speed

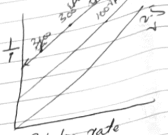
Experimental procedure:

Run LSV at 20 mV/s from (0 – 3 VRHE) (-0.492 to 2.507) at different rpm (100, 400, 900, 1600, 2500, 3600)

Plot LSV



Take values $\frac{1}{I}$ at different potential eg 0.1V, plot 0.5V, 2.1V, 2.5V



$$\frac{1}{I} = \frac{1}{iK} + \left(\frac{1}{0.62\, n F A D^{2/3} \nu^{-1/6} C} \right) \frac{1}{\omega}$$

(y-intercept: Kinetic current (mass transfer corrected))

iK vs E o pure tafel plot (mass transfer corrected)

Second experiment:

Electrolyt (0.5M Na_2SO_4 + H_2SO_4) pH 5.

Blank LSV at 100 RPM

Acetic acid LSV at 200, 400, 900, 1600, 2500, 3600

Constant current experiment 25 mA/cm² for 5 min each at 100, 400, 900, 1600, 2500, 3600

add concentration at different interval.

01_Blank CV @ 100, at 50 mV/s $n_c = 2$ (0–3 VRHE)

02_CV_OCV_03_CV – OCV_04CV_05OCV_06CV_07OCV_08CV

100 | 400 | 400 | 1600 | 2500 | 3600

03_CV OCV CV ⇒ same sequence like 2 but with Pt

with increasing rpm current is decreasing

20220729 Pt RRDE — 0.5 M Na_2SO_4

Electrolyte: 0.5 M Na_2SO_4

Disk channel 2 = Pt
Ring channel 5 = Pt

@1 blank 100 rpm
Disk: -0.49 V vs Hg (OVRHE) to 2.3076 (2.8 V vs RHE)
Ring: -0.8 V RHE to -1.7926 V vs Ag/AgCl (CP)

@100 mV/s
n=2

10 mV/s n=2

01 blank 400 rpm

01 blank 900 rpm

01 ~~blank 1600 rpm~~

02 50 mM acetic acid rpm 100

02 50 mM acetic acid rpm 400

02 50 mM acetic acid rpm 900

02 50 mM acetic acid rpm 1600

02 200 mM acetic acid rpm 100

02 200 mM acetic acid rpm 400

02 200 mM acetic acid rpm 900

02 200 mM acetic acid rpm 1600

02 1M acetic acid rpm 100

02 1M acetic acid rpm 400

02 1M acetic acid rpm 900

02 1M acetic acid rpm 1600

02 1M acetic acid rpm 100 _ more acetic acid
02 1M acetic acid rpm 400 _ more acetic acid
02 1M acetic acid rpm 900 _ more acetic acid
02 1M acetic acid rpm 1600 _ more acetic acid

; added more acetic acid to see the effect.

last
02 1M acetic acid rpm 1600 _ more acetic acid _ positive 0.8 VRHE

02 1M acetic acid rpm 100 _ more acetic acid _ positive 0.8 VRHE

02 1M acetic acid rpm 400 _ more acetic acid _ positive 0.8 VRHE

02 1M acetic acid rpm 6000 rpm _ acetic acid more

02 1M acetic acid rpm 8000 rpm _ more acetic acid

02 1M acetic acid rpm 4000 _ more acetic acid

02 1M acetic acid rpm 1500 _ more acetic acid.

02 1M acetic acid rpm 1500 _ more acetic acid _ ring 0.2 VRHE (Bastian said)

Electrolyte 1M acetic acid + sodium acetate

pH 5

from 0.5 VRHE to 3 VRHE : 0.0074 – 3 disk

RRing : -0.293 V vs Ag/AgCl : 0.2 VRHE

01_ 4000 RPM @ 50 mV/s (test)

01_ 4000 RPM_2 @ 50 mV/s nc 2

from -0.090 to 2.507 : 0.5 VRHE to 3 VRHE

02_ 6000 RPM : same conditions

03_ 3000 RRM : same condition

04_ 1500 RPM : same condition

05_ 900 RPM : same condition

06_ 400 RPM : same condition

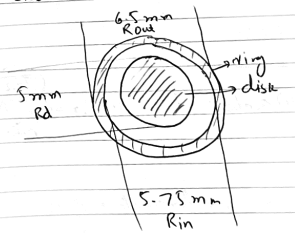
07_ 100 RPM : same condition

08_ CV : to be done : 69.010_52

RRDE Scheme

We 2 : ring	We 1 disk
	to channel 1 : work + Ref 3 + CE/CA 1 counter + wkt Ref 1 + ground. Reference wkt ref
	Cable E

Metrohm RRDE



Area of circular ring (Annulus)

$= \pi (R_{out} - R_{in}) \times (R_{out} - R_{in})$

Ring area = 0.07 cm²

Disk area : 0.19635 cm²

T31

20220801_1M acetic acid + 0.5 M Na_2SO_4

RRDE
initial pH 2.54
Argon purge 60ml
from 0.5 VRHE to 3 VRHE : 0.15254 to 2.652 Vag/agcl
Ring 0.2 VRHE : -0.14746
01_CI : 6.597Ω
02_4000rpm : same condition
03_CI
03_6000 rpm : same condition
04_3000 rpm : same condition
05_1500 rpm : same condition
06_400rpm : same condition
07_100rpm : same condition

T32

20220801_1M acetic acid + 0.5 M Na_2SO_4 + NaOH pH adjust

Initial pH 5
Argon purge : 60 ml/min , volume electrolyte 100ml
from 0.5 VRHE to 3 VRHE : 0.0074 Vag/agcl to 2.5074
Ring : -0.2926
02_4000 rpm : same condition
03_6000 rpm same condition
04_3000 rpm same condition

05_1500rpm : same condition
06_400 rpm : same condition
07_100rpm : same condition

Did not work too many problems with drop in fluctuation

01_CI :

T33

20220801_~~1M acetic acid~~ + 0.5 M Na_2SO_4 Blank

pH 5 , adjusted with H_2SO_4
Argon purge : 60 ml/min , volume electrolyte : 100ml
from 0.5 VRHE to 3 VRHE : 0.0074 Vag/agcl to 2.5074 Vag/agcl

02_4000rpm : same condition
03_6000rpm : same condition
04_3000 rpm same condition
05_1500 rpm : same condition
06_400 rpm : same condition
07_100 rpm : same condition
08_CI : 18.345Ω @ 4000 rpm

T34

20220801_0.5M Na_2SO_4 + 1M acetic acid

02_4000rpm : same condition
03_6000rpm : same condition
04_3000rpm : same condition
05_1500 rpm : same conditions
06_400rpm :
07_100 rpm

08_CI 19.7 @ 4000rpm

20220801_ Calibration GC file name: 20220801_2500ppm CO2

Co	mfc	Error	
1000	1009	±3.5%	
2500	2500	±3.5% → injection 2	to injection 12
5000	5012	±2.3% injection 13	to 17
10000	10000	±2% injection 18	to 22
30000	30000	±2% inject 23 - 27	
60000	60070	±2% inject 28 - 33	
90000	90104	±2.8% inj 34 - 37	
120000	120354	±3.5%	
45000	45089	±2.5% 38 - 42	
20000	20018	±2.1% 43 - 46	
20000	20018	49 - 52	
30000	30001	±2.8% 55 - 58	
60000	60008	±4.6% 59 - 62	
120000			
65000	65077	±2.3 64-6566-67	

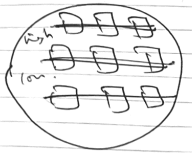
20220801_ Calibration Ethane

C_2H_6		
1000	-	inject 8 to 10
2500	2502	2% 11 to 16
5K		17 - 21
10K	10052	26 - 31
30K		32 - 37
60K		39 - 43
90K	90081	44 - 50
150K	100922	±4% 51 - 56
5 Kogan		59 - 68

Important point for working with Cordias Cell

- Cell comes with Nafion 324, it is in dry H form. Dry H+ membrane requires pretreatment in slightly alkaline water
- Side marked CATH must be installed facing Cathode, otherwise the membranes will be irreversibly damaged in use. If membrane is not marked, simply look / feel at both sides of membrane, the smoother / glossier side will be the cathode side.

BDD active diameter = 19.64 mm
active area ≈ 3.0295 cm²
1L 2L



Cleaning BDD electrode surface

Wash once with 2 propanol
twice with di water in ultrasonic bath
polarize diamond at 10 mA/cm² in 1M perchloric acid for 30 mins.

500 ml Perchloric $HClO_4$ acid 24.062 ml in 500 ml water

T35

20220804_ High doped BDD_ flow

Working electrode = BDD - high doped = area (active) =
counter silver. ss
Reference electrode: Hg/HgCl
Membrane = Nafion pretreated in water

Flow W.E 40 ml/min, pH 5, Electrolyte 1M Na acetate / Acetic acid
Flow C.E 40 ml/min, pH 5 (adj with H_2SO_4), Electrolyte 0.5 Na_2SO_4
Electrolyte volume = 120 ml both sides

01_ He purge: 392 ml/min (actual flow)
(gas flow during electrolysis = 33 ml/min)

02_CV: 0-3 VRHE : −0.493 to 2.507 V Ag/AgCl
@ 50, 100, 150 mV/s, nc = 1

03_LSV: 0-3 VRHE @ 50 mV/s, @ 100 mV/s (2 LSVs)

04_CI = 13.698 Ω

05_CC: 25 mA/cm² = 1.7573 mA = 1/A = 25 mA/cm² × 3.0295 = 1
(O_2 before = 0.0796, injection 21)
From injection 22 to 45
pH = 4.9 after 1 hr : Acetate
pH = 12.65 : sulfate

sample after 1 hr, 2 hr
→ test on GCHS + test on GCHS
for methanol for methanol
methyl acetate methyl acetate
→ test for H_2O_2 + test for A_2O

Didn't do the CV after forgot

Data GCHS

	1 hr	2 hr	1 hr	2 hr	2 hr	2 hr
MeOH	10739	13038	13165	28184	26939	28285
MeOH acetate	3623	4935	5232	6872	8769	7995
Inj 2				15907	12886	
				7395	491	

T32

20220804_ Newly fixed BDD flow

Divided cell, 40 ml/min flow both sides, no ml electrolyte both sides

He flow: 32 ml/min (same conditions as experiment T36 / sem before nanolab)

01_ He purge

02_ CV: 0-3 V RHE @ 50, 100, 150 mV/s — -0.495 to 2.507 Vag/agCl

03_ LSV: 0-3 V RHE @ 50, 100 mV/s

04_ CI = 15.710 Ω

05_CC: 25 mA/cm² 2 hr — 5) I = 75.73 mA, 10 before 0.0826, injection 6) from injection 7

06_ CV: 0-3 V RHE

07_ LSV: 0-3 V RHE

08_ CI = 16.409 Ω

(sample taken after 1 hr, 2 hr)

He flow during reaction: 33.4 ml/m → test for GC-HS

1) if test for H_2O_2 → test for GC-HS

2) test for H_2O

pH = 14 (after) cathode

I forgot to take the 2 hr sample, so maybe I do an experiment without gas product analysis and do for methanol but I took 1 hr sample already.

	1 hr	1 hr	1 hr	2 hr	2 hr	2 hr
Methylacetal	6631	8851	5827			
Methanol	12288	12257	13990			

(SEM image: after shavings) first pictures are low doped diamond 2 pics

T37

20220809_ RRDE: Ring collection efficiency

Bubble with Argon

RRDE: metrohm Pt disk-Pt Ring

Counter electrode: Pt mesh

Reference electrode: Ag/AgCl

Solution:

Ferricyanide $Fe(CN_6)^{-4}$ (5×10^{-3} mol/L) 10 mM

KCl = 0.5 mol L⁻¹ as supporting electrolyte. 1 M

Stock preparation: 200 mL of 10 mM $K Fe(CN_6)^{-4}$ in 1 M KCl

0.65848 g $K Fe(CN_6)^{-4}$ in 200 mL of 1 M KCl

i.e. 14.9102 g of KCl

01_ purge

02_ CV_RCA @100 rpm: I_d = ~~6.527~~ = 3.648 = 28.23 %; I_r = ~~-0.57~~ = 1.03

03_ CV_RCA @400 rpm: I_d = ~~4.9~~ 3.126 = ~~3.648~~ = 32.62 %; I_r = ~~-0.89~~ = ~~1.05~~ 1.02

04_ CV_RCA @ 2000 rpm: I_d = ~~3.145~~ = 2.6586 = 44 %; I_r = ~~0.5236~~ = -1.1725

05_ CV_RCA @ 4000 rpm: I_d = 1.9735 = 58.32 %; I_r = 1.151

06_ CV_RCA @ 500 rpm: I_d = 2.9 = 33.6 %; I_r = 0.975

07_ CV_RCA @ 6000 rpm: I_d = 1.17 = 86 %; I_r = 1.01

By biologic method

08 CV_RCA @ 6000 100c — 1disk 3236, 1ring 0.53 = 16.35%

09_ CV_RCA @400 rpm — 2disk 2.24 2ring 0.52 = 23.2%

10_ CV_RCA @ 1000 rpm — 2disk = 1.525 2ring = 0.34 = 22%

11_ CV_RCA @ 2000 rpm — 2disk = , 1ring

solution change because it turned to dark green.

012_ purge Ar

13_ CV_RCA @4000
(no purge Ar

T30

20220809_RRDE_1M ~~Nacl~~ acetic acid pHS

pH adjusted with NaOH & H_2SO_4
Working electrode Pt/Pt
Counter electrode Pt mesh
Reference electrode Ag/AgCl.

{ the time it is really 1M

01_ Purge Ar : 60 ml/min @ 50 mV/s

02_ 400 rpm : from 0.5 VRHE to 3 VRHE : 0.15254 to 2.652 $V_{Ag/AgCl}$
ring : −0.2926 $V_{Ag/AgCl}$: 0.2 VRHE
(first CV cycle is cycle better because bubble absorbs on the ring after first cycle
↳ bubbles prohibits the detection of ORR current

03_ 400 rpm : same conditions (but bubbles are not problem at this case.

04_ 900 rpm : same conditions

05_ 1500 rpm : same conditions

06_ 3000 rpm : same conditions

07_ 4000 rpm : same conditions

08_ 6000 rpm : same conditions

09_C1 : 28.217

2022 0810_RRDE_0.5M acetic acid in 0.5M NaOH

PHS : 100 ml : pH adjusted with NaOH
Working electrode : Disk Pt, Ring Pt
Counter electrode : Pt mesh
Reference electrode : Ag/AgCl

01_ purge Argon : 60 ml/min

09_ C1 = 24.782 Ω

02_100 rpm from 0.5 VRHE to 3 VRHE : ~~0.15254~~ to 2.507 @50mV/s 0.0074
ring : 0.2 VRHE ⇒ −0.2926 VAg/AgCl

03_ 400 rpm : same conditions

04_ 900 rpm : same conditions

05_1500 rpm : same conditions

06_ 3000 rpm : same conditions :

07_ 4000 rpm : same conditions

08_ 6000 rpm : same conditions

2022 0810_RRDE 0.5 acetic acid + Nacetate _ pHS

PHS, volume = 100 ml
WE, Disk Pt, Ring Pt
CE. Pt mesh
R.E . Ag/AgCl

01_ purge Argon 60 ml/min

02_100 rpm from 0.5-3 VRHE (0.007 to 2.507 VAg/AgCl) @ 50mV/s
ring : 0.2 VRHE ⇒ −0.2926 VAg/AgCl

03_400 rpm : same conditions

04_900 rpm : same conditions

05_1500 rpm : same conditions

06_3000 rpm : same conditions

07_4000 rpm same conditions

08_6000 rpm : same conditions

09_C1 : 23.559 Ω

20220810_RRDE_BDD_1M Nacetate acetic acid pH5

pH 5, volume 100 ml

WE: Pt ring, BDD disk ↳ 5mm dia

pH 5

CE: Pt mesh

RE: Ag/AgCl

01_purge Argon 60 ml/min

02_100rpm from 0.5-3 VRHE (0.007 to 2.507 VAg/AgCl)
ring at 0.2 VRHE ≙ -0.2926 VAg/AgCl

08_6000 rpm : same condition

07_4000 rpm : same condition

06_3000 rpm : same condition

05_1500 rpm: : same condition

04_900 rpm : same condition

03_400 rpm : same condition

02_100 rpm : same condition

09_CL ≈ 45.389

① test for CO_2 reduction

20220811_RRDE_BDD_1M Nacetate acetic acid pH5_methanol oxidation

methanol oxidises at 0.6 VRHE so first i try with CV to see if it works

01_purge Argon 60 ml/min

02_100rpm : from 0.5 to 3 VRHE ≙ (0.007 to 2.507 VAg/AgCl)
ring : 0.6 VRHE

03_1000rpm : same condition : no methanol detected

04_2000rpm : dis : 0.5 to 3 VRHE ≙ (0.007 to 2.507 VAg/AgCl)
ring : 0.6 VRHE
Disk: chronopotentiometry, ring: chronoamperometry
10 min ↳ at 25 mA sq/cm

05_2000 rpm_10 mA sq/cm : other conditions same

06_2000rpm CV

no methanol detection

T43

2022 0811_RRDE_ORR check:

Due to uncertainty of ORR potential, it was recommended to use electrolyte with a know concentration of methanol and do the CV on ring. In this way, we will be able to determine the correct onset potential, same can be done with O_2 purge solution.

Once we have optimum potential, we do the scan on disk and set the ring to one of the predefined potentials allowing for MeOH or O_2 detection.

02 CV 1000rpm

1VRHE to 0VRHE : 0.507 to -0.4926 @50mV/s

CVs with many noise.

onset ~ 0.1V Ag/AgCl

03 CV 1000rpm

1.2VRHE to -0.4926 ⇒ 0.7074 to -0.9926 VAg/AgCl.

onset ~ 0.15 VAg/AgCl

T44

2022 0812_RRDE_~~ORR~~ 1M Naacetate pH5

So to correct proper collection efficiency, we did CV on ring for ORR and now with experiment, want to check if we get proper collection efficiency

01_ purge Argon

02_1000 rpm:
Disk: 0.5 - 3VRHE : 0.0074 to 2.5074 VAg/AgCl
Ring: 0.35VRHE : -0.1426 VAg/AgCl

03_1000rpm
Ring at -0.1VAg/AgCl ~~0.5~~

04_1000rpm
Ring at -0.15VAg/AgCl

05_1000rpm:
Ring at -0.296VAg/AgCl

06_1000rpm:
Ring -0.3926

07_1000rpm: Ring : -0.3926 without Argon

08_1000rpm: Disk at 1.9625 mA
Ring 0.3VRHE : -0.1926

09_1000rpm: Disk at 1.4V $CV_{0/1Ag}$ = 95% FE
Ring -0.~~393~~V CV

10_1000rpm: CV: disk 0-1.4VAg/Ag
Ring -0.4 VAg/AgCl

11_1000rpm: CV: 0-1.4Ag/AgCl
Ring: -0.5 Vg/AgCl

12_1000rpm CV: 0-1.4 VAg/Ag ≃ above 100%
Ring: -0.7 VAg/AgCl

13_1000 rpm CV: 0 – 1.4 V Ag/AgCl ≈ above 100
Ring: 0.6

14_1000 rpm: CV: 0 – 1.4 V Ag/AgCl ≈ 36%
Ring: -0.5 V Ag/AgCl

15_2000 rpm: CV = 0 to 1.4 V Ag/AgCl ≈ 30%
Ring -0.5 V Ag/AgCl → no dips

16_1000 rpm CV: 0 to 1.4 V Ag/AgCl ≈ 70%
Ring = -0.55

17_1000 rpm: CV: 0 to 1.4 V Ag/AgCl ≈ 89%
Ring = -0.57 V → dips at ring curve

18_1600 rpm: CV: 0 to 1.4 V Ag/Ag ≈ 79%
Ring = -0.57 V still dips
→ dips gone by stop purging

19_1600 rpm: CV: 0 – 1.4 V Ag/AgCl ≈ 97.5%
Ring = -0.590 V Ag/AgCl

20_1600: CV: 0 – 3 V RHE
Ring = -0.590 V Ag/AgCl

21_1600 CV: 0 – 3 V RHE
Ring = -0.7

22_1600: CV: 1 – 2.2 V Ag/AgCl ≈ 23%
Ring – 0.7

23_1600: CV: 1 – 2.2 V Ag/AgCl
Ring – 0.9

24_1600: CV: 1 – 2.2 V Ag/AgCl
Ring = 0.8

25_1600: CV: 1 – 2.1 V Ag/AgCl above 100
Ring = 0.9 V Ag/AgCl

26_1600: CV: 1 – 2.1 V Ag/AgCl
Ring = -0.85

26_1600 rpm: CV: 1 – 2.1 V Ag/AgCl
Ring 0.85

27_1600 rpm: CV: 1 – 2.1 V Ag/AgCl

28_1600 rpm: CV: 1 – 2.5 V Ag/Ag
Ring = -0.85

29_1600 rpm: CV: 1 – 2.5 V Ag/Ag
Ring = 0.342 V RHE → change to 0 vs Eoc ≈

29_1600 rpm: CV: 1 – 2.5 V Ag/Ag
Ring – 0.193 V RHE X

30_1600 rpm: CV: 1 – 2.5 V Ag/AgCl
Ring:

T45

20220815_BDD_Low loading_50mA/cm²

WE: BDD Condias low doped
CE: SS
Electrolyte volume = 120ml
Anode electrolyte: 1M Nacetate pH5
Cathode electrolyte: 0.5M Na_2SO_4 pH5: adjusted with H_2SO_4
RE: Ag/AgCl
Gas purge flow rate = 33.1 ml/min
Flow rate 40 ml/min both sides

01_ He purge

02_ CV: 0-3 VRHE ⇒ −0.493 to 2.507
@ 50, 100, 150 mV/s

03_LSV: 50 mV/s : 0-3 VRHE
04_LSV: 50 mV/s : 0-3 VRHE
05_CI: 14.1178 Ω

06_CC: 50 mA/cm² = 151.398 mA (0.0748)
t = 2hr (10 before 0748, Injection 12)
from injection 13

Sample taken T45(1)

07_CV: 0-3 VRHE
@ 50, 100, 150 mV/s

08_LSV: 50 mV/s 0-3 VRHE, 100 mV/s

09_CI: 15.760 Ω

33.4 ml/min

sample taken T45(2)

Liquid analysis

	1hr	1hr	1hr	2hr	2hr	2h
pH catholyte = 13.1	Methyl acetate	6704	6895	9007	10646	11250	10948
pH anolyte = 5	Methanol	21403	21457	28237	43063	42780	47992

T46

20220815_BDD_high loading_50mA/cm²

WE: BDD Condias high doped
CE: SS
Electrolyte volume 120ml
Anode electrolyte: 1M Nacetate pH5
Cathode electrolyte: 0.5M Na_2SO_4 pH5: adjusted with H_2SO_4
RE: Ag/AgCl
gas purge 33.1 ml/min
flow rate: 40 ml/min both sides

01_ He purge

02_CV: 0-3 VRHE ⇒ −0.493 to 2.507 Vag/AgCl

03_LSV: @ 50, 100 mV/s ⇒ 0-3 VRHE

04_CI: 15.320 Ω

05_CC: 50 mA/cm² ⇒ 151.398 mA
t = 2hr (10 before 0.0790, Injection 7)
from injection 8 to

07_CV: 0-3 VRHE ⇒ −0.493 to 2.507 Vag/AgCl

08_LSV @ 50, 100 mV/s ⇒ 0-3 VRHE

09_CI: 13.938 Ω
pH catholyte: 13.1
pH anolyte: 5.

Flow output 33.6 ml/min

Liquid analysis

	1hr	1hr	1hr	2hr	2hr	2hr
Methylacetate	10447	8587	11240	15495	17794	15262
Methanol	31440	31875	37528	73012	80452	73839

(ec data stored in impact of cations folder) 147
(FE data
to 20220705_Impact of cations full)
20220816_ 1M Kacetate pH12_10mAsqcm
WE: Pt foil double side 1cm²
CE: Pt/Ti mesh
32.3 ml flow He
volume electrolyte 20ml
RE: Ag/AgCl

01_ He purge : 32.3 ml

02_ CV : 0-3 VRHE ⇒ −0.9056 to 2.0944 VAg/AgCl
@ 50, 100, 150 mV/s   nc=1

03_ LSV: 0-3VRHE ⇒ −0.9056 to 2.0944 VAg/AgCl

04_ CI : 11.938 Ω

05_ CC   10mA sqcm   (O_2 before = 0.0411   injection 9)
for 90 mins   (from injection 10 to)
(3.75 start injection)

06_ CV: 0-3 VRHE   @ 50, 100, 150 mV/s

07_ LSV = 0-3VRHE   , 100 mV/s

08_ CI = 11.663 Ω

PH after : 9.9

	1hr 30min	1hr30	1hr30 min	~~2hr~~	~~2hr~~	2hr
Methanol	8582	8157	7134			
Methyl acetate	816	495	5281			

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(same le ...
to 20220705 - Impact of cations full

20220816_ 1M Kacetate_pH 12_50mAsqcm
WE: Pt foil double side 1cm²   RE: Ag/AgCl
CE: Pt/Ti mesh
32.3 ml flow He
volume = 20 ml

01_ He purge

02_ CV   0-3VRHE ⇒ −0.9056 to 2.0944 VAg/AgCl
@ 50, 100, 150 mV/s   nc=1

03_ LSV   0-3VRHE   @ 100 mV/s

04_ CI : 12.234 Ω

05_ CC : 50 mAsqcm : I = 50 mA   90 min
(O_2 before 0.0384, injection 3)
from injection   to

PH: 11   (correct CV with pH)
corrected 0.847 to 2.153

06_ CV : 0-3 VRHE
@ 50, 100, 150 mV/s   nc=1

07_ LSV: : 0-3 VRHE   ⇒ −0.847 to 2.153 VAg/AgCl
@ 100 mV/s

08_ CI. = 15.382 Ω

	1hr 30 min	1hr 30 min	1hr 30 min	~~2hr~~	2hr
Methanol	42625	38962	40967		
Methyl acetate	5208	4973	6854		

T49

20220817_1M Kacetate pH12_25mA/cm electrolyte volume 20 ml

He flow 62.4 ml/min; it is 424 because i forgot to do 38
WE: Pt foil 1cm²
CE: Pt/Ti
RE: Hg/HgCl

01_He purge

02_CV = -0.9856 to 2.0944 : 0-3VRHE
@ 50, 100, 150 mV/s

03_LSV = @ 100 mV/s : 0-3VRHE

04_CI: 11.728 Ω

05_CC: 25mA/O. before. OP380 injection 7 from injection 8

0 PH= after = 10

06_CV: 0- 3VRHE : from -0.786 VAg/AgCl to 2.272 VAg/AgCl
@ 50, 100, 150 mV/s

07_LSV: 0- 3VRHE : from -0.786 VAg/AgCl to 2272

08_CI: 12.033 Ω

Sample taken

Methylacetate	4144	2293	4416
Methanol	19496	22685	23670

T50

20220817_1M Cacetate pH12_10mA/cm

He flow = 31.3 ml/min
WE: Pt foil
CE: Pt/Ti
RE: Hg/HgCl

electrolyte volume = 20ml

01_He purge

02_CV: 0-3 VRHE : VAg/AgCl
@ 50, 100, 150 mV/s

03_LSV: @ 100 mV/s ⇒ 0-3VRHE

04_CI: 10.575 Ω

05_CC: 10mA/cm² ⇒ 10 mA : inject = 0.0[illegible]. 0 before
from injection no 20 to

PH after = 10.4

06_CV = -0.812 to 2.188 VAg/AgCl

07_LSV = 0-3VRHE

08_CI = 10.181 Ω

Methyl acetate	397	5618	692
Methanol	9887	10249	11922

T51

20220818_1M Ce acetate_pH12_25 mA/cm

He flow = 32.6 ml/min
Electrolyte volume = 20ml
WE: Pt foil
RE: Ag/AgCl

OL He purge :

02_CV : -0.905 to 2.094 V$_{Ag/AgCl}$: 0-3 V$_{RHE}$
@ 50, 100, 150 mV/s

03_LSV : 0-3 V$_{RHE}$

04_CI = 10.600 Ω

05_CC: 25 mA/cm^2 ⇒ 25 mA for 90 mins (0, before ~~0.0396~~ 0.0419, injection 15)
from injection 15 to
pH = 10.2

06_CV: 0-3 V$_{RHE}$ from -0.812 to 2.094
@ 50, 100, 150 mV/s

07_LSV: 0-3 V$_{RHE}$

08_CI: 9.870

Sample collected

Methyl acetate 1766 6482

Methanol 27041 28088

20220818_1M Ce acetate_pH12_50 mA/cm^2

He flow = 32.6 ml/min
Electrolyte volume = 20 ml, CE = Pt/Ti
Pt foil
Ag/AgCl

He purge

CV: -0.905 to 2.094 V$_{Ag/AgCl}$: 0-3 V$_{RHE}$
@ 50, 100, 150 mV/s

LSV: 0-3 V$_{RHE}$

CI: ~~9.870~~ Ω 13.945

CC: 50 mA/cm^2 ⇒ 50 mA (0, before 0.0419, injection 3)
from injection 4 to
pH after: 10.

06_CV: 0-3 V$_{RHE}$ -0.812 to 2.188 V$_{Ag/AgCl}$

07_LSV: 0-3 V$_{RHE}$

08_CI: 12.572

Sample collected:

Methyl acetate	8366	10974	9753
Methanol	132105	107948	139171

20220831 Meeting

Meeting with Bastian for H_2O_2

Run LS : BDD stability in flowcell for water oxidation is

Plot Ls missing

What they in paper:
- continuous H_2O_2 at current density 100, 200, 300 mA/cm² at constant flow 100 ml/min.
- H_2O_2 conc highest 200 mA/cm² after 150 mins
- Catin exchange membrane
- single pass flow

Effect of stabilizer: NO_3 SiO_3

Effect of flow rate

Future work: high capital for BDD ⇒ look for other carbon based material.
- coupling with H_2 evolution or other cathodic reaction

two different couples
with pillars & non pillars.
- continuous flow.

+High doping density & high dense pillar ÷

- we don't need expensive ...

⇒ Gas ... of ...

20220831_ Kolbe Divided two electrode Pt foil Condition (153)

20220832_ flow cell condition Kolbe.

20220831_ Impact of

WE: Pt foil : active area

CE: Stainless steel

Electrolyte volume = 240 ml

Anolyte purge: He: 31.8 ml/min : connection to GC

Catholyte volume: 40 ml : no purge : no connection to GC

: no to find optimal conditions:

⇒ just at optimum current density, we change the flow rate.

flow rate: 10 ml/min = 22.96 rpm

01_ ~~He~~ purge: 31.8 ml/min

02_CC : 30 mins at 25 mA/cm² (O_2 before = ~~0.0626~~ 0.0626, injection 12)
from injection 13
(due to the connection problem from catholyte & unavailbity of connecta, flow was not going properly from catholyte and ; could see the catholyte flow impact on the cell potential because I tried to change the catholyte flow for higher flow

03_CC: 30min at 25 mA/cm² again (O_2 before = 0.0361: injection 3)
at 10 ml/pm from injection ~~6~~ 11 : fluctuations again
so change the membrane & solution & started again

04_CC: 30 min at 25 mA/cm² again (O_2 before .006 ; injection 6)
at 10 ml/pm from injection ~~7 to~~ 12 to 18.

05_CC: 30 min at 25 mA/cm² (O_2 before: 0.0636, injection 26)
at 20 ml/pm from injection 27 to 32

06_CC: 30 min 25 mA/cm² (O_2 before 0.0659, injection 39)
at 40 ml/pm from injection 40 to 9)

continue next page

07_CC 30 mins at 25 mA/cm² → 75 mA
at 80 ml/min 10, before 0.0684, injection 54)
from injection 55

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Parent folder
20220901 - different doped BDD flow 100mA sq cm

20220902_BDD low doped_100mA sq cm

WE
condias BDD low doped
CE : SS
Anode electrolyte 40ml 1M Naacetate pH5
Cathodyte: 0.5M Na_2SO_4 pH5 (adjusted with H_2SO_4) 40
flow rate: 40ml/min both sides
He flow

01_He purge

02_CV: (0-3 VRHE) ⇒ -0.493 to 2.507
@ 50, 100, 150 mV/s

03_LSV: 0-3 VRHE @ 50, 100 mV/s

04_CI: 17.421 Ω

05_CC: @ 100 mA sq cm (O_2 before = 0.0563 injection 9)
from injection 10 to
300 mA CC
it is not working with 100 mA sq cm, amplifier overload.
at 5V, don't know the reason.
So i switched it to 225 mA means 75 mA sq cm
⇒ increased the flow to more solves it
So, if we use Repelectrode 41 run g constant current, the voltage shows WE voltage:
from injection 56:
The cell voltage goes to 10V for reaching 300 mA ≈ 100 mA sq cm

Didn't work so have to do it again

20220905_BDD new Ta_Parameters

goal is to use BDD for following observations in a flow cell.
The voltage would be overall cell voltage
liquid samples will be taken after one hour experimental
Parameter needed:
⇒ impact of PH 13, 5, 9, 12) ⇒ Naacetate

TS6-1

20220905_pH5_Naacetate

WE BDD
CE SS
membrane: Nafion
flow catholyte, anolyte: 40 ml/min
Electrolyte volume: 100 ml both catholyte anolyte
Catholyte: 0.5 M Na_2SO_4 pH5: adjusted with H_2SO_4
Anolyte: 1M acetic acid + sodium acetate pH5
He flow: 33.2 ml/min

(two electrode setup, so the potential is cell voltage)

01_He purge

02_CC: 50mA sqcm, 151398mA (O_2 before 0.0768, inj 19)
from injection 20 - 31 1hr

after sample, i took the second sample 41 perform 1hr again.

(sample taken for both)

			Methanol	Methyl acetate
03_CC_50mA sq cm ⇒ 151.398mA	from injection 33-44	1hr	66166, 63589	138, 72248, 67762
		2hr	51926	11600 11930
			103901, 99289	10840
			104067	

20220905_pH5_1M Naacetate_100 mA sq cm

TS6-2

with power supply
same conditions as above
New catholyte/anolyte.
01_He purge 34 ml/min

	Methyl ace	Methanol
1hr	12255	97836
	13020	91489
	13430	91127

02_CC: 100 mA sq cm ⇒ 300 mA by power supply
from injection 10
(O_2 before = 0.0677 injection 9)

pH catholyte 12.4
pH anolyte 4.9

T56-5

20220905-pH3_50 mA/sq cm

WE: RDD
CE: SS
two electrode divided cell with Nafion membrane
of 100 ml catholyte, 100 ml anolyte 40 ml/min
0.5M Na_2SO_4 1M Acetate/acid acid
pH 5 pH 3

01_ He purge ... after purging overnight there is still O_2

02_CC 50 mA/sq cm = 151.398 mA
for 1hr
O_2 before = 0.0847 injection 183
from injection no 184

very high voltage ~ 30V, membrane destroyed
samples collected.

	T_{R_1}		
Methyl	92242	48607	
Methanol	153424	15606	
T_{R_2} Methyl acetate	45633	9484	
Methanol	164188	16879	

T56-4

20220906-pH9_50 mA/sq cm

same conditions
just anolyte pH change
(some anolyte lost due to connection ~ 5-10 ml) so I select 80 ml.

01_He purge.

02_CC = 50 mA/sq cm or 151.3 mA
O_2 before 0.0711, injection 9
from injection 10

	pH 6.2 anolyte		Catholyte 12.4
Methanol	~~8669~~ 47782	48808	53142
Methyl acetate	8994	9998	8590
Unknown could be ethanol	23208	24752	23484

20220907-pH12_50 mA/sq cm

same conditions
just analyte pH change
0
01_ He purge 32.4 ml/min
02_CC 50 mA/cm² ⇒ 151.398 mA O_2 before = 0.0756
60 mins from injection 9 to

samples taken and analyzed.

T56-6

20220907-pH 9_50 mA/sq cm repeat.

It was weird because I found that there was ethanol in liquid sample, could be by contamination, so repeating

* Also it is important to be careful while using voltcraft as the data acquisition only considers one point after decimal.

01_He purge : 32.4 ml/min

02_CC : 151.398 mA or 50 mA/sq cm O_2 before 0.0711, inj 9)
from injection 10 to

T57

20220908

Because during progress meeting on 20220908 with Guido & Bastian, we observed that with BDD, the FE is not reaching to 100% for CO_2 so this experiment is meant to confirm my hypothesis is that CO_2 dissolved better with flow recirculation by forced convection. Bastian also mentioned that I should do an experiment with Pt foil in batch

WE: BDD : 7mm area: 0.3875
CE: Pt/Ti
RE: Ag/AgCl
Velectrolyte = 25ml

01_ He purge
02_CV: 0 - 3 VRHE ⇒ -0.492 to 2.507 $V_{Ag/AgCl}$
n_c=2: 50 mV/s, 100 mV/s, 150 mV/s
03_LSV: 0 - 3 VRHE @ 100 mV/s
04_Cl : 30.720 Ω
05_CC: 25 mA/cm² : 9.6875 mA
injecte O_2 before 0.0404 from injection 15 to 114

so the CO_2 FE is still the same around 40%. ⇒ reason: the solubility of CO_2 is higher in organic solvent than water.

Prof Paper: Critical assesment of CO_2 solubility in volatile solvents at 298.15K

Methyl acetate is one of the best compand for CO_2 solubility because it is rich in carbonyl and or ether groups that favours lewis acid/lewis base interaction with CO_2 and solvent steagth decreases with increasing molar mass.

Second aspect: CO_2 is readily soluble in ...than

06_CV : 0-3VRHE
07_LSV : 0-3VRHE
08_Cl : 32.625 Ω

T58

20220909 1M Pt Condias flow
Nacetate pH5 both sides

WE: Pt foil
CE: SS
Nafion membrane
Electrolyte 1M Nacetate pH5 on both sides
flow: 40 ml/min
01_ He purge. 34ml/min during electrolysis
02_CC: 15 ~~13.98 mA 10~~ 25 mA sq/cm = 75 mA
O_2 before = 0.0746, inject 17 from injection 18
time: the for the 5 min for liquid... 65 mins
liquid samples to both catholyte & anolyte for GC-HS

Liquid Anode no liquid product at all

pH stays 5 after electrolysis both sides

T59

20220910 1M Pt Condias flow
Nacetate pH5 both side catholyte gas analysis

WE: Pt foil
CE: SS
Nafion membrane: catodic side gas analysis
01_ He purge
03_CC: 45 mA ~~RSOmA~~ sq cm (O_2 before = 0.0838
ethane before = 0.0067

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20220913_H_2O_2_Structured BDD

20220913_BDD_no pillar_H_2O_2_2H_high doping

BBD high density high doped without pillar

BBD area 12mm × 12mm = 1.44 cm²

Catholyte: 100 ml 1M K_2CO_3

Anolyte: 100 ml 1M K_2CO_3

flow rate: 100 ml/min : 229 rpm both sides

Membrane Nafion

Electrolysis time: 1.5 hrs

Sampling rate: 15 mins : 8 samples.

01_ He purge: 32.8 ml/m.

02_ CC: at 100 mA/sq cm, FE to O_2: 82-90%

from injection 3 to injection 20 (O_2 before = 0.0674, inj 2), cell V reaches to 4V

Sample 1	-1.5-2 ml	Sample 4	Sample 7
UV vis	0	UV	UV
Strip	0	Strip	Strip
pH	12	pH	pH
Sample 2	-3.0-2 ml	Sample 5	Sample 8
UV	0	UV	UV
Strip	0	Strip	Strip
PH	12	PH	pH
Sample 3	-1hr 5 ml	Sample 6	
UV		UV	
strip	0	strip	
pH	12	pH	

no detectable

important point

with carbon paper they mentioned bicarbonates are essential to achieve selectivity for water oxidation to H_2O_2

in Kasper Liler paper they use anion exchange membrane.

After 1 h 30 mins no H_2O_2 detected, high FE to O_2, Bastian recommend to first try with low flow 10x, ; also suggested to try with low current density because Fernanda did it and she get high FE at low current density 5 mA/cm², at high current density (100 mA/cm²) she gets around 3-5% FE.

90 ml solution

at 40 ml/min

03_ CC_ 40 ml/min

at 100 mA/sq cm

O_2 before = 0.0728 from injection 25

from injection 26 to 30 mins, cell voltage reaching above 4.7-5

26-31

no H_2O_2 detectable, only FE to OER 80%.

↳ no sample taken

at 40 ml/min

04_ CC_ 50 mA/sq/cm²

O_2 before 0.0633 injection 37

from injection 38 to 44 35 mins

the cell V goes to 4V.

at 40 ml

0

T61

Parent folder

20220914_BDD divided flow with diff conc
Hofer moest

20220914_0.1M Nacetate pH5

He flow = 33 ml/min
WE: BDD full disk 3cm²
CE: SS
divided electrode: Nafion 324
Catholyte: anolyte: 100 ml each; 0.1M Nacetate pH5, 40 ml/min

01_ He purge

02_ CC: 50 mA ugem = 151.398 mA
O_2 before = 0.0876, injection 14 → high because of low concentration
from injection 15

the cell voltage reaches to =12V, so it would not be possible to work with it, i added 1ml H_2SO_4 0.5m & 0.5M Na_2SO_4 3 ml in both anolyte & catholyte.
so then i will use power supply
O_2 before = 0.0869 ilk 23
from inj 24 - 35
PH catholyte after:
PH anolyte after:
Two liquid sample
① → T61-1: no dilute because it is 0.1M acetate
② T61-2: same 10 times dilute

H_2O_2 = skip 3mg/L

0.000081 mol/L

UV vis - = 0.030
interesting

T62

20220914_0.5M Nacetate pH5

He flow = 33 ml/min
BDD full disk 3cm²
WE:
CE: SS
Divided electrolyte: Nafion 324
Catholyte anolyte - 100 ml each, 0.5M Nacetate, pH5, 40 ml/min
01_ He purge:

02_CC: 151.398 mA
O_2 before: 0.0844 injection 5
from injection 6 to 16

T62-1 4 times diluted

20220705_ Impact of Cations full
↳ in Macetak
↳ 20220927_ pH4_ 25 mA/cm²

WE: Pt foil double side area = 1 cm²
CE: Pt / Ti mesh
RE: Ag/AgCl

Electrolyte volume = 20 ml
Batch cell pH. 4

01_ He purge = 331 ml/min

02_ CV (0 - 3 VRHE) → -0.4336 VAg/AgCl to 2.5664
@ 50, 100, 150 mV/s ; nc = 2

03_ LSV 0 - 3 VRHE @ 100 mV/s

04_ CI = 85.83 Ω

05_ CC @ 25mA/cm² → 25 mA
O_2 before = 0.0421 , injection 7
from injection 8 to

06_ CV: 0-3 VRHE → -0.4336 VAg/AgCl to 2.5664
@ 50, 100, 150 mV/s nc = 2

07_ LSV 0 - 3 VRHE

08_ CI Ω

20220922 diff and doped BDD flow 100 mA/cm²

WE: low doped BDD : 3 cm² area
CE: Stainless steel
Divided membrane Nafion
Catholyte, anolyte 100 ml each flowing

O1_ the purge 33.2 ml/min

last time, I tried with the same electrode but the resistance was too high
Reason, the BDD on Si are very thick 40 µ it is difficult to make side contact and thereby the resistance was very high. So this time, I made contact with copper tape to the back side of electrode and at 100 mA/cm² ≈ 300 mA the potential is stable and within the range of potentiostat.

02_CC

O_2 before : 0.0832 injection 7
from injection 8 to 19

- liquid sample is stored in fridge because I don't have the slift for GC-FID/HS
- also I want to see if methanol and methyl acetate are stable.

cathode turning black yellow (image on phone)

03_CC BDD cleaning in 0.5 M H_2SO_4 at 25 mA/cm² for 25 min

20220928 diff doped BDD flow at 100 mA/cm²

The experiment was wrong because of two reason

Reason 1: the current density was wrong, because I applied 25 mA/cm² instead of 300 mA (100 mA/cm²)

Reason 2: the copper solution FE reaches to 80 - 100% and no CO_2 detected, so the electrolysation did not take place. And the O_2 must be coming from leakage of volume.

Based on both reason, I assume that this experiment is wrong for 100 mA/cm²

→ I did sent sample for Raman analysis for 100 mA/cm² for Raman to supplied so by the end, the results which I will get on 29-2-2022 I must consider only the low doped BDD Raman of this day.

→ The next time, I need to sent try high doped at 100 mA/cm² electrolysis and send it for Raman.

T67

2022 0929 Aqueous phase bio-oil
BDD at 25 mA cm^{-2} → pH5

1 cm^2 : L x W
1 cm = 0.986
2 / 0.986 = 10.41

area covered
length 2 cm → area = 2 cm^2
width 1 cm

45 ml aqueous phase pyrolysis oil 50 wt%
PH = 2.5
PH adjusted with 1M NaOH to pH5 = 15 ml NaOH
for experiment i used 45 ml.

{SAP_1 are aqueous phase pyrolysis oil with pH5 for NMR
SAP_2

Undivided three electrode
stirred
purged with Helium.
He flow
CE: Pt/Ti
WE: BDD 2 cm^2
RE: Hg/HgCl

01_ He purge: ml/min

02_ CV: -1.063 V RHE (-0.492 to 2.307)
nc: 50, 100, 150 mV/s

03_ 25V: -0.3 V RHE (-0.492 to 2.307)
@ 100 mV/s
04_ CI: 35.876 Ω

So i changed the range because i want to see how it good with HER

05_CC @ 25 mA sq cm = 50 mA
t = 90 mins
from injection 4 to 0. before 0.0613 injection 3 (time start 45 min)

so many higher fractions of hydrocarbons which are not calculated for injection 4-5 show OER
no OER, also so much heavier hardocarbons so i am not doing gas analysis, only liquid samples.

→ those peaks on GC for heavier hydrocarbon are the contaminents of pyrolysis oil which are volatile, i think so.....

Sample after electrolysis 90 min
SAP_3, SAP 4, samples becomes more transparent. pH after 4.9

PE at 25 mA cm^{-2} - PH5 T68

Undivided cell three electrode stirred
purged with Helium
CE: Pt/Ti
WE: Pt foil double sided 2 cm^2
RE: Hg/Ag Cl.

B

Parent folder 20221013_BDD Batch after GC HS F2 (T69)

So the sensitivity of GC-FID headspace was changed for some reason, I so i calibrated the GC-F.I.D HS now with discussion with Bastian, we want to observe few thing:

→ first we will do the test in Batch at different current density then other parameters such as pH, effect of supporting electrolyte. then we will do the flow undivided with ~~Batch~~ later divided batch / flow.

20221013_BDD 2cm² 1M Na acetate_pH5

01_ He flow = ml/min electrolyte = 45ml volume

20221013_BDD 2cm²_1M NaAc_pH5 (T70)

02_ CV: (0 - 3 VRHE) ⇒ -0.4926 to 2.5074 VAg/AgCl
nc @1: 50, 100, 150 mV/s

03_ LSV: @ 100 mV/s ⇒ 0 - 3 VRHE

04_ CI: 18.75 Ω

05_ CC: 25 mA/cm² ⇒ 50 mA for ~~65~~ 60 mins
stirring on: turn on the C.E potential record option. : O_2 before = 0.0433 injection 16
from injection 17 to .

06_ CV (0-3VRHE) ⇒ -0.4926 to 2.5074 VAg/AgCl
nc = 1, 50, 100, 150 mV/s

07_ LSV @ 100mV/s

08_ CI = 15.542

Liquid sample collected

Methyl acetate: 9881, 9243 8637

Methanol: 39242 40233 40233

BDD cleaning in 0.5M H_2SO_4 @ 25 mA/cm² for 20 mins

(Due to calculation error, the real current density is 12.26 mA/sq.cm

There was a problem with BDD/Ti long length electrode in CVs and it was visible that some impurities like are oxidizing

So now, change the electrode to the disk, covered back side with kapton on BDD/Si no CVs are perfect.

T10

20221013_BDD 2.6cm² 1M Nacetate_pH5

due to calculation error, the real current density is 12.26415 mA/cm²

He flow: 31 ml/min
Electrolyte volume = 45 ml
WE: BDD/Si ⇒ 26 mm ⇒ 2.6 cm²
CE: Pt/Ti

01_ He purge

02_ CV: 0-3 VRHE ⇒ -0.4926 to 2.5074 VAg/AgCl
nc = 3 @ 50, 100, 150 mV/s

03_ LSV: (0-3 VRHE) ⇒ -0.4926 to 2.5074 VAg/AgCl
@ 100 mV/s

04_ CI: 11.589 Ω

05_ CC: 25 mA/cm² ⇒ 65 mA/cm² (wrong 12.26415 mA/cm²)
for 65 mins. O₂ before = 0.0554, injection 8
from injection no 9 to
pH after = 5

06_ CV: 0-3 VRHE

07_ LSV

08_ CI = 9.704

	Methyl acetate		
	11087	11327	10797
	Methanol		
	50158	52096	49531

20221017_Graphite 2.56 cm²_1M Nacetate pH5

due to calculation error, the real current density is 12.26415 mA/cm²

He flow: ml/min
Electrolyte volume = 45 ml
WE: Graphite disk 2.6 cm²
CE: Pt/Ti

01_ He purge: 31.8 ml/min

02_ CV: 0-3 VRHE ⇒ -0.4926 to 2.5074 VAg/AgCl
@ 50, 100, 150 mV/s nc = 1

03_ LSV: 0-3 VRHE : -0.4926 to 2.5074 VAg/AgCl

04_ CI: = 5.503 Ω

05_ CC: 25 mA/cm² (wrong: actual: 12.26415) ⇒ 65 mA for 65 mins
O₂ before = 0.0810 injection 10
from injection 11 to
pH after
- stirring

06_ CV: 0-3 VRHE ⇒ -0.4926 to 2.5074 VAg/AgCl
@ 50, 100, 150 mV/s nc. 1

07_ LSV: 0-3 VRHE : -0.4926 to 2.5074

08_ CI = 5.351

Liquid samples taken.

↓ Wrong area by mistake so the current density also wrong.

The real area of full disk is 5.3 cm²

Methyl acetate:	52193	50744	54127
Methanol	44546	44353	47407

T72 2022 1017_BDD undivided - T

Volume of electrolyte = 100 ml
flow rate = 40 ml/min
BDD on tantalum = 20 mm² active area (3.14 cm²)

CE: stainless steel

O2_He purge = 31.9 ml/min

O2_CC = 25 mA/cm² ⇒ 78.5 mA
for 65 min : O_2 before = 0.0804 , injection 4
from injection 5 to 18 .

pH after =
H_2O_2 =
because i see the O_2 evolution

Sample in GC { Methanol : 9301 8637 11490
methyl acetate : - - -

T73 2022 1017 BDD_Graphite 26mm 1M Naacetate undivided batch real current density 25 mA/cm²

volume of electrolyte = 45 ml
Graphite disk 2.6 cm² dia ⇒ area = 5.3066 cm²

CV's & all other data will be used of experiment T71

O2_CC = 25 mA/cm² = 132.665 mA
for 65 mins : O_2 before = 0.0854 , injection 3
from injection 4 to 16
pH 5.2

Liquid product:
⇒ methyl acetate ⇒ 71662 , 74418 , 72117
⇒ methanol ⇒ 66004 , 65223 , 65638

T74 2022 1018 BDD 26mm full disk Batch undivided real current density

He flow: 30.5 ml/min
Electrolyte volume = 47 ml
BDD full disk area = 5.3066 cm²
pH 5
CV & all other data will be used of experiment T70

O2_CC = 25 mA/cm² or 132.665 mA
for 65 mins, O_2 before = 0.0619 injection 13
from injection 14 to .

Stopped because holder was disconnected

continue after:

O_2 before = 0.0774 injection 45
from injection 46 to

Methyl acetate : 14892 16680 16785
Methanol : 86163 89614 82628

(75) 2022 1018_ Graphite flow 25mA/cm² undivided

Volume of electrolyte : 100ml
flow rate : 40ml/min
Undivided cell
Graphite active area : 3.14cm²
/CE: stainless steel

01_ He purge

02 CC : 78.5 mA @ 25mA/cm² = 65mins
O_2 before. 0.0808 injection 3
from injection 4 to 16

pH after :

Sample taken for the GC

Methyl acetate 34217 32456 38171
Methanol ~~8616~~ 29588 29230 32780

(During the data analysis of liquid product i found that : applied current was much higher, 132.66 mA current making current density about >40 mA/cm²

(T-16) 20221019_ Graphite flow 25 mA/cm² undivided

Volume of electrolyte : 100ml
flow rate : 40 ml/min
Undivided cell
Graphite active area : 3.14cm²
CE: stainless steel

01_ He purge

02_CC : 78.5 mA @ 25 mA/cm² @ 65 mins
O_2 before, 0.0854, injection : 15
from injection 16 to

Methyl acetate - 22099, 21729, 22366
Methanol - 19105, 20464, 18889

T78

Parent folder : 20221020_BDD_Electrodeposition_Kolbe

20221020_BDD_ED-1

Strategy:

Electrodeposition → CV in 1M H_2SO_4 0.05 to 1.2 V_{RHE} ✓

1hr electrolysis : 100 mV/s CV in acetate + Cl + 25 $\frac{mA}{cm^2}$ CC + CV + CV ✓

① Blank CV in 1M H_2SO_4 n_c (3) ✓
0.05 to 1.2 V_{RHE} → −0.2066 to 0.9434 ✓

② Electrodeposition ED1 ✓

③ Blank CVED in 1M H_2SO_4 n_c (3) = 100 mV/s ✓

④ CV in acetate 100 mV/sec ✓ : 0.05-3 V_{RHE}, n_c = 3
+Cl = 6.64

⑤ CC @ 25 mA/cm^2 (electrolyte volume, 50ml O_2 before 0.074 inj: 5)
from injection 6 to → current = 132.665 mA ✓ | 1M H_2SO_4
↓ 5 mins | pH 1

⑥ CV in acetate + Cl = 7.248 Ω | Methyl: 12714, 11104, 13938

⑦ CV in H_2SO_4. | Methanol: 46660, 49025, 61120

T79

20221020_BDD_ED3

01_ Blank CV in 1M H_2SO_4 = 0.05 − 1.2 V_{RHE}

02_ Electrodeposition ED-3 → 15 mins

03 - CV after ED in H_2SO_4

04_ CV after ED in acetate : Cl = 4.770

05_ CC @ 25 mA/cm^2 electrolyte vol = 50ml

O_2 before = 0.0540, injection 3

from injection 4 to injection 12.

06_ CV acetate after CC
+Cl = 6.348 Ω

07 CV in H_2SO_4

→ cleaning cycles.

Methyl: 8985, 8890, 10889

Methanol: 28257, 30470, 33224

T80 — 20221020_BDD_ED-4

02_ED4
03_CV after ED: H_2SO_4
04_CV acetate after ED
Cl: 6485Ω
O_2 before = 0.0697 _ inject 6

05_CC
inj 7:
06_CV acetate: CE — Methyl acet 13492, 13004, 11465; Methanol 52307, 51083, 50696
07_CV H_2SO_4

T81 — 20221020_BDD_ED-6- 30~~mins~~.

02_ED6 : 15 mins.
03_CV after ED6 — 1M H_2SO_4
04_CV acetate: Cl: 4.614 — O_2 before = 0.0636 ; inj 4
05_CC:
from injection 5 to 13
06 CV after CC — Cl: 5.701
07 CV H_2SO_4 — Methyl: 10489, 11798, 12286; Methanol: 42353, 41359, 44793

T82 — 20221020_BDD_ED3_30 mins

02_ED3_30 - 30 min
03_CV after ED: — 1M H_2SO_4
04_CV acetate — Cl: ~~4.614~~ 5.096
O_2 before 0.0765 injection 4
05_CC
from inj 5
↳ no ethane ; how. — No methanol; No methyl acetate
06_CV acetate

T83 — 20221026_Graphite Divided flow at 40 — ↳ 20221026_at 25 mA/cm² 40 ml/min divided Nafion 324 acetate both sides pH5

WE graphite : area: 3.14cm²
CE: S.S
flow: 40 ml/min both side
He flow 32.6 ml/min
Electrolyte volume : 100 ml each both sides

01_He ~~purge~~ 32.6 ml/min

02_CC: 25 mA/cm² : 78.5 mA for 65~~mins~~ (60 mins)
O_2 before = ~~0.899~~ 0.807, injection 1 to 6
from injection 6 to 19

{ Methyl acet : 20379, 20604, 19235
{ Methanol : 19830, 21173, 19675

T84 — 20221026_at 50 mA/cm 40 ml/min divided Nafion 324 acetate both sides pH5

WE graphite : area = 3.14cm²
CE: S.S
flow = 40 ml/min both sides
He flow = 32.6 ml/min
electrolyte volume = 100 ml each

(for catholyte i used the same electrolyte because pH did not change ≈ 15)

01_He purge : 32.6 ml/min

02_CC = 50 mA/cm² = 157 mA — 65 mins
O_2 before = 0.0807 injection 6
from injection 7 to 19

{ Methyl acetate 44327, 43980, 38790
{ Methanol 39959, 37547, 37842

T85

20221027_at 75 mA/sq.cm 40 ml/min divided Nafion 324 acetate both sides pH5

WE: graphite 3.14 cm²
CE: SS
flow: 40 ml/min
He flow
Electrolyte = 100 ml each on both side. (catholyte was used from T83, T84)

01_He purge = 31.6 ml/min

Methyl acetate: 62827, 64489, 61626
Methanol: 53269, 53511, 53390

02_CC = 75 mA/cm² ⇒ 235.5 mA for 65 mins

O₂ before = 0.0870 injection 11
from injection 12 to 24
liquid samples to GC

Catholyte pH turned 5.5 after experiment

20221027 at 100 mA 40 ml/min divided
Nafion 324 acetate both sides pH5

T86

WE = graphite 3.14 cm²
CE: SS
flow = 40 ml/min
He flow = 31.6 ml/min
Electrolyte = 100 ml acetate (Na) both sides (fresh catholyte)

01_He purge = 31.6 ml/min

Methyl acet: 50651, 49143, 47254
Methanol: 39220, 39456, 36573

02_CC_ 100 mA/cm² = 314 mA 67 mins
O₂ before = 0.0862 injection 3
from injection 4
(I started the experiment with flow rate (60 ml/min), the cell voltage was 6V, but i changed the flow rate back to 40 ml, the cell voltage went to 8V
(Graphite particles in electrolyte
Graphite surface looks like becoming paste)

T87

20221027 at 50 mA/sq.cm 40 ml/min divided Nafion 324 acetate both sides pH5 w/o GC, He

I want to try without gas analysis and purging to see if it affect the FE, further more it will give me freedom to work with GC and hence the experiments will be quick

02_CC = 157 mA or 50 mA/cm²
65 mins
Methyl acetate = 48282, 48110
Methanol = 41657, 40922

T88

20221028_at 25 mA sq/cm 40 ml/min divided Nafion 324 acetate both sides pH9 w/o GC He

02_CC = 65 min 25 mA/cm² ⇒ 7850 mA

	After	Before
pH catholyte =	5.6	5.5
pH anolyte =	6.7	9

Methyl acetate = 19791, 18446, 18233
Methanol = 16786, 16877, 16749

T89

20221028_at 25 mA/sq.cm 40 ml/min divided Nafion 324 acetate both sides pH 12 w/o 12

02_CC_ 65 mins : 25 mA/cm² ⇒ 78.50 mA

	Before	After
pH catholyte =	5.6	5.8
pH anolyte	12	6.7

Methyl acetate = 21246, 22895, 20438
Methanol = 18991, 19239, 16122

Goal: BDD with different density at different pH

Expt:
Con:
rpm:
Plot:

BDD- 75
BDD- 50
BDD- 75
BDD- 100
BDD- 150
BDD- 200
BDD- 75

→ pending

Single & multipass
1, 2, 3, 4, 5, 10, 20, 50, 100

Effect of pH
@ 50 mA/cm² = 3, 5, 9, 12

Effect of flow rate:
10 ml, 20 ml, 40 ml, 60 ml, 100 ml, 150 ml, 200 ml/min
250 ml/min

PHS: 50 mA/cm², counter flow similar

Effect of counter flow on cell voltage, no liquid products & only electrochemistry.

20221031 pump calibration

rpm	ml/min
10	7.594
40	30.150
80	61.855
100	77.922
200	157.891
300	240
350	285.7143

ml/min to rpm: divide ml/min by 0.8041 = rpm

T90

20221031_10ml/min_50mA/sq cm_BDD_1M Na acetate

WE: BDD, CE = SS
3.14 cm²
Divided = 10 ml/min both sides = 12.436 rpm
100 ml catholyte: 100 ml anolyte each at 10 ml/min

O2_CC: 50 mA/cm² ⇒ 157 mA @ 60 min (w/o QC)
1 hr

Sample to analyze for liner.
Methyl = 10947, 10369, 8992
methanol = 41538, 42122, 41144

T91

20221031_30ml/min_50mA/sq cm_BDD_1M Na acetate

Same conditions as above except flow rate
30 ml/min both sides = 37.3 rpm

O2_CC: 50 mA/cm² ⇒ 157 mA @ 60 min

Methyl = 7763, 8805, 10355
Methanol = 38769, 40810, 43018

T92 20221031_60ml/min_50mAsqcm_BDD_1M Naacetate

→ same conditions like before except flow rate both sides (Voltage both outlier)

60ml/min = 74.6 rpm

1hr @ 50mAsqcm ⇒ 157mA

Methyl · 10364, 9016

Methanol · 47600, 38750 44447

T93 20221101_100ml/min_50mAsqcm_BDD_1M Naacetate

→ same condition like before except flow rate both sides (Voltage outlier)

100ml/min: 124.3626 rpm

1hr @ 50mAsqcm ⇒ 157mA

(30 min) Methyl · 9380

Methanol · 47926

T94 20221101_200ml/min_50mAsqcm BDD_1M Naacetate

→ same conditions like before except flow rate both sides

200ml/min = 248.72 rpm

1hr @ 50mAsqcm, 157mA

Methyl	9447
Methanol	48647

T95 20221101_250ml/min_50mAsqcm BDD_1M Naacetate

→ same condition like before except flow rate both sides

~~280~~ 250 ml/min = 348.215 rpm

= 310 rpm

Methyl ·	9467	10421
Methanol ·	50091	47021

T96 20221101_100ml/min_50mAsqcm_BDD 1M Naacetate_rep

→ Because the T93 voltage seems to be outlier so, i am reproducing it again

same condition like before except flow rate (similar to T93) both sides 100ml/min ⇒ 124.36 rpm

1hr @ 50mAsqcm ⇒ 157mA

Methyl 10347

Methanol 48981

T97 20221101_

Effect of cell voltage by CE flow_BDD_1M Naacetate at 50mA/cm²

time (mins) = 15 mins

WE	CE	Run
① 10 (rpm 12.43)	30 (37.3)	T97_1 ✓
	60 (74.6)	T97_2 ✓
	100 (124.36)	T97_3 ✓
	200 (248.72)	T97_4 ✓
	250 (348.2)	T97_5 ✓
② 30 (37.3)	10 (12.43)	T97_6 ✓
	60 (74.6)	T97_7 ✓
	100 (124.36)	T97_8 ✓
	200 (248.2)	T97_9 ✓
	250 (348.2)	T97_10 ✓
③ 60 (74.6)	10 (12.43)	T97_11 ✓
	30 (37.3)	T97_12 ✓
	100 (124.36)	T97_13 ✓
	200 (248.72)	T97_14 ✓
	250 (348.2)	T97_15 ✓
④ 100 (124.36)	10 (12.43)	T97_16 ✓
	30 (37.3)	T97_17 ✓
	60ml outlier (missing)	T97_17a ✓
	200 (248.72)	T97_18 ✓
	250 (348.2)	T97_19
⑤ 200 (348.2)	10 (12.43)	T97_20
	30 (37.3)	T97_21
	60 (74.6)	T97_22
	100 (124.36)	T97_23
	250 (348.2)	T97_24
⑥ 250	10 (12.43)	T97_25
	30 (37.3)	T97_26
	60 (74.6)	T97_27
	100 (124.36)	T97_28
	200 (348.2)	T97_29

T98 → Reproduce

* After analyzing the data of TQD-92, I found that the results are not reproducible because I am getting FE very low to methanol and methyl acetate as compared to before where I get around 100% FE

→

* Reproduce it again in flow cell with new membrane

What to do

pH, 5, 9, 12, 3

current density, 25, 50, 75, 100, 250, 200

flow rate: 10, 20, 50, 100, 200, 250 ml/min

single pass multipass

Cations impact, supporting electrolyte impact.

2022 11 03_BDD_25 mA/cm²_pH5_100 ml/min

Electrolyte volume. 40ml

He purge

WE area: 3.14 cm²

SS count electrode BDD working electrode

Catholyte, anolyte 40ml each 1M Nacetate pH5

OL He purge 33 ml/min

02_CC: 25 mA/cm² = 78.5 mA

O_2 before 0.0686 inj 10 65 minutes

from injection 11 to

Methyl	5896	4084
Methanol	21263	20913

T99 2022 11 03_BDD_25 mA/cm²_pH5_100 ml/min_12 hrs

Similar situation as above except the electrolysis time

1hr OCV + 12 hr CC

25 mA/cm² = 785 mA for 12hr

injection 13 O_2 before 0.0698

Methanol	173622	173205
Methyl	1711	2792

Impact of cations continuation

T100 2022 11 16_1M Nacetate pH4_25 mA/cm²

pH 3.7

1M acetic acid + Nacetate

V: 50 ml

He flow: 30 ml/min

Pt foil one sided = 1 cm² second side covered with Kapton

RE: Ag/AgCl

CE: Pt/Ti

01_He purge: 33 ml/min

02_CV: (0-3 VRHE) : pH: 3.8 -0.3718 to 2.5782 -0.4218

nc = 3 @ 50, 100, 150 mV/s

03_CI: 44.612

04_CC: at 25 mA/cm² ⇒ 25 mA

O_2 before: 0.0413, injection 7

from injection to

pH same 3.8

So the CV conditions same

05_CV: 0-3 VRHE

06_LSV: 0-3 VRHE @ 100 mV/sec

07_CI = 33.638 Ω

T101 2022 11 16_1M Nacetate pH5_25 mA/cm²

pH 4.8

1M acetic acid + Nacetate

V: 50 ml

He flow 30 ml/min

02_ CV (0-3 VRHE) : pH 4.8: -0.481 to 2.519 V

03_ LSV: 0-3 VRHE

04_ CI: 16.220 Ω

05_OCV 1hr

06_ CP 25 mA/cm² 90 min from injection 7: O_2 before 0.0429

07_ CV

08_ LSV

09_ CI = 14.455

T102

20221117_Liacetate 1M_pH 2.5_25 mA/sq cm

desired pH was 3 but the CI was too high, i added one drop of 1M H_2SO_4, pH was 2.6., so CVs are accordingly

01_ He purge
02_ CV (0- 3VRHE) → - 0.35V to 2.649 VAg/AgCl
03_ LSV
04_ CI : 264439 Ω
06_ CC: 25 mA/cm² 60 min Velocity = 50 ml
0. before. 00513, injection 5.
from injection 6
06_ CV
07_ LSV
08_ CI : 275.210

T103

20221117_Ceacetate 1M pH 2.5_25 mA/sq cm

actual pH 2.6

Same conditions

02_CV (0-3VRHE) → -0.35V to 2.649 VAg/AgCl
03_LSV
04_ CI = 168.344 Ω
05_ CC → 25 mA/cm² → 0. before = 0.0452 injection 26
From injection 27 to ..

06_ CV
07_LSV
08_CI
weird that the OER conditions, Ceacetate performing worst than Li
09_CC from injection 44 to 49 as it is reproducible.

To do.

EIS:
1M Naacetate pH 5, Pt foil
1M Kacetate pH 5, Pt foil
1M Ce acetate pH 5
1M Li acetate.

Li acetate

→ Kolbe onset potential changes as function of pH → with RRDE in function with cations.

Points to confirm:
- some papers suggest that the influence of cations increases with pH
- OER activity with different cations at the low pH has no difference in activity.
but the difference is more prominent at high pH.

20221121_ impedance.

N acetate pH5

0 VRHE
1 VRHE
1.5 VRHE
2 VRHE → perfect semicircle
2.5 VRHE
3 VRHE → not working due to nitrate bubble

} hold for 1 min
from 100KHz to 500 mHz
Nd = 6
amplitude = 10 mV

15mA GEIS
10 mA GEIS
25 mA GEIS

} → same method. → not successful
amplitude 10 mA → not successful.

20221121_ impedance 1M K acetate pH5 in impact of cath folde
2VRHE : 100KHz to 500mHz amplitude 10mV
1min hold.

20221121_ impedance 1M Cs acetate pH5
impact of catin folde
2VRHE : same conditions

20221121_ impedance 1M Li acetate pH5

20221121_ impedance 1M Na acetate pH5
2VRHE_Repeat

20221121_ impedance 1M Na pH9

impact of cation folder

T104 20221122 - 1M Na acetate pH 3 25 mA/cm²

He flow 32.3
Velectrolyte : 40 ml

01_ He
02_CV: (0-3 V RHE) -0.375 to 2.625
nc = 1 @ 50, 100, 150 mV/s
03_LSV 0-3 V RHE @ 100 mV/s
04_ CL
05_ OCV, 30 min
06_ CP: 50 mA ⇒ 25 mA/cm² = 65 min
O₂ before 0.0619

07_ CV
08_ LSV
09_ CL = 118

T105 20221122_1M Na acetate pH3 25 mA/cm²

same conditions as above
CL: 94.44.

O₂ before - injection 6.
injection 7.
CL: 93.595

weird results

T106 20221122_ 1M Li acetate pH3 at 25 mA/cm²

same conditions
Velectrolyte 40 ml
02_CV: 0-3 V RHE ⇒ -0.375 to 2.625
nc = 1 @ 50, 100, 150 mV/s
03 LSV: 0-3 V RHE
04_CL:
05_ 227.704 Ω = O₂ before 0.0598
O₂
06_CV
07_LSV 1
08_CL 123.1975 Ω

compare this with previous Na/K acetate pH3 from presentation

T107 20221122_ 1M Li acetate pH3 at 25 mA/cm²

same conditions
Velectrolyte 40 ml

CL, 101
for liquid analysis

T108: liquid product Li acetate pH 3 10 mA/cm²

T109

2022 11 28_ 1M Li acetate pH 12_ 50 mA/cm²

He flow = 30.8

Electrolyte volume = 47 ml

OCV : 30 mins
02_CV : -0.906V to 2.094V (0 - 3V RHE)
03_LSV @ 100 mV/s
04_EI : Ω
05_CC 50 mA/cm², 100 mA
for 1 hr 5 mins
06_CV : -0.906 V to 2.094V (0-3V RHE) nc = 50, 100, 150
07_LSV : @100 mV/s 0 - 3V RHE
08_CI : 9608 Ω (liquid sampling)

T110 2022 11 28_ 1M Li acetate pH 12 - 50 mA/cm²

He flow : 30.8 ml/min
Electrolyte volume : 45 ml

02_CV: -0.906 to 2.094V (0-3V RHE) ⇒ nc = 50, 100, 150
03-LSV: (0- 3V RHE) @100 mV/s

04_CI = 8.940 Ω
05_CC 20 mA ⇒ 10 mA/cm² O₂ before = 0.0567
for 1 hr 5 mins
06_CV : 0-3 V RHE
07_LSV 0-3 V RHE
08_CI. 8.90 Ω

No liquid product found. OER 7801. suspect

T107

2022 11 28 pH 5 10mA cm⁻²

He flow
electrolyte volume = 45 ml

O₂ before = 0.0538 injection 8.

CI = 11.385 Ω

flow cell

video 1	10 ml	- 12.436	Lost
2	20 ml	24.872	
3	40 ml	50	
4	60 ml	74.6175	
5	100 ml	- 124.36	

T112 V 20221201- impedence repeat Li acetate

2 VRHE

20221201- pH4p - 1M Li acetate
40 mL- 1L

01_CV: 0-3 VRHE ⇒ -0.906 to 2.094 V vs Ag/AgCl
03_LSV: 0-3 VRHE
04_Cl: 12.705 Ω
05_CC: 10 mA/cm² ⇒ 20 mA
for 1hr 5 mins
O_2 before = 0.0606 injection 5

	injection	6 - 19.
measured:	7949	156
	8768	14121
	7113	55

Cl: 12.810

T113 20221201- Proof kolbe at 2VRHE Naacetate

To check if we get kolbe at such condition

O_2 before.

at 2VRHE pH5 ⇒ no kolbe. 1- <0.5- 1mA
⇒ 1.507 V vs Ag/AgCl.

BDD experiment for Green chemistry

effect of flow, 20, 40, 60, 100, 200
effect of current density, 25, 50, 100, 200
effect of current density, 25, 50, 100, 200
effect of pH, 3, 5, 9, 12 3, 4, 5, 9, 12,

Single pass	pH5	flow rate
1		20, 40, 60, 100
2		20, 40, 60, 100
3		20, 40, 60, 100
4		20, 40, 60, 100
6		20, 40, 60, 100
10		
20		20, 40, 60, 100
30		20, 40, 60, 100
100		

Effect of pillar vs non pillar
↳ Effect of counter electrode
↳ Effect of counter flow.
↳ cost effective analysis

Conversion single pass
1. 20, 40, 60, 100, 200
2. 20, 40, 60, 100, 400
10, V

2VRHE

1mA/cm² = 1/2 cm²
@ 1×2 = 2

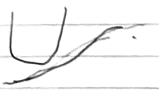
do experiment at kolbe region

flow rate calibration

rpm	V: 100 ml	time (sec)	flow rate - ml/min
30	100	4: 30	
60	100	2: 10	
100	100	1: 18	
200	100	40 sec	
300	100	26 sec	

RPM
~~ml/min~~ = 1.3092 x rpm/l/min

pump calibration



This also gives us information if methanol is oxidizing or not →

1:38
1:30
2:76
60
3:16
60sec + 38sec
98

Multipass experiment

flow rate = 20 ml/min

40ml electrolyte

T114

26.184 rpm

40ml electrolyte = 1.40sec

1:38sec

T114 01_	single pass	20ml/min	50 mA/sq cm = 157mA	3:16
T115 02_	two pass	20 ml/min	50 mA/sq cm (3min16sec)	✓
T116 03_	three pass	20 ml/min	50 mA/sq cm	✓
T117 04_	six pass	20 ml/min	50 mA/sq cm	✓
T118 05_	10 pass	20 ml/min	50 mA/sq cm	✓
T119 06_	~~30 pass~~	~~20 ml/min~~	~~50 mA/sq cm~~	
T120 07_	~~100 pass~~	~~20 ml/min~~	~~50 mA/sq cm~~	

3:16sec but it stopped (57secs circum) earlier so i need to do it separately. 40ml/min = 5236ml.

T119 01_	single pass	40 ml/min	50 mA/sq cm
T120 02_	two pass	40 ml/min	50 mA/sq cm
T121 03_	three pass	40 ml/min	50 mA/sq cm
T122 04_	six pass	40 ml/min	50 mA/sq cm
T120 &	ten pass.	40 ml/min	50 mA/sq cm ✓

th Or
O
NO
liquid
samples
for G.

Methanol: 535 to 5.6

T124

Impact of flow continuous on BDD

20221203_ 200ml/min both sides divided BDD into PHS.

01_ He flow: 30.9 ml/min
volume of electrolyte 40 ml both sides 1M Na2SO4
PHS cathodyte & anolyte.

02_ CC @50 mA/sqcm = 157mA 65 mins
Q before 0.1015 injection #1
from injection 5.

pHaffer 4.6	Methyl	4327	3243	4023
	Methanol	21691	19747	20809

T125 20221203_ 100 ml/min both sides divided flo
1131 rpm
He flow: 30.9 ml/min
volume 40ml both sides

OLOCV = 20 min
02_ CC @50 mA/sqcm = 1 65 mi

→ forgot to do gas analysis. (test for gas again)
→ needs to repeat.

Methyl	8555	8382	8610
Methanol	57099	54986	57204

T126 20221203_ 60 ml/min both sides divided flo.
40ml.

1OCV - 25 mins

02-CC @50 mA/sqcm 65 min
O2 before = 0.0751

Methyl	11422	11506	11866
Methanol	100030	96903	100938

T127 20221203_ 100 ml/min both sides divided flo
repeat for gaseous →

Because; for the gas produced from T125

	Methyl	Methanol
	11733	26836
	8269	26154

T128 20221204_ 40 ml/min both sides Nacetate (BDD/Si non pillar chip 1 cm²)
82.360

area of chip electrode : 1 cm²

01_ He purge

02_ OCV - 30 min

03_ CC = 50 mA/cm² ⇒ 50 mA 65 mins

T129 20221204_ 60 ml/min both sides (Nacetate PHS chip) ⇒ no gas
electrode broke ↳ flow rate wrong in computer actual flow 60 ml not 40) 65 mins
leakage. copper contact was oxidized.

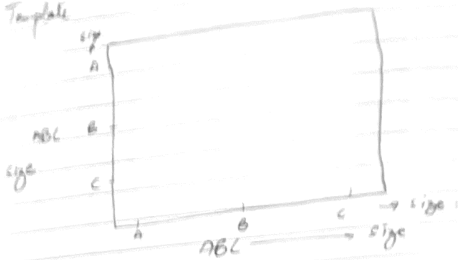
~~100, 150, 200~~ No gas analysis → 60 ml/min Both sides
High density high dopant

T130 T131 60 ml/min both sides
low density high dopant

		Methyl	Methanol	
129	18-29	4334	111	30069
				28997
130	30-52	21220	22012	
		19577	21819	
131	31-42	18489	31655	
		19086	31980	

Data analysis plan

Template



Dimensio

Paper structure
- cations impact at OER → last
- cations impact at Kolbe conditions → pH5
- cations impact at combination Kolbe en hofer meest pH9
- cations impact at hofer meest pH9, 12
- cations impact at low current density
- cation effect at high current density

CV, LSV, FE avrag, FE wst, EIS, ECMS

Time table
- Jan 30 - Jan 4 Cation paper
- Jan 5 - 20 Review paper complete
- chemical orders
- Jan 20 - Feb 14 nanoparticles pt on BDD - experiment + write up
- Jan 20 - Feb 25 RRDE paper + experiments
- Feb 19 - Feb 25
- Feb 20 submit paper faraday discussion
- Feb 28 - March 5 conferen
- March 6 - 12 Holiday
- March 15 Membrane project + pillar project

Work to do
- oxidation of levoglucosan
- reduction of methyl acetat (hydrogenation of methyl acetals)
- acid separation from pyrolysis oil using electrodialysis
- paired electrolys

- CO_2 reduction
- bio oil compounds (aldehydes / ketones reduction)
- hydrogen evolution
- Methyl acetate to ethanol (hydrogenation)
- Glucose reduction

Cation paper write up

Section : introduction
: materials & methods
: results & discussions
: conclusion
: References
: Title
: acknowledgment
: Key words
: Supporting information

Cation effect at OER : ⓐ
at pH 5 Kolbe
at pH 9
at pH 12

Current density impact at hofer Moest
low current density at kolbe condition

Faraday discussion

BDD → concentration impact 0.5M - 1M
BDD - pH impact.
→ BDD vs other substrates -
↳ BDD vs Pt vs Graphite vs Ni, vs Ni foam vs FTO
Batch vs flow (BDD & graphite)
Stability of BDD → Raman
Reaction mechanism on BDD. EC-MS

Electrodeposition.
ed1 } on diff substrates & low i
ed2 } effects
ed3 } stability by electrodeposition
ed4 }

Thin layer Pt
5, 20, 50, 100 nm

FE:
Stability
Roughness difference

disorder

PTFE coated BDD.

Done:
reaction mechanism ec-ms.

Nano

Channel J-7 → WE 2
Channel E-5 → WE

-0.005 to 2.495

OER start Nacetate 1.4 $V_{Ag/AgCl}$ + ring
onset anodic disk 1.1 @ 1800 rpm

100
400
900
1800
3030
4030
6030

6970

201 wala

Ethanol	10	381614, 397774, 384605	202
	50	1901055, 1879578, 1832026	203
	100	~~3943184~~, ~~3824570~~, , 4176650	204
		3970494, 3993389 "	
		(6.030 – 6.240) →	
Methanol	10	122087, 129704, 137157	205
	50	857439, 854863, 854053	206
	100	~~1735479~~, 157239, 1879936	207
		1017535	

208 – wala

Ethyl	3276731, 3497648, 3226666	209
	5090144, 5481259, 5235977	210
	5063712, 5130058	211

Showing same as 210.

↳ calculation problem

Methyl acet.	345830, 350002, 351782	
	~~333742, 340286, 341585~~	212
	1778480, 2082911, 1710094	213
	2866194, 3208318, 3107860	214

T164

Ethanol :	~~6997~~ 22036, 23399, ~~23359~~	216
Ethyl Pho.	6263, 6989 6927	

Methanol (Previous 6.10)

Methyl acetate (Previous 5.10)

Ethanol (Previous 6.992)

Ethyl propionate (Previous 7.3401)

RRDE - for pH

- ring HER: Hup 0.05 - 1.2
- disk Kolbe: 3.2 VRHE

Channel 5 ⊃ E
Channel 7 ⊃ J (disk, ring)

set disk to HER
set ring to Kolb

now
channel E connect to ring
channel J connected to disk

Exp
Run
Spec
Plot
1
Secon
Elec
Conv
adc

Prepare solution of 100mM methyl acetate

Prepare : in 0.1M acetate (sodium)
Prepare in 0.1M H_2SO_4.

500ml of 100 mM methyl acetate
→ 3.704g of methyl acetate
ρ m/V V_s m/ρ = 3.704/

T135

20230515_ methylacetate reduction Pb 25mA/cm²

WE = lead
CE: Ni foam
RE: Ag/AgCl
pH = 5 before
Batch cell, 50 ml volume electrolyte
Area of Pb . 3.327 cm²
Stirring on Ru = 26.868Ω
01. CC -25 mA/cm² ⇒ 83.194 mA for 1hr

pH after: 4.6

T136

20230515_ methylacetate reduction Pb -1V.

WE: lead
CE: Pt/Ti
two electrode cell.

01. CV: -1V for 30 minutes
↳ -1.5 V_{RHE}

This one i did with two electrode (do it again with 3 electrode
↓
repeated (data in same folder)

T137

20230515_ MeAc reduction Pb -2V Ref (Ag/AgCl) ⇒ 2.5 V_{RHE} 30 mins

WE lead
CE Pt/Ti
three electrode cell

T138

20230516 MeAc reduction lead -3V Ag/AgCl 3.5 V_{RHE} 30 mins

Same conditions as above

T139

20230517_500mM glucose in 1M acetate_25mA/cm²

WE: GDD
CE: Pt/Ti
RE: Ag/AgCl
Velectrolyte = 125ml
pH = 4.7

01_CV = 0-3 VRHE ≈ -0.474V to 2.525 V @50mV/s

02_CC: 1hr : 25 mA/cm² ≈ 96.720mA.

03_CV = 0-3 VRHE @50mV/s
04_CP = 12.304

m_{CAc} = 9834, 8764, 7741
m_{EOH} = 15806, 15975, 15991

T140

20230517_500mM glucose in 1M acetate_100mA/cm²

01_CC = 1hr 100 mA/sq.cm² = 193.439 mA

6 in 2 rounds, i forgot to put time to 1hr so the electrolysis is in between 20 & 40mins

m_{CAc} = 10416, 10712, 8397
m_{EOH} 18243, 20872, 16838

PK 79BAHL 00060081037200 01

NMR → Wiktoria

friday

15.6. - 16.4

to do

1) Cation RRDE graph (both disk + ring) ✓
2) Cation pH table + pic (pH 5 table + list)
3) Cation pH CV - Li Cl K
4) Cation impedance
5) Cation FE
6) RRDE: figure done:

$\varepsilon = \frac{n_{ir}}{i_d \times N_{eff}} =$

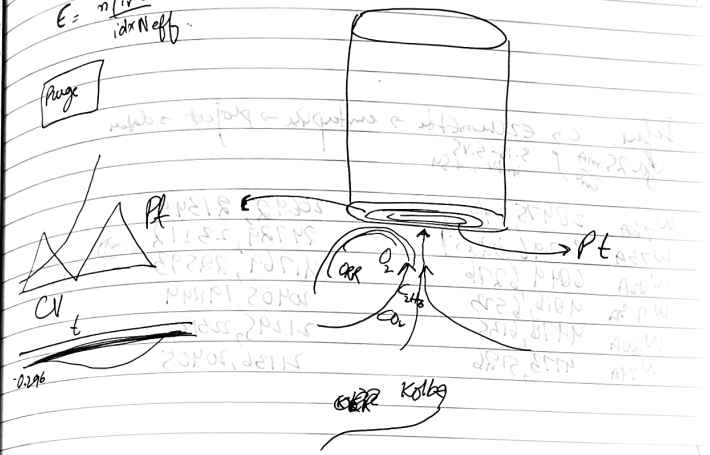
(we should not do the faradaic efficiency because ORR can be either one electron or two electrons

$N_{eff} = 3.555$ disk

0.0455 ring

$\varepsilon = \frac{2(0.0455)}{3.55 \times (26)} = 9.3$

$\varepsilon = \frac{n(ir)}{id \times N_{eff}}$



Wiktoria → folka-test 1

		MeAc	MeOH
01	H_2O	–	–
02	W_{77A}	45092, 18306, 20191	95328, 95508
03	W_{79A}	8034, 6478	29870, 26748
04	W_{80A}	9174, 9363	29169, 30047
05	W_{81}	19743, 21839	104998, 103753
06	H_2O	–	–
07	W_{83A}	8242, 6523	32633, 27111
08	W_{78A}	12281, 12854	61942, 63147
09	W_{84A}	14274, 15004	87881, 87672
10	W_{82A}	8861, 9528	29022, 32692
11	H_2O	–	–
12	W_{73A}	37769, 37409	33189, 36021
13	W_{74A}	48333, 47903	102376, 105176
14	W_{75A}	47808, 44498	63474, 66071
15	W_{76A}	32016, 31962	26917, 30833
16	W_{72A}	38671, 45098	89285, 95320
17			
18			
19			

∫ 5.1–5.660
∫ 4.2–4.650 for 100 mA 64.6 cm

Defaul: C → EZChromElite → enterprise → project → data

for 25 mA/cm² ∫ 5.15–5.45, 4.34, 4.54

W_{76A}	28475, 29877	20692, 21346
W_{73A}	32296, 32259	24729, 23212
W_{82A}	6849, 6276	21769, 22593
W_{83A}	4816, 6523	20405, 19844
W_{80A}	4978, 6135	21295, 22318
W_{79A}	4773, 5126	21136, 20405

∫ 4.3–4.55, 5.15–5.65 → for 62.4, 100 mA/cm²

(M11)

		MeAc	MeOH
201	H_2O		
202	W_{68}	15497, 14241, 14616	95258, 102739, 96541
203	H_2O	–	–
204	W_{69}	30497, 31899, 30196	27886, 28313, 27138
204	H_2O	–	–
205	W 86	18604, 20241, 17670	103109, 105282, 100566
206	H_2O	–	–
207	W 87	10033, 11226, 13015	59854, 58425, 59062
208	H_2O		–
209	W 70	41592, 39336, 41468	105673, 99905, 102237
210	H_2O		
211	W 71	53020, 52291, 54149	65986, 67744, 66923
212	H_2O		
213	W 88 (86)	4366, 4711, 5424	21673, 22360, 21430
214			

Wiktoria → 20230526

201	H_2O	–	–
202	W_{90A}	13602, 12288, 12244	70566, 69149, 66831
203	H_2O		
204	W_{91A}	6110, 5274	35211, 29974
205	H_2O		
206	W_{92A}		
207	H_2O		
208	W_{93A}		
209	H_2O		
210	W_{94A}		
211	H_2O		
212	W_{95A}		
213	H_2O		
214	W_{96A}		

} Baseline problem

Cation transport in diffuse double layer.

→ simulation of transport of different cation at the vicinity of electrodes.

Exp

Run

Apm

Plo

Secon

Elec

Cons

adc

---

pump → be careful
check chamber might broken.

tip.
pressure gauge

filament & off. to close
multiplier and filament.

100% → work

tips should be vaccum. ∴ system is sensitive
→ type clean and dry.

lines at back are gases.

learn to clean lines.
using new to vaccum the line
He, H_2, Ar, NO

yellow sheet should be there due to pressure in ECMS.
→ gases should be written in yellow card

Zilien.exe → software to control EC-MS
→ setup
→ electrochem

important EC labpaper.

→ to change parameter and calibrate PV Mass spec.

safety mode. PC setup 1100 mbar → set when finish experiment

Valve 14 always open

Valve five → chamber to atmosphere.

1.2e-9 → pressure in chamber in safety mode.
6

-6 → operating mode.

He should be there as main gas.

to clean line use valve 13 vacuum
before that close valve 1 and valve ...

Clean line by end of day

~~purge~~ protect chip during ~~purge~~ clean line.

15 mbar more pressure than pressure in lab
PC setpoint.

tip → can block → changes slow.
→ break → make sure filament goes off.

if tip breaks → pressure is going up.

switch off device, the signals are show the
open PV max spec

multiplexer | x multiplexer.

0.15 N m for plates screw.

0.1 N m for big screws.

chip is hydrophobic

pH should not be above 12

test electrolyte with contact angle

 → the bubble should be like this

PTFE important → clean with ethanol
and H_2O.

V4-2

V4-3

V4 and V_2

< 4 mbar

< 3.5 mbar

purge with gas, other good.

4 → 2 open
then close 3.

Never go home with valve 5 open.

V4 - V3 V1.

→ presentation:

- ⊙ impact of fuge / not fuge
- ⊙ impact of glucose
- ⊙ flow cell.
- ⊙ with platinum flow undivided
- ⊙ with BDD flow undivided
- ⊙

Connection are important..

20mV/sec normally..

E range: 100μA

bandwidth 7.3

[illegible] ∫∫ → bad = due to bubble. -

O_2 & N_2 are high..

↳ take pic of paper, push electrolyte

N_2 & O_2 = − 9 range..

Trigger EC MMs.

W100
W10n

→ push in connectors

→ seal material. , Product material

→ uniflow..

Anolyte.

three focus!!.

New experiments / new work.

- RRDE
- acetic acid / sodium acetate
- acetic acid / sodium propionate
- sodium propionate

with pt and BDD

- acetic acid + glucose
- acetic acid + cresol with platinum and BDD.
- acetic acid with different concentration
- check of OER (by ORR on ring)
- check for Methanol (by methanol oxidation on ring)
- check for Ethanol (by ethanol oxidation on ring)

Target 1

use the BDD:

run blank

1. 1M acetic acid / sodium acetate
2. 0.5 M acetic acid / sodium / potassium acetate
3. 0.1M acetic acid / sodium / potassium acetate

Methanol oxidation = 0.4 VRHE – 1.0 VRHE

Ethanol oxidation = 0.5 – 1.5 VRHE

RRDE BDD on Pt

20230612_ BDD RRDE_ new Pt Ring BDD disc

Cable B → Ring → channel 2

Cable S → disc → channel 3

goal: to see methanol oxidation on platinum ring.

WE: BDD : from cordias

CE: Pt/Ti

RE: Ag/AgCl with nitrogen (the solution) and make a purge blanket.

first step is to determine where the MeOH current.

CV @ 100 rpm.

0 – 3 VRHE : – 0.505 VAg/AgCl to 2.495 VAg/AgCl

dec. 0.4 VRHE = 2~~.905~~ – 0.105 VAg/AgCl.

Ring : 2 @ 50 mV/s.

02. 2000 rpm: ring = – 0.005 VAg/AgCl à 0.5

to see if we have methanol.

CVs shows no OER no MeOH

03. 2000 rpm: ring = 0.6 VAg/AgCl RHE ⇒ 0.095 VAg/AgCl

no MeOH.

04. 2000 rpm : ring = 0.7 VRHE ⇒ 0.195 VAg/AgCl.

05. 2000 rpm: ring = 0.8 VRHE ⇒ 0.295 VAg/AgCl.

(try to run the CV on methanol on ring).

Ring with normal working electrode.

06. 2000 rpm ring 1.0 VRHE ⇒ 0.495 VAg/AgCl

0.5 M EtOH CV on Pt

pH: 5.9.

07_CV: 0 - 1.5 V Ag/AgCl -0.5581 to 0.9419 V RHE

no rpm

08_CV: - 0.568 to 1.8 no rpm

here i added the NaOH and then i could see methanol oxidation.

in acidic condition, the methanol is not oxidizing

Paper: "Why methanol electro-oxidation on platinum in water takes place only in the presence of adsorbed OH."

Now trying with alkaline solution.

pH = 8.3 1M acetate

09_CV: 0 - 3 V RHE : 0.7 V RHE Ag/AgCl — 2.303 V Ag/AgCl

@ 2000 rpm

ring: 0.5 V RHE : -0.1997 V Ag/AgCl

no 2. → no MeOH

10_CV: 0 - 3 V RHE = 0.7 to 2.303 V Ag/AgCl

ring: 0.8 V RHE @ 2000 rpm

↳ 0.1V.

11_CV: 0-3 V RHE =

@ 100 mV/s ring: 0.8 V RHE

12_CV: 0-4 V RHE 0.7 to 3.300 V Ag/AgCl

@ 100 mV/s ring 0.8 V RHE

1.9625

13_CC: 10 mA/cm² disk. ~~4.9662~~ mA

ring 0.8 V RHE no MeOH

14_CC: 25 mA/cm² = 4.90625 mA

ring 0.8 V RHE no MeOH

15_CV: 0-3 V RHE @ 50 mV/s no rotation.

ring = 0.8 V RHE.

NOW switch to pH. 11

16_CV: 0-3 V RHE : -0.859 2.141

ring 0.8 V RHE -0.059 V Ag/AgCl

no rotation.

@ 50 mV/s

17_CV: 0-3 V RHE = -0.859 to 2.141.

ring 0.8 V RHE -0.059 V Ag/AgCl

no rotation

@ 20 mV/s.

18_CV: 0-3 V RHE = -0.859 to 2.141

ring 0.8 V RHE

@ 100 no rotation

mV/s

19_CV: 0-3 V RHE

ring = 0.8

2000 rpm @ 50 mV/s

20_CV: 0 - 3 V RHE : -0.859 to 2.141 V Ag/AgCl

ring = 0.8 V RHE = -0.059 V Ag/AgCl

@ 3500 rpm

pH 3: acetic acid + acetate
pH 9: acetic acid + acetate
pH 12: acetic acid + acetate.

Li / K.

pH 3

RHE 0.05 ↓ E Ag/AgCl −0.337 to 2.613

RHE 3 E Ag/AgCl

Ring = −0.687 (0.3 V RHE)

@ 50 mV/s.

01_Li — Peak Dis = 2.1; Peak Ring = −0.1

02_K — Real Dis 2.6; Ring = −0.138

03 1M LiOH pH 12.1 (−0.6539) (disk −0.8739 to 2.0761)

04_1M KOH pH 12.9 (ring = 0.2) (−0.6711) Peak dis (115) (−0.9211 to 2.028)

05_1M KOH new 13.5 (−0.7065) (−0.9565 to 1.9935)

06_1M Kacetate pH 12.6 (−0.6534) (−0.9034 to 2.046)

07_1M Kacetate pH 11.11 (−0.56549) (−0.81549 to 2.13451)

08 1M