N.E.W.B.I.E.S Pilot Plant

Progress Update

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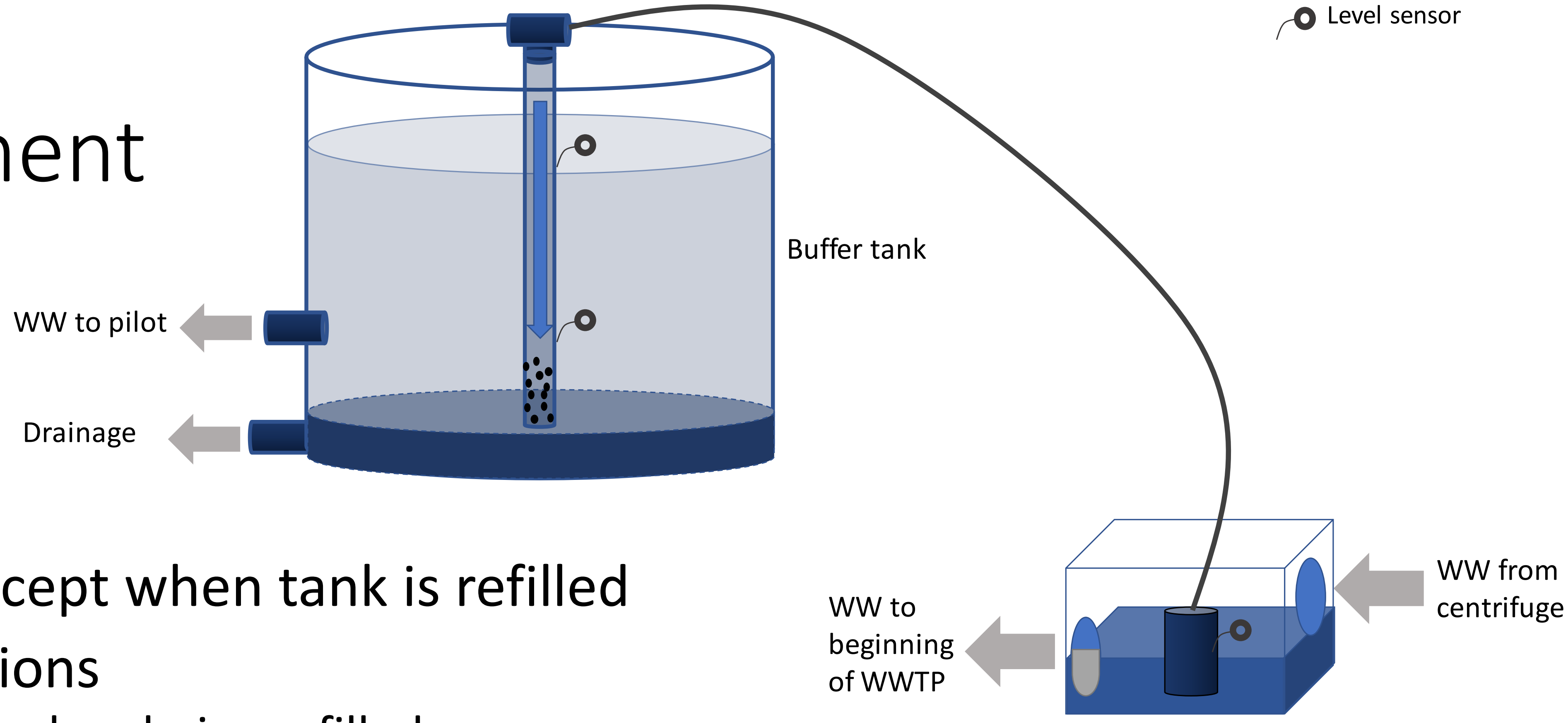
19 November 2019

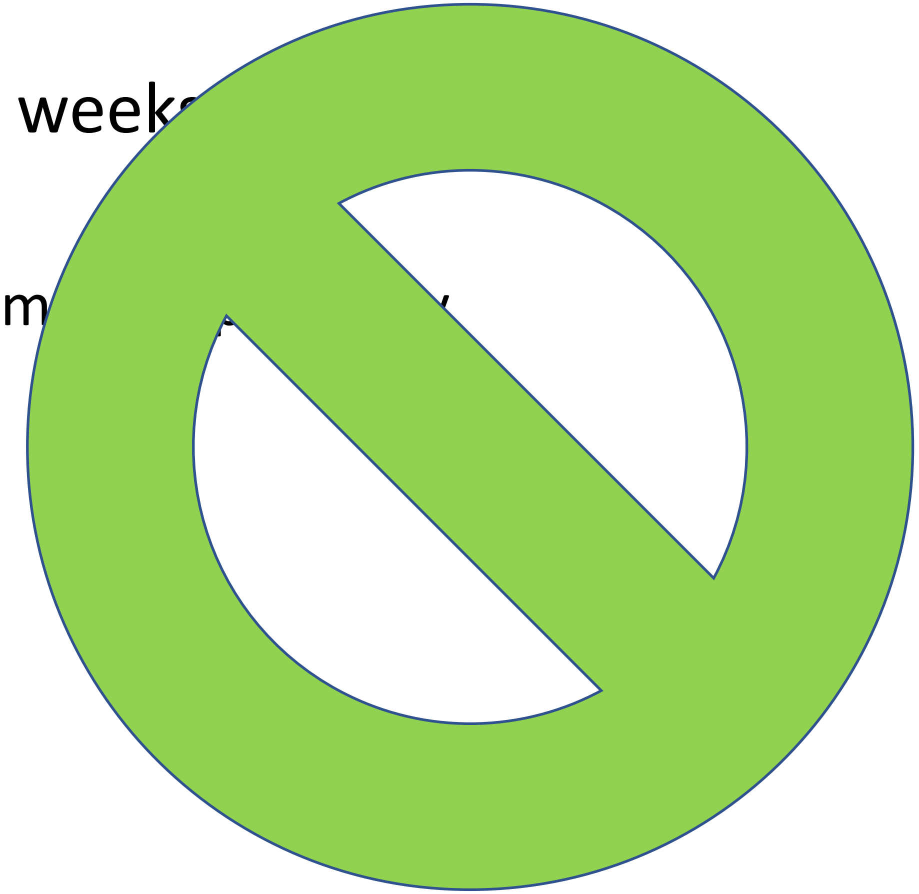
Pretreatment



1. Works well except when tank is refilled
2. Possible solutions
   - Don't use tank when being refilled
   - Include additional filter (with backwashing possibility)
3. At higher filter pressures:
   - the feed pump can't match the internal feed rate.
   - Prevents full pressure range of filters to be used.

Software and hardware control

- Many small bugs and errors solved over last week
- Still some unresolved issues:
    - Large amount of variables not properly logged, m
        - Current and voltage
        - Feed and Stripper pH and conductivity
        - Temperatures
    - Data export to Excel not working
    - Database sometimes erases without warning
    - Feed pumps overdose during start-up



Initial operational strategy

Pilot designed to control process with:

(1) Conservation of cation concentrate throughout cleaning cycles

(2) Cleaning of stack/TMCS triggered by:
- high voltage on stack
- Time interval on TMCS
- pressure drop of recirculate pumps

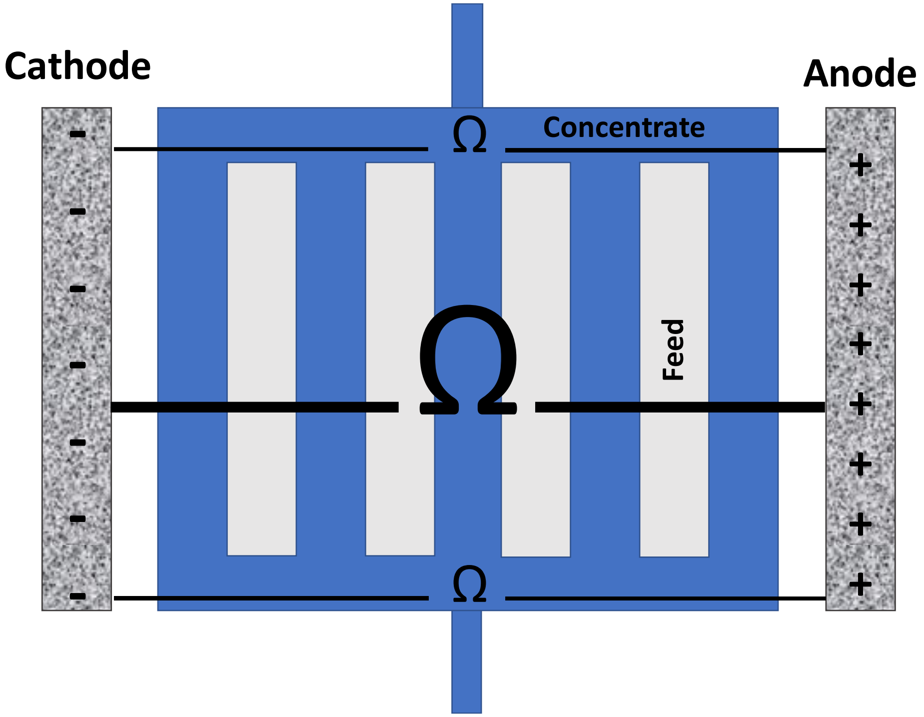
(3) Only TMCS and Stack compartments included in cleaning

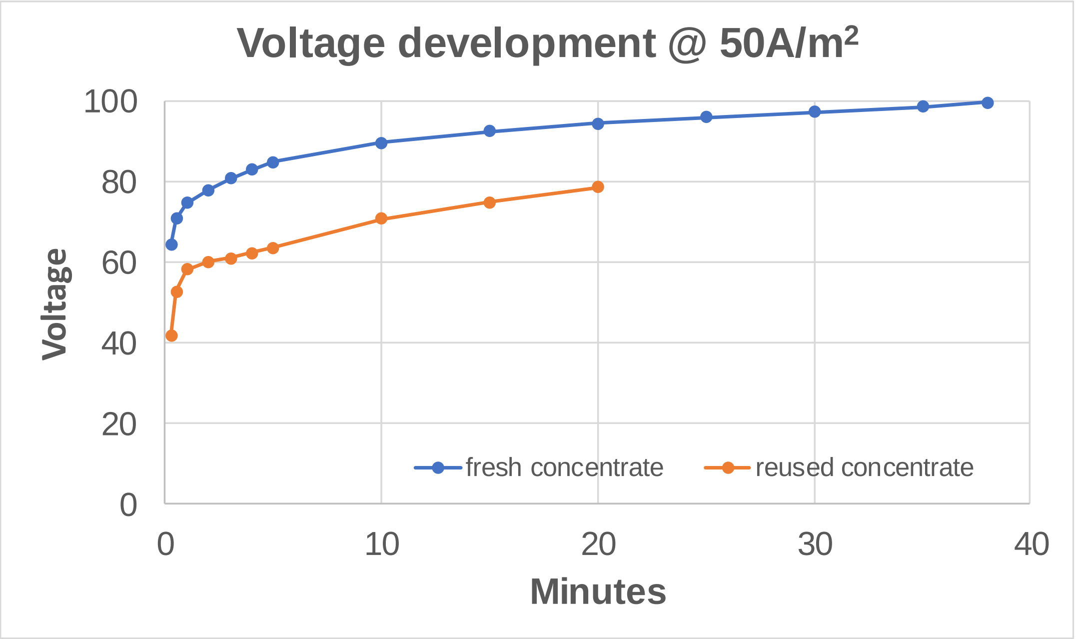
Practical issues encountered

1. Small voltage window at required current density led to very frequent cleaning of stack
2. Fast onset and rate of flow reduction led to frequent cleaning or required substantially decreased flow rates
3. Slow resumption and (progressively) decreased rate of stripping after cleaning cycles
4. Progressive shortening of runs throughout cleaning cycles
5. Pump and flow sensor failures
6. Doubts about TMCS performance

Operational Voltage Window

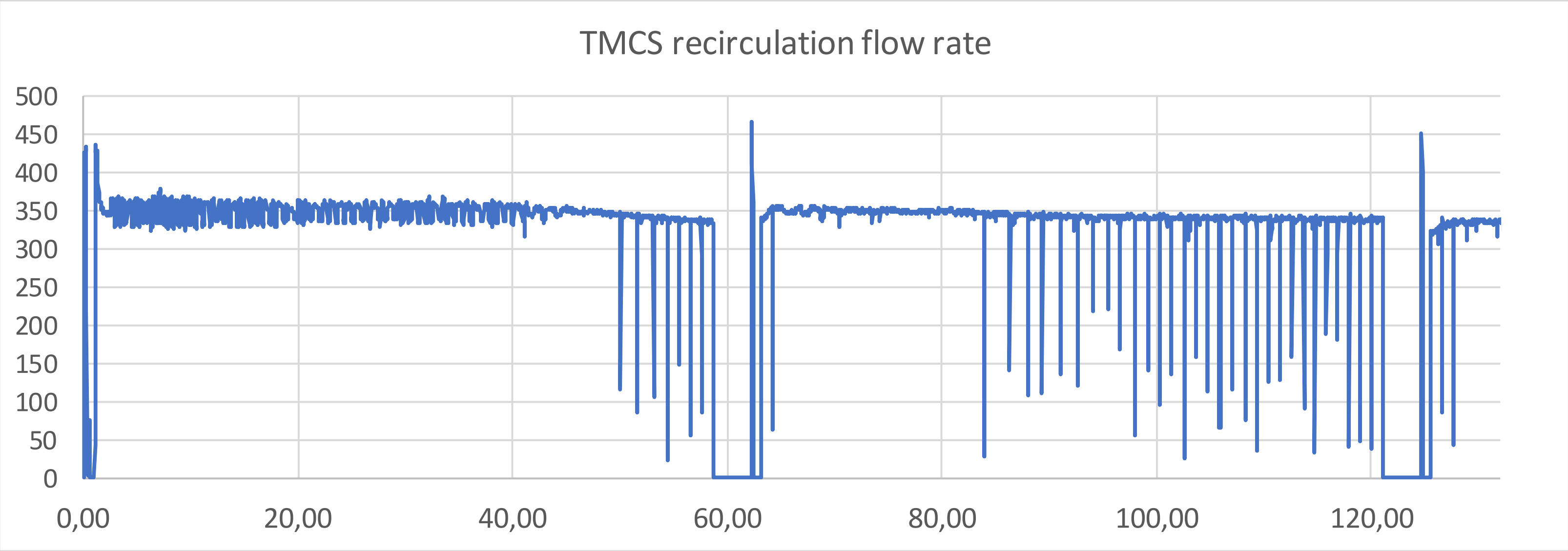
- Conductivity lower than anticipated
  - Requires higher voltage to drive current through stack
  - Larger transport number w.r.t. calcium
- Power supply limited to 100V
  - Won’t allow testing over relevant time intervals beyond 50A/m2
  - May increase relative share of ionic shortcut through concentrate

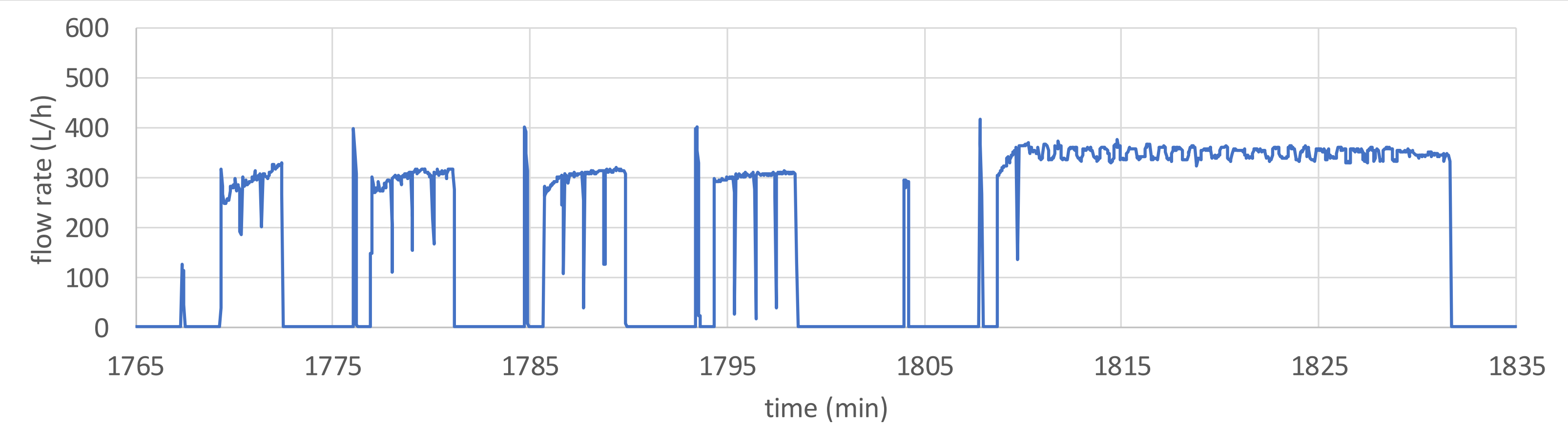




Recirculation flow rates

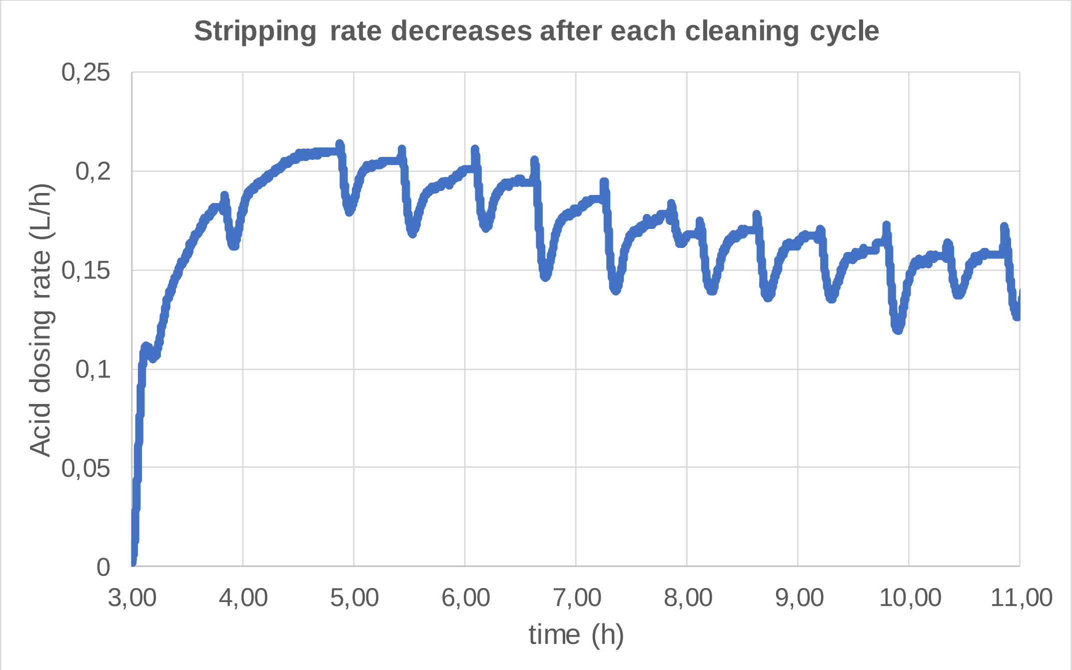
- Fast onset of flow drop in recirculation observed.
  - Less useful for cleaning triggering
- (Temporary) solution:
  - run at full capacity
  - lower setpoint for measured flow triggers cleaning

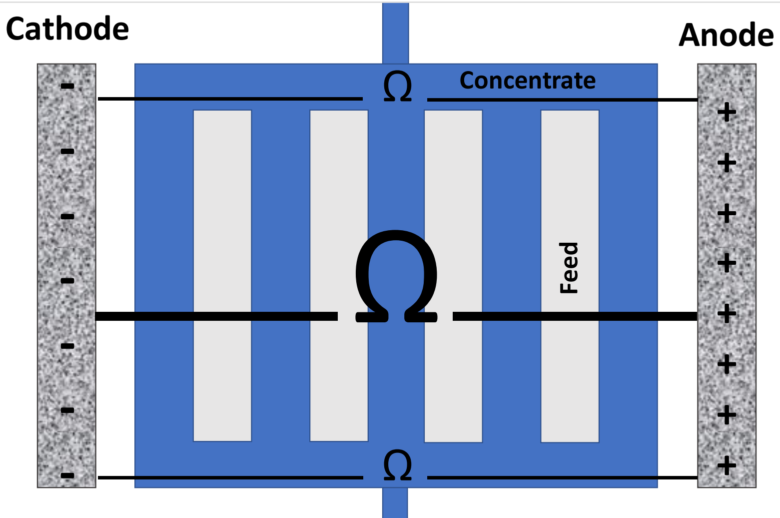




Slow resumption/decreased rate of stripping after cleaning

- Increase in conductivity cation concentrate after cleaning due to carry-over
- Resumption of stripping became progressively worse:
  - Carry-over of rinsing acid leads to lower pH of cation concentrate: needs to be neutralized
  - Higher osmotic pressure difference between feed en concentrate causes increased water transport, diluting concentrate over next run
  - Co-ion transport of chloride over CEM may decrease ED selectivity
  - Greater difference in conductivity between feed and concentrate enlarges ionic shortcut issue





Carry-over of rinsing solutions

- Cleaning cycles progressively more frequent
- Impairing process efficiency
- Carry-over main suspect:

(i) Previously dissolved calcium reintroduced into concentrate.
  - (i) Rapidly precipitates during the following run.
  - (ii) Becomes more “spreading out the dirt”
  - (iii) Problem evolves over time as rinsing acid contains more calcium

(ii) Cleaning acid loses acidity faster than anticipated
  - (i) More calcium in wastewater?
  - (ii) Too large carry-over? Measured *in situ* to be 100-300mL. More than designed for (<50mL).

Pump and flow sensor failure

Precipitation of calcium carbonate caused issues with moving parts in cation concentrate flow:

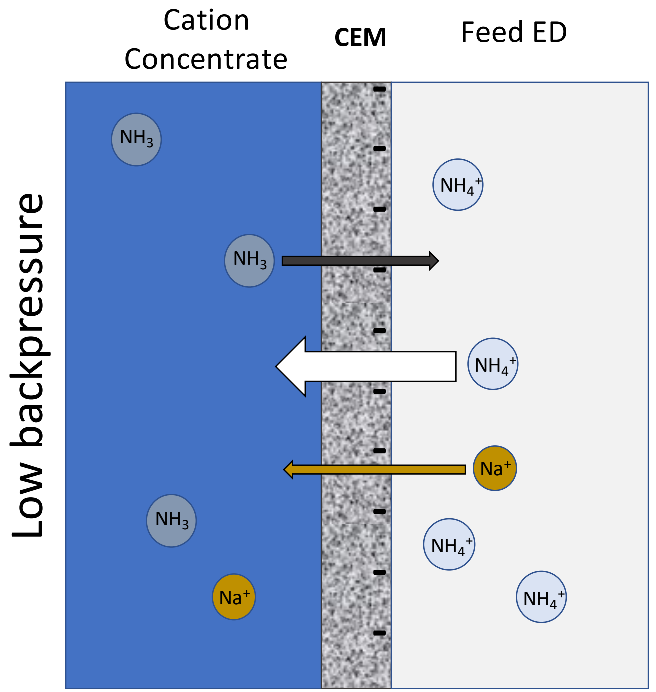
- Gradual decrease of recirculated flow rate, independent of cleaning
- Complete blocking of pumps. Recovered by cleaning with acid
- If only pumps are cleaned, eventually also flow meters get stuck
- Rate of precipitation within pumps strongly dependent on amount of cleaning cycles and operation of stack:
  - ✓ Keep current going
  - ✓ Don't backwash concentrate through pumps
  - ✓ Prevent carry-over of rinsing acid

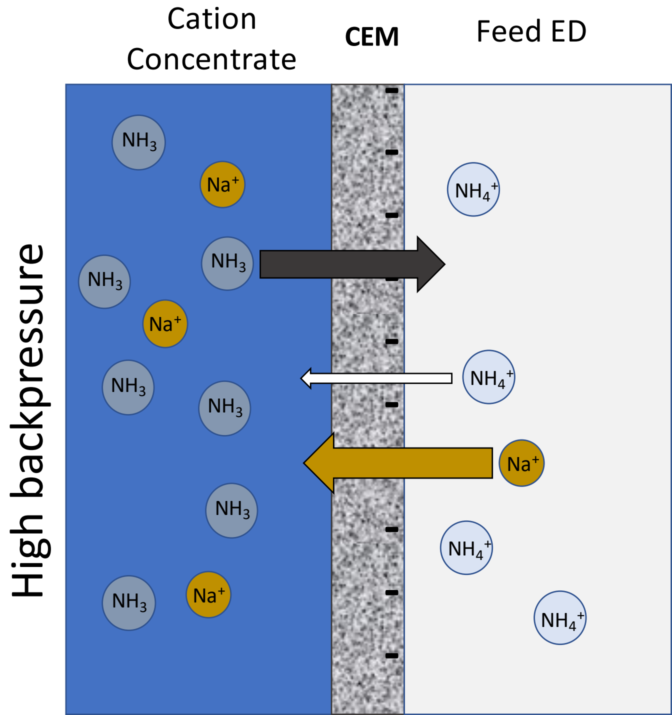
Effectiveness of currently used TMCS

When NH_3 not sufficiently stripped, higher concentrations accumulate in concentrate, leading to:

(i) higher concentrations of co-ions:
   (i) Larger water transport
   (ii) Higher pH
   (iii) Larger absolute transport number of co-ions (lower CE)
   (iv) Larger calcium deposits (scaling issues)

(ii) Backdiffusion of NH_3





Effectiveness of currently used TMCS

Tests that can further clarify the influence of stripping:

(i) Vary stripping membrane area
(1 vs. 2 TMCSes)

(ii) Vary stripping pH

(iii) Vary stripping temperature

Best practices in system characterization

- Start run clean
- Disable all automatic cleaning procedures
- Put flow rates at maximum for sensitive process flows
- Run at constant voltage
- Observe system performance
- Run ends when problem occurs:
    - Stack fouling / scaling
    - TMCS blocked
    - Pump failure
    - End of stripping

Characterization run: data acquisition

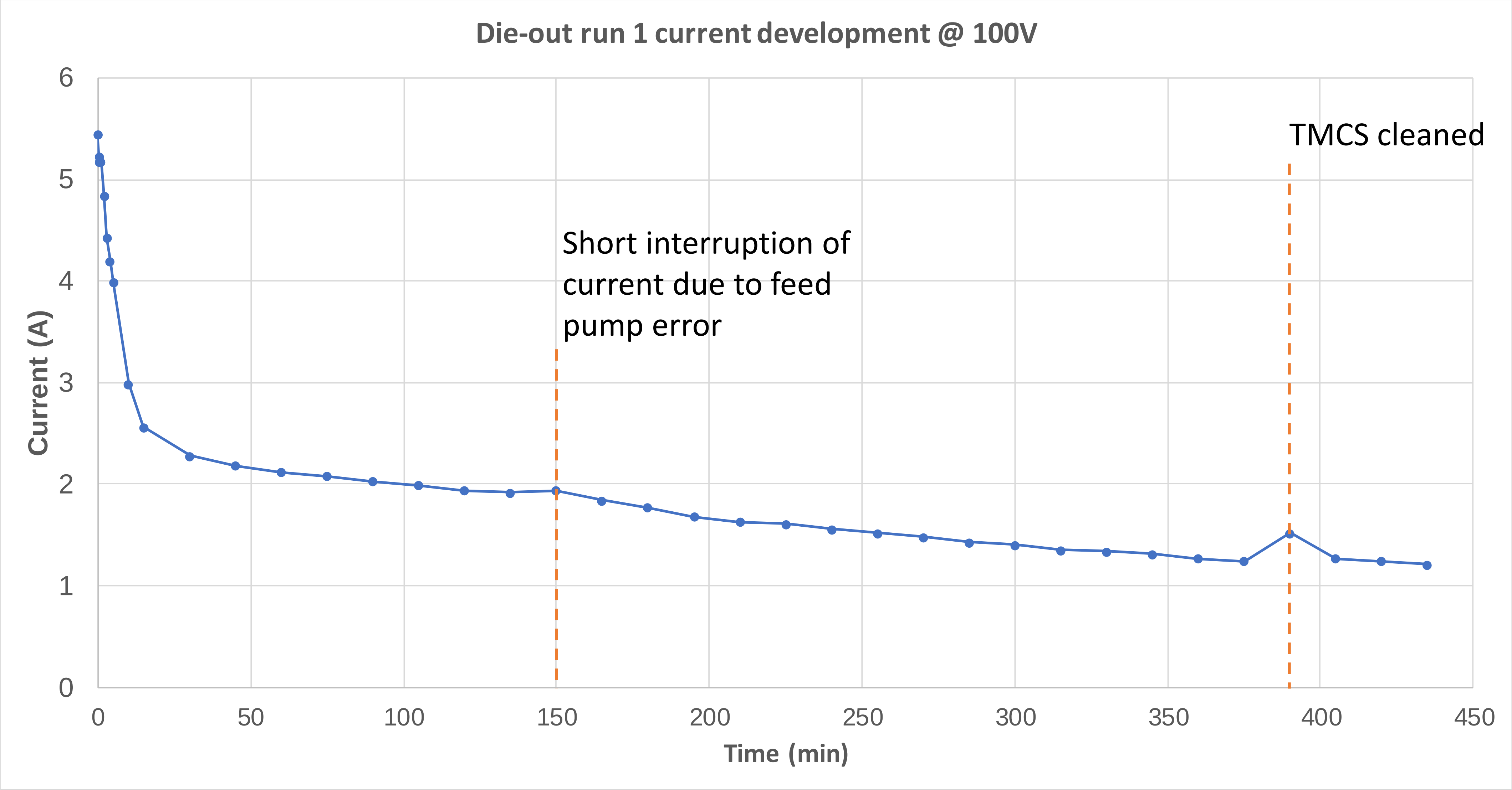
Recovery Efficiency

1. Current over time
2. Acid dosing (effective stripping)
3. Product flow rate
4. Recirculation flow rates (indicate pump, stack and TMCS blocking)
5. Cation concentrate flux (water transport)
    1. Sample composition of concentrate
    2. Clean system after run and measure Ca2+ increase in rinsing liquid
6. pH and conductivity of process flows
    1. Provides information on realized concentration gradients
    2. Sample feed recirculate composition for removal efficiency

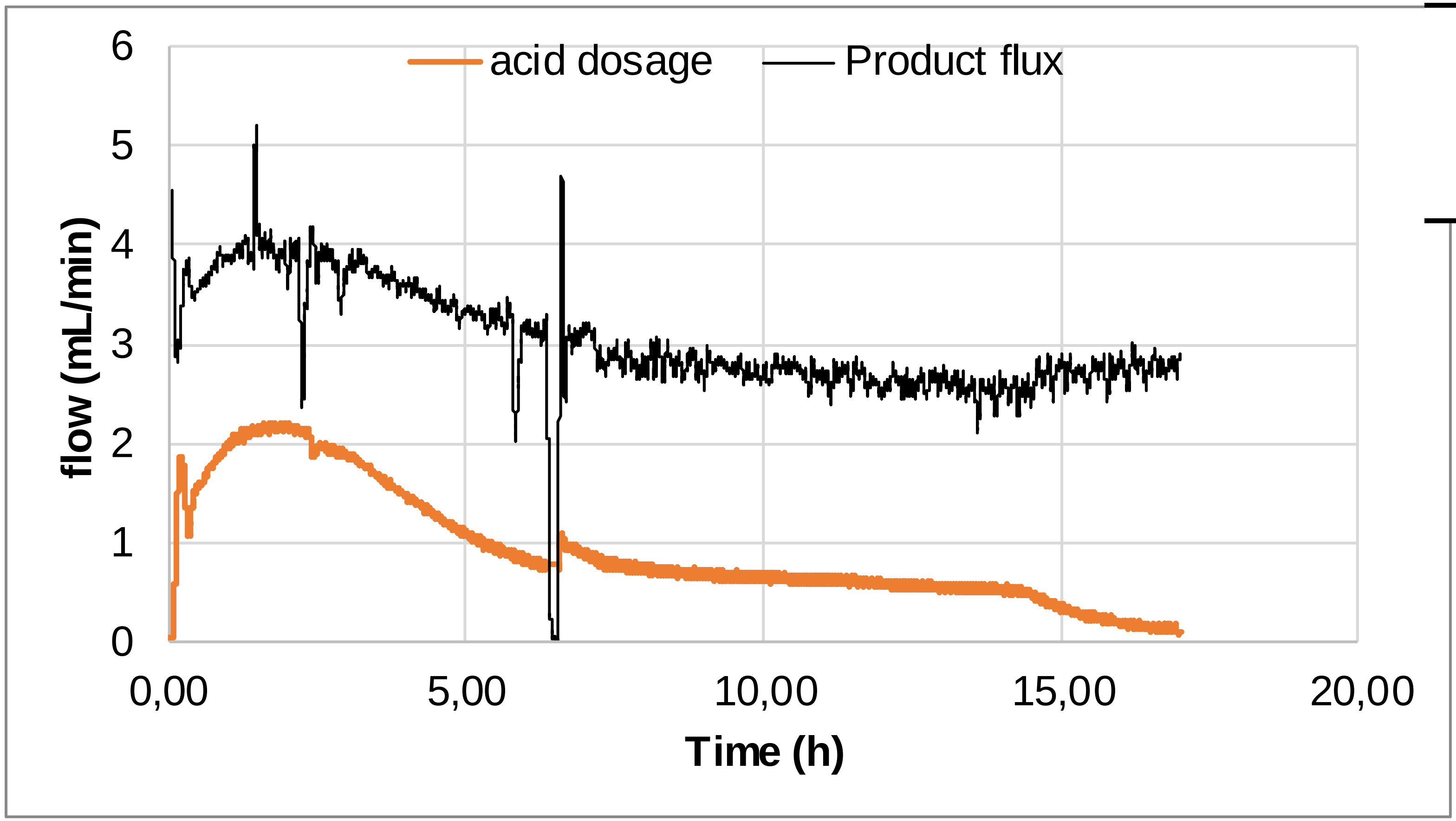
Product Density

Transport Numbers

Chronoamperometry



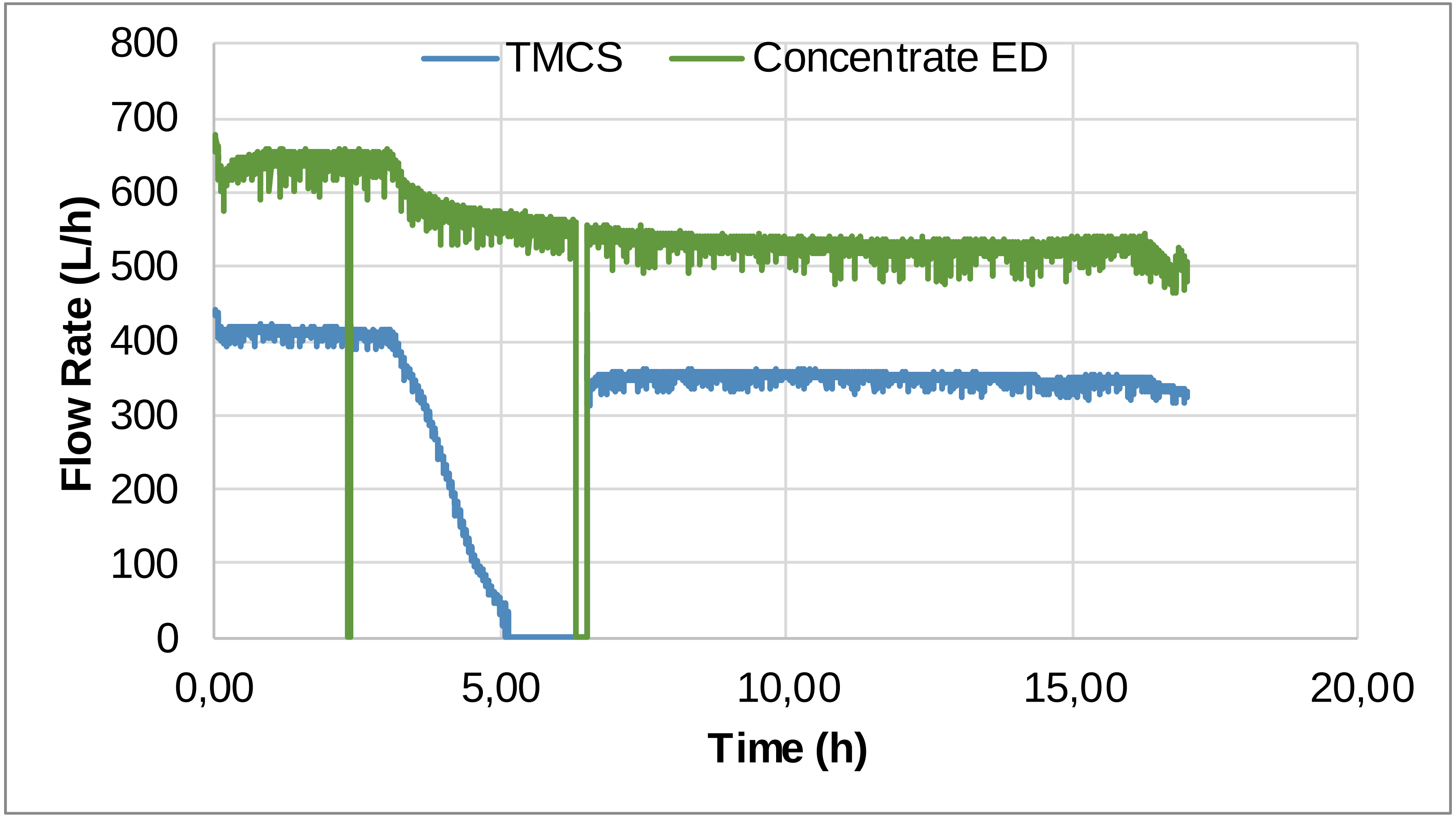
Product flow rate and acid dosing (effective stripping)



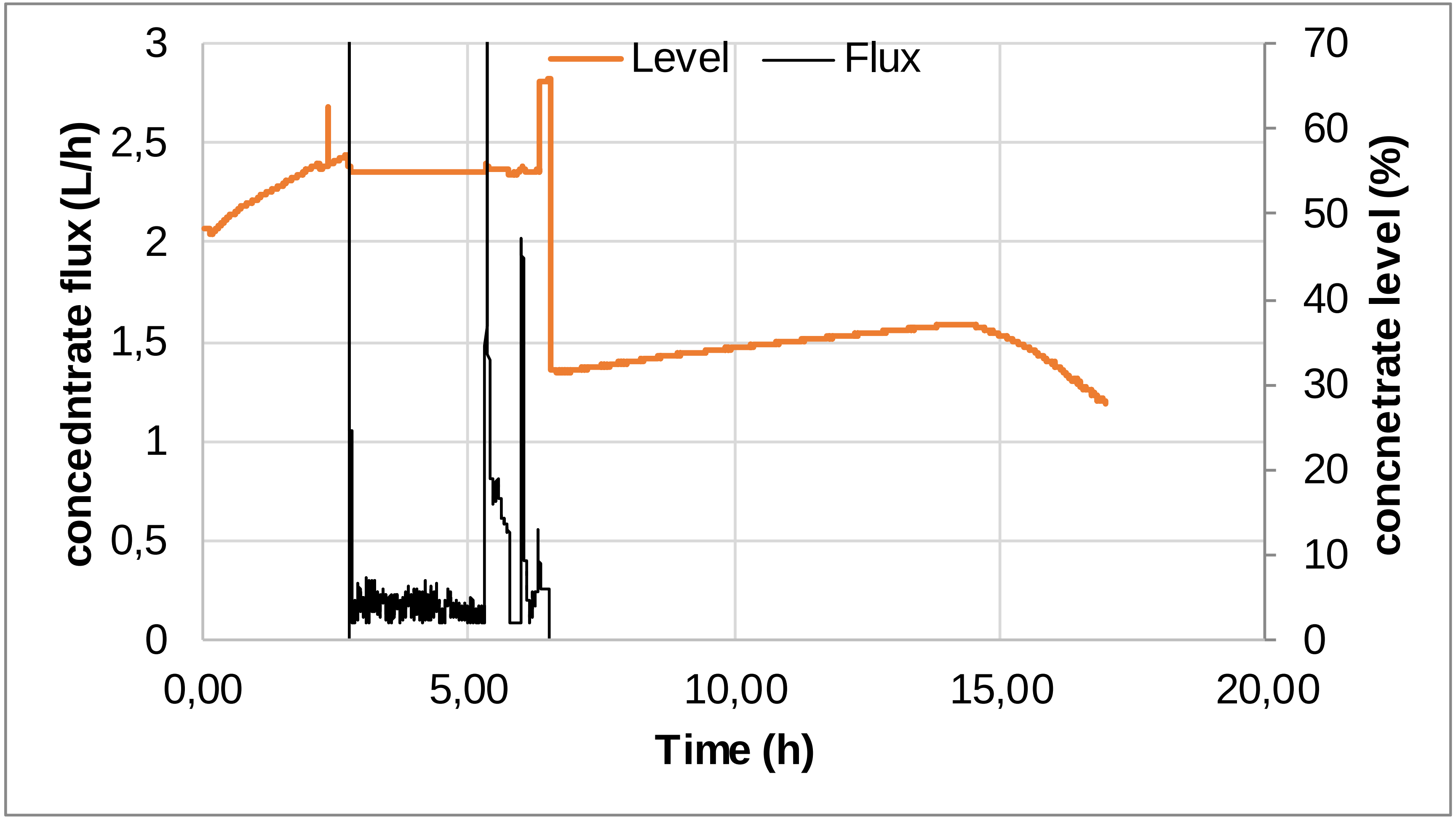
concentrations N measured in process flows:

mg/L Time/	Feed ED	Cation Concentrate	Stripper
0	94,7		
2,25	83,6	583	38300
5,75	155	503	38900

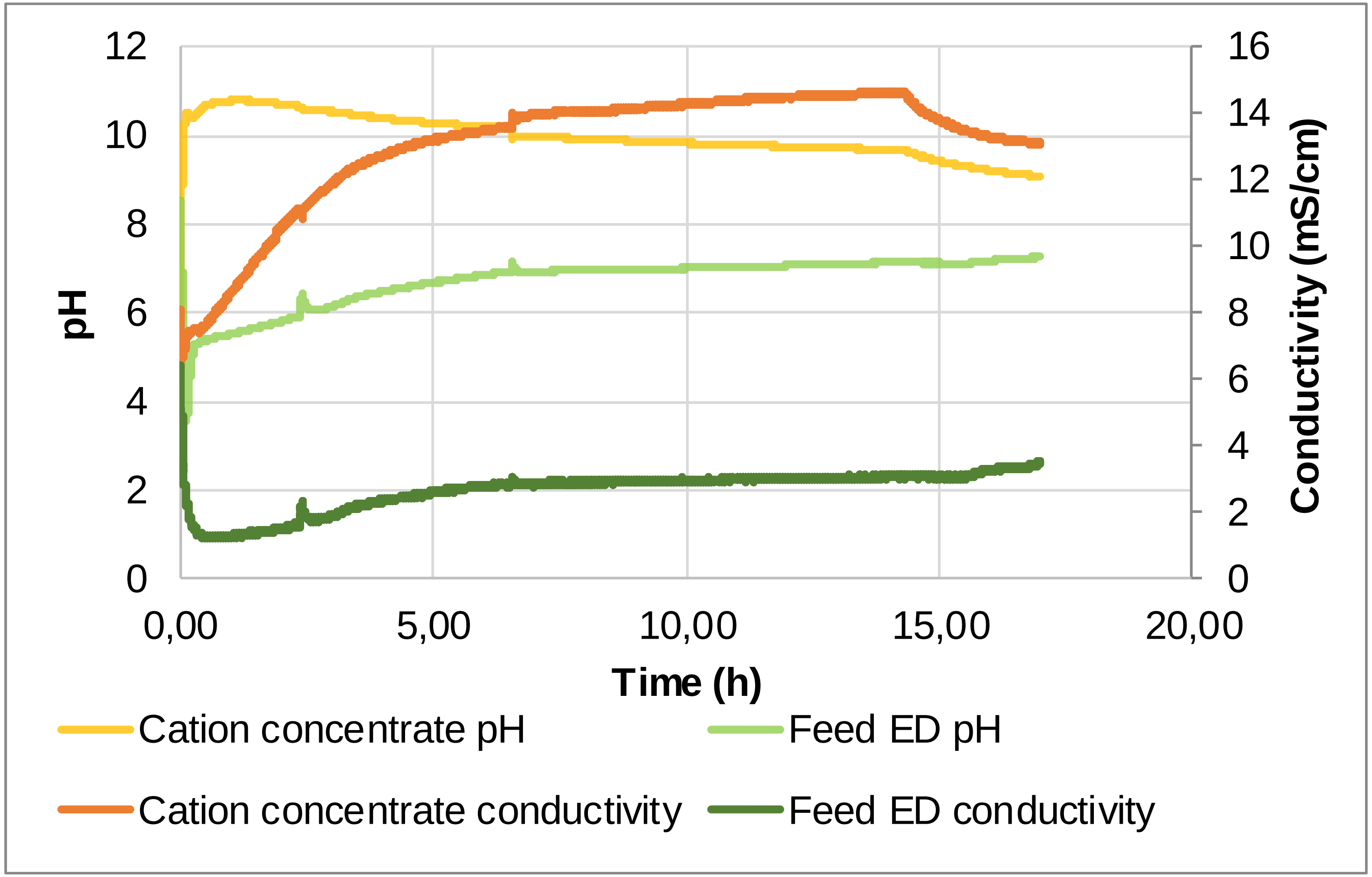
Recirculation flow rates (indicate pump, stack and TMCS blocking



Cation concentrate flux (water transport)



pH and conductivity of process flows



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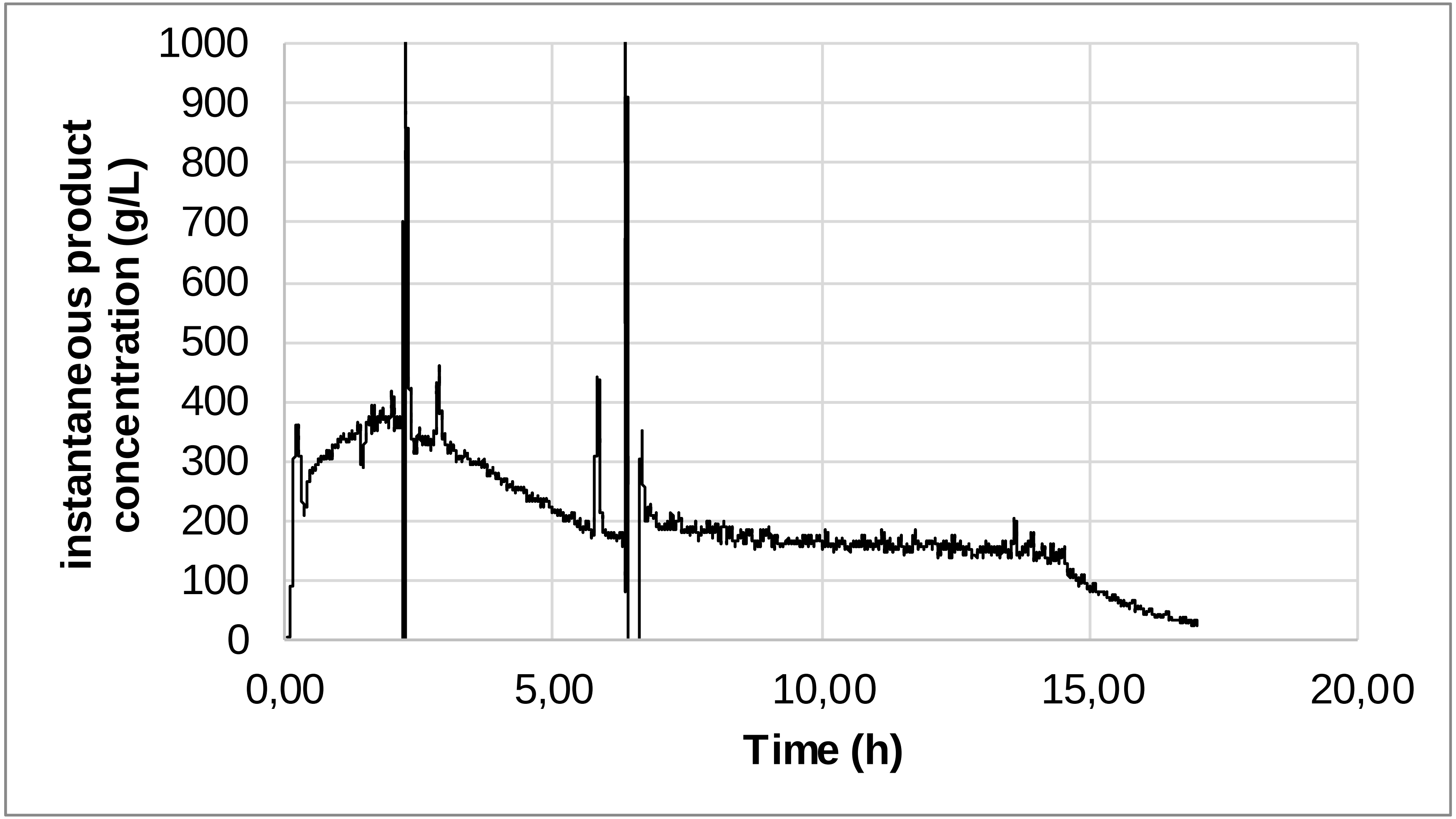
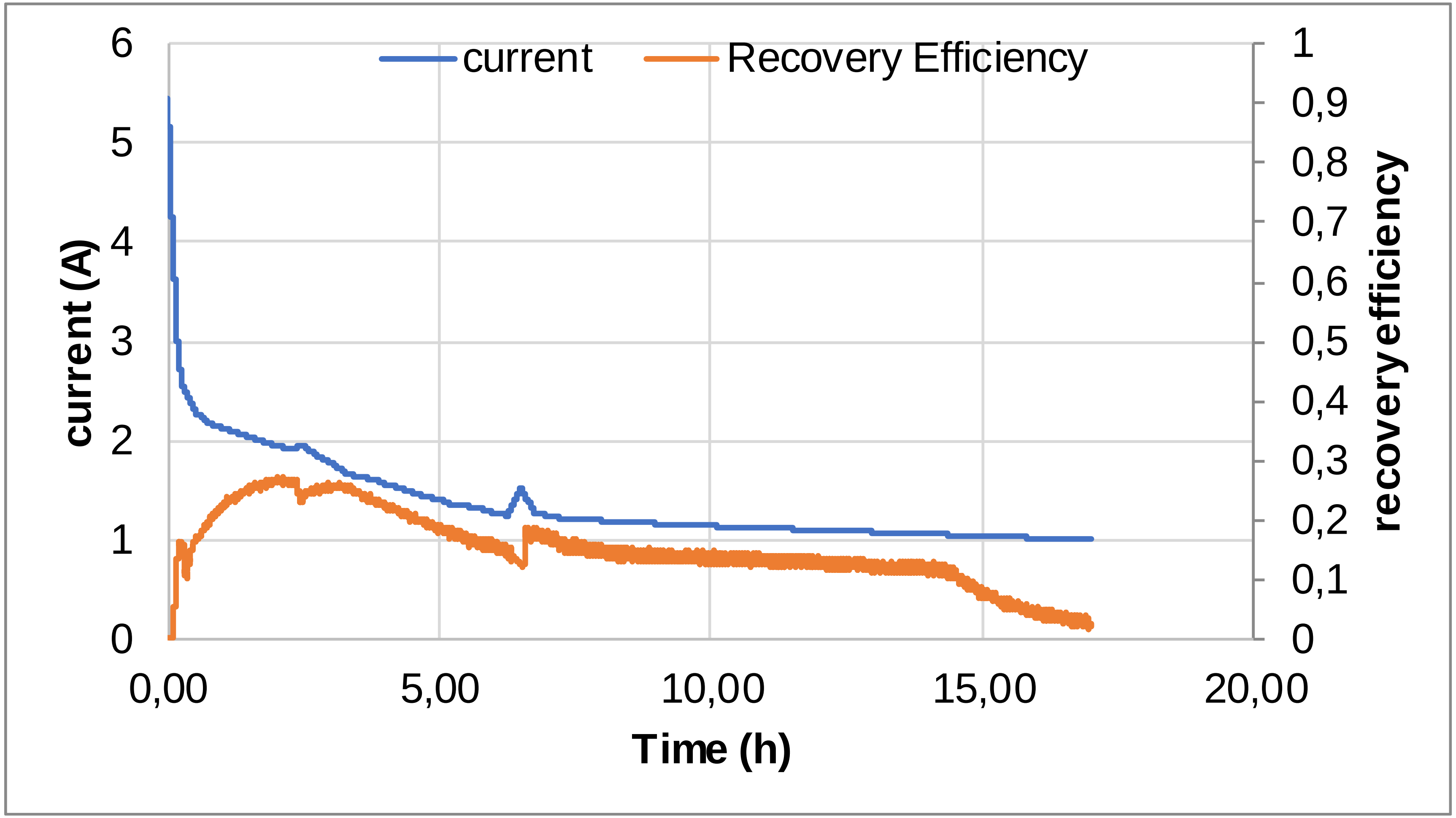


pH and conductivity of process flows



Follow-up research questions

1. Effect of TMCS
   - 1-2 TMCS
   - Higher stripping temperature
   - Lower stripping pH
2. Effect of concentrate conservation
   - Exclude carry-over manually
   - Compare repeated runs using new concentrate vs. keeping the old
3. Effect of Current density
   - Testing at lower voltage using current PSU
   - Reduce stack size
   - Test at higher currents/voltages (new PSU)
4. Effect of current pulsation
   - Distinguish permselectivity of membranes / co-ion transport and ionic shortcuts
5. Effect of recycling cation flux to feed
   - slight increase of feed recirculate conductivity
   - slightly lowered voltage
6. Effect of carry-over on pump related issues
   - Run experiments using manual cleaning, preventing carry-over
7. Composition / purity of obtained product

Considerations towards continuous operation

After previous questions are answered by experiment, decisions may be made on:

1. Stripper
   - Preferred stripping pH
   - Other type of stripper
   - Heating of stripping process
2. ED process
   - Thinner stack
   - Heavier PSU
   - Pulsation of current
3. Adaptation of cleaning procedure
   - conservation of concentrate?
   - carry-over minimalisation required? additional/other triggers:
     - minimal flow rate,
     - time intervals,
     - minimal current,
     - minimal acid dosing
   - Include pumps/flow-meters in cleaning?
   - If pumps not included: other type of pumps needed for concentrate?

Proposed project planning

- Nov-Dec: conducting further characterization experiments
- Jan-Feb: implementing adaptations based on previous outcomes
- Mar-Apr: continuous operation on digestate
- Periodically: testing product quality
- Feb-Apr: preparations for next phase:
  - Pre-treatment tests?
  - Lab scale tests on urine?