

Salinity Reduction of Yellow water

Final Presentation

15 March 2023

Plan

- 1. Objectives
- 2. Requirements
- 3. Technology Trade-off
- 4. Feasibility tests
- 5. Proposed scenario
- 6. Conclusions and future work





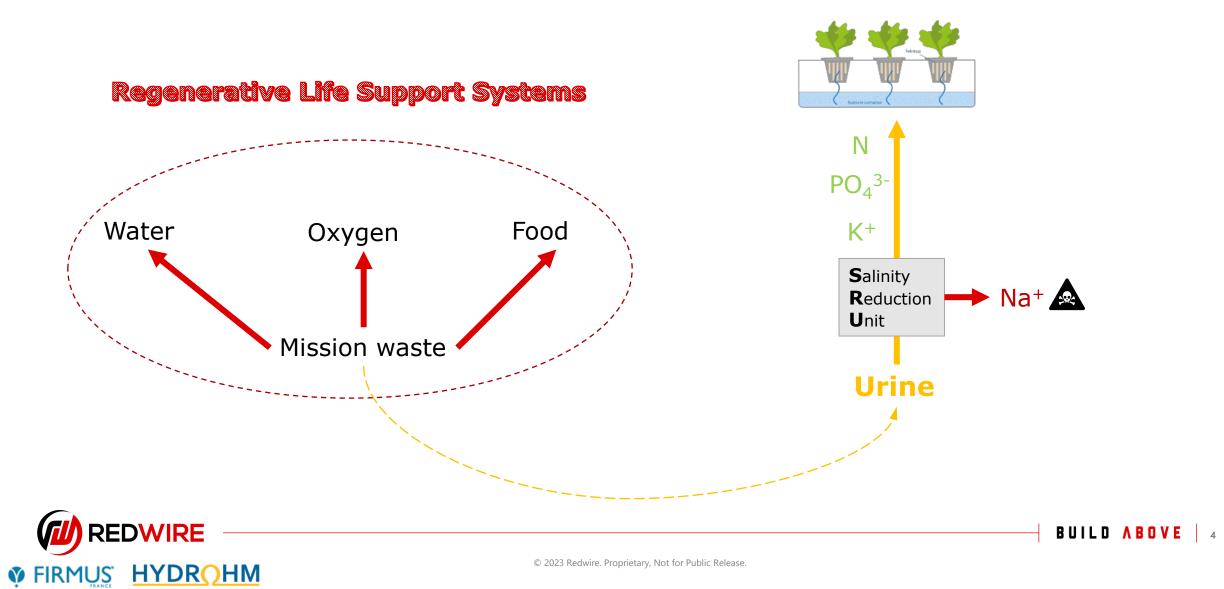








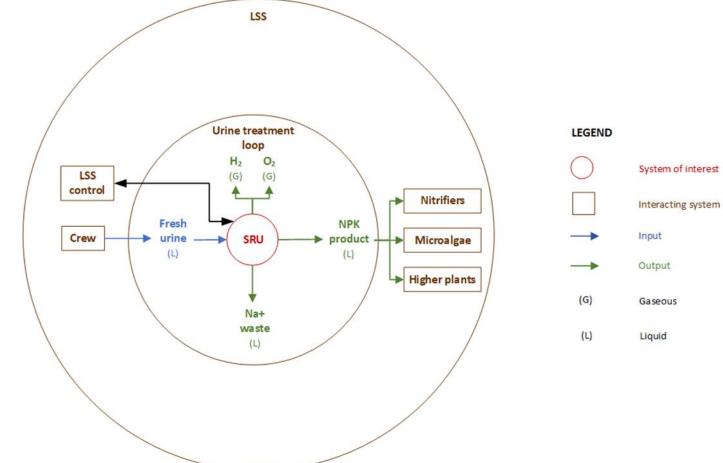
Context



Objectives

- Elaborate the requirements of a Salinity Reduction Unit (SRU)
- Review of relevant technologies to reduce salinity of urine
- Perform a trade-off of the technologies according to the ALISSE metric supported by trade-off tests
- Demonstrate the relevance of proposed processes through performance of a relevant test campaign
- Define future activities

Context diagram in the frame of a future LSS



BUILD ABOVE 5



Project Team



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<u>Prime contractor</u> Dries Demey – Project Manager/System Engineer Amanda Luther – Process Engineer Céline Coene – Process Engineer



<u>Sub-contractor</u> Nathalie Pujol – Studies & Realisation Engineer Jean-Christophe Lasserre – Project Manager & Process Engineer

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Sub-contractor

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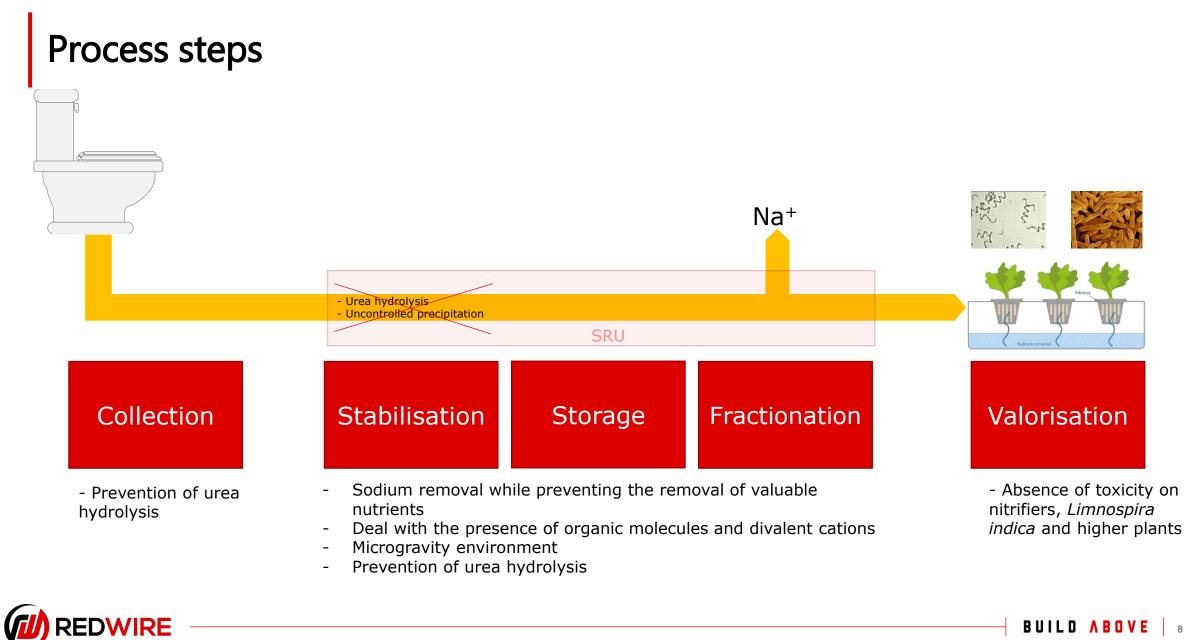




2. Requirements







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Urine collection

Aim: enable collection of <u>fresh</u>, <u>undiluted</u> and <u>non-hydrolysed</u> <u>urine</u>

| ID | system | category | subcategory | Requirement description | Requirement justification |
|-------|----------|----------|-------------|--|---|
| R0201 | SRU_COLL | FUN | PERF | The SRU shall use as an input non-diluted urine collected using no-flush urinals. | no dilution, as aim of project is to remove salts |
| R0202 | SRU_COLL | FUN | PERF | The collected urine shall be stored at 4°C prior to stabilization. | to prevent urea hydrolysis |
| R0203 | SRU_COLL | FUN | PERF | The collected urine shall not be hydrolyzed . | to prevent ammonia volatilization |
| R0204 | SRU_COLL | FUN | PERF | The pH of the collected urine shall be below 7.5 | |
| R0205 | SRU_COLL | FUN | PERF | The TAN (total ammonia nitrogen) concentration in the collected urine shall be below 500 mg N/L. | |



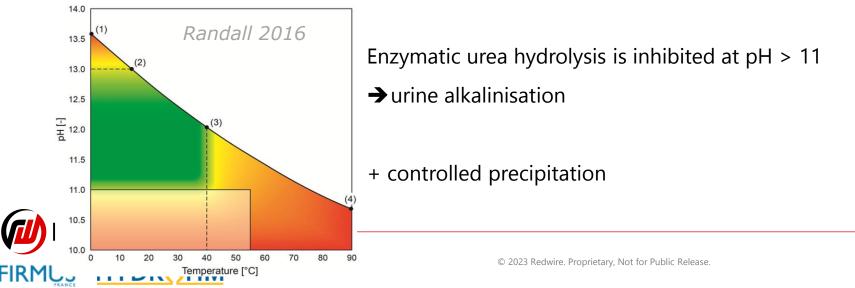
Urine stabilisation

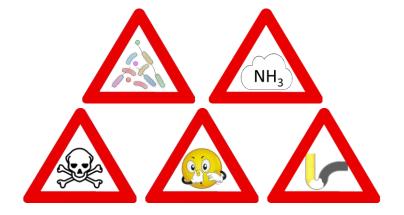
Aim: prevent urea hydrolysis during storage, shipment and fractionation

urease $CO(NH_2)_2 + 2H_2O \rightarrow NH_3 + NH_4^+ + HCO_3^-$

- Ammonia volatilization \rightarrow N loss + health risk
- Precipitation (pH increase) \rightarrow P loss

 \rightarrow stabilisation







BUILD ABOVE

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Urine stabilisation

Aim: prevent urea hydrolysis during storage, shipment and fractionation

| ID | system | category | subcategory | Requirement description | Requirement justification |
|-------|----------------------|----------|-------------|--|--|
| R0301 | SRU_STAB | FUN | PERF | The urine shall be stabilized through alkalinisation to prevent urea hydrolysis. | Compatible with nitrification |
| R0302 | SRU_STAB | FUN | PERF | The urine shall be alkalinized preventing addition of chemicals. | ALISSE criteria on mass and sustainability |
| R0303 | SRU_STAB | FUN | PERF | The alkalinized urine shall have a pH between 11.5 and 12.5 . | pH > 11 to prevent enzymatic urea hydrolysis, pH < 13 to prevent chemical urea hydrolysis |
| R0401 | SRU_STOR | FUN | PERF | The alkalinized urine shall be stored for max TBV months in a closed container . | |
| R0402 | SRU_STOR | FUN | PERF | The alkalinized urine shall be stored at max 25 ° C . | High temperature can promote urea hydrolysis |
| RED | NIRE — | | | | BUILD ABOVE |
| | YDR <mark>OHN</mark> | 1 | | © 2023 Redwire. Proprietary, Not for Public Release. | |

Urine fractionation

Aim: remove selectively sodium from the urine

| ID | system | category | subcategory | Requirement description | Requirement justification |
|-------|----------|----------|-------------|---|---------------------------|
| R0501 | SRU_FRAC | FUN | PERF | In case of fractioning of stabilised urine, the | |
| | | | | urine shall have a pH at least 11 before | |
| | | | | treatment in the SRU. | |

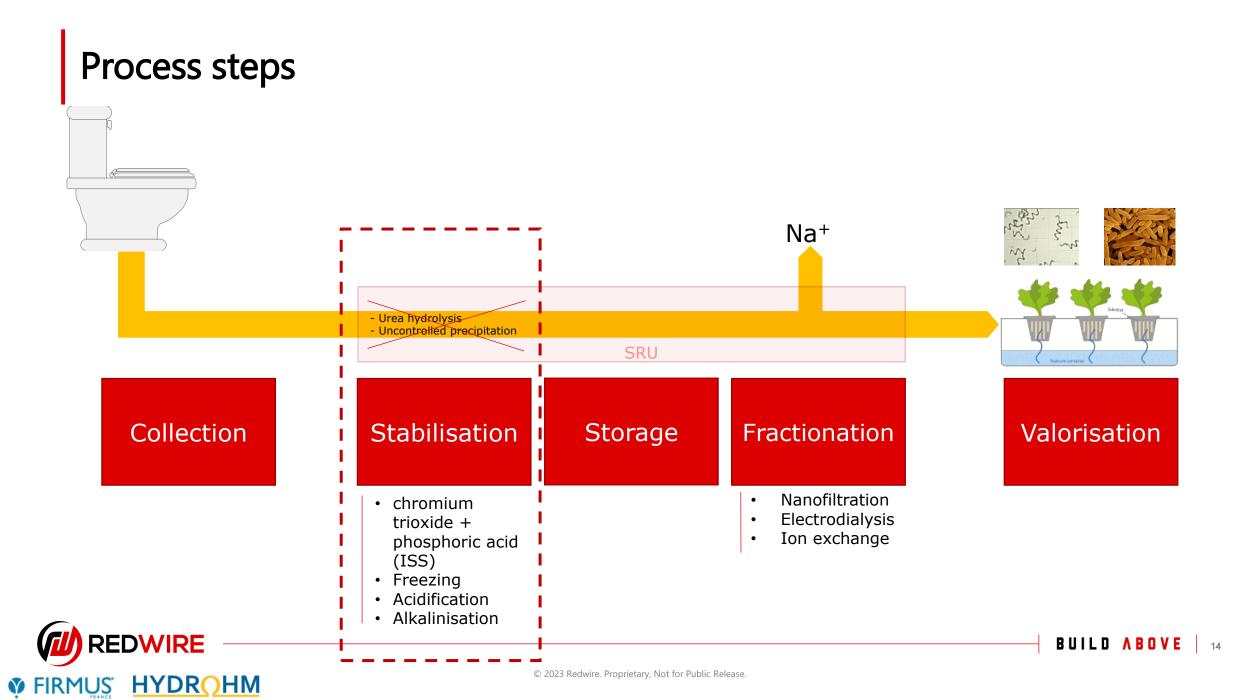


3. Technology Trade-off

3.1 Urine Stabilization

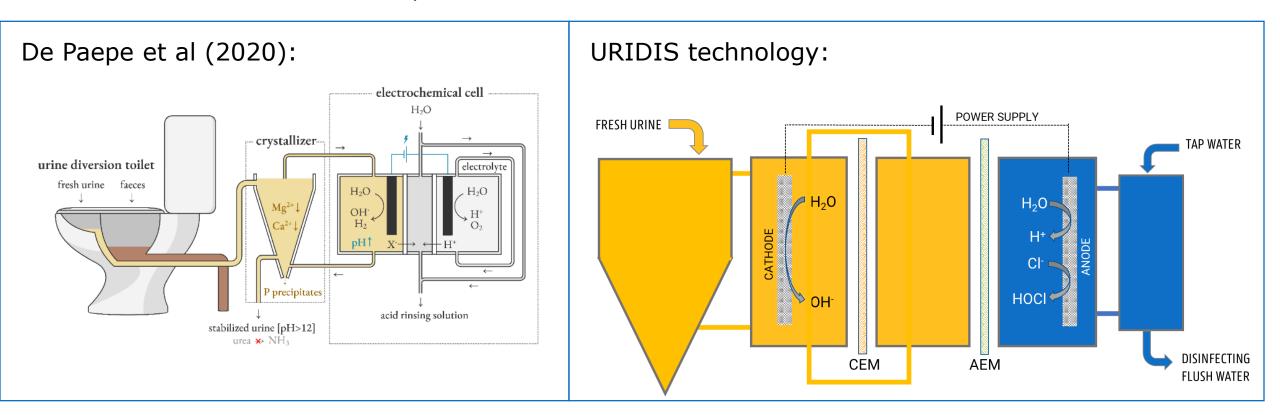






Urine stabilisation

Electrochemical stabilisation to prevent use of consumables







Trade-off testing: urine collection

- Urine collection in CAPTURE building
 - Research center and incubator of Ghent University

• Donors

- Employees and visitors of CAPTURE building
- Male (urinals)
- Age: ~20-65
- Panel-mounted waterless urinal with collection vessel \rightarrow undiluted urine
- Urine storage at 4°C prior to alkalinisation to prevent urea hydrolysis





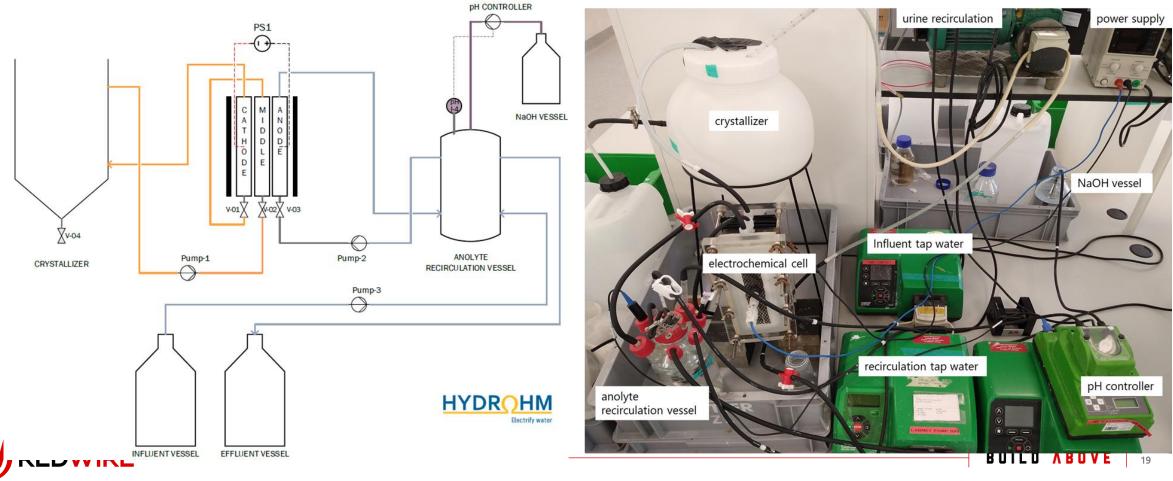




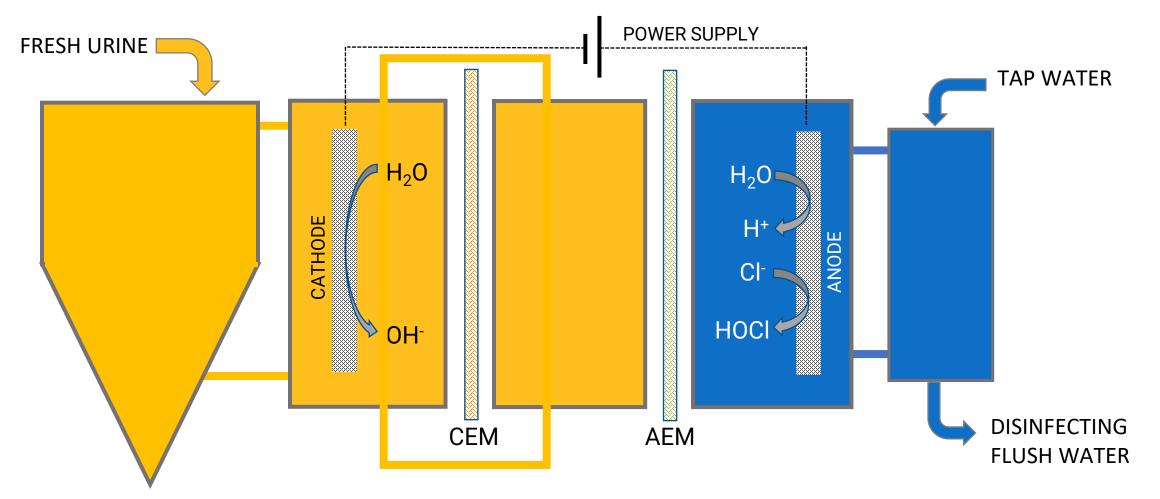
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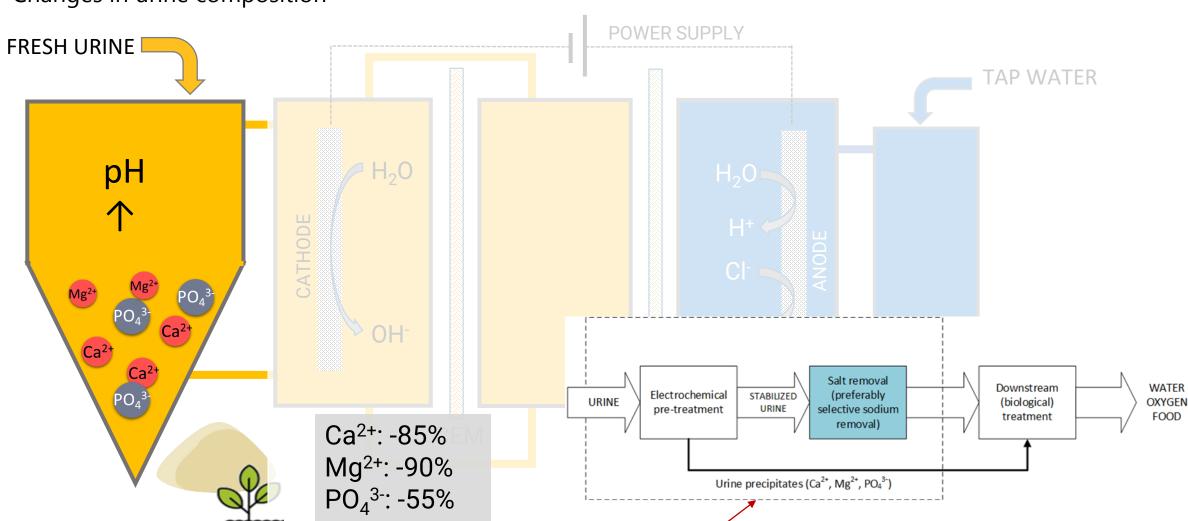
Lab setup

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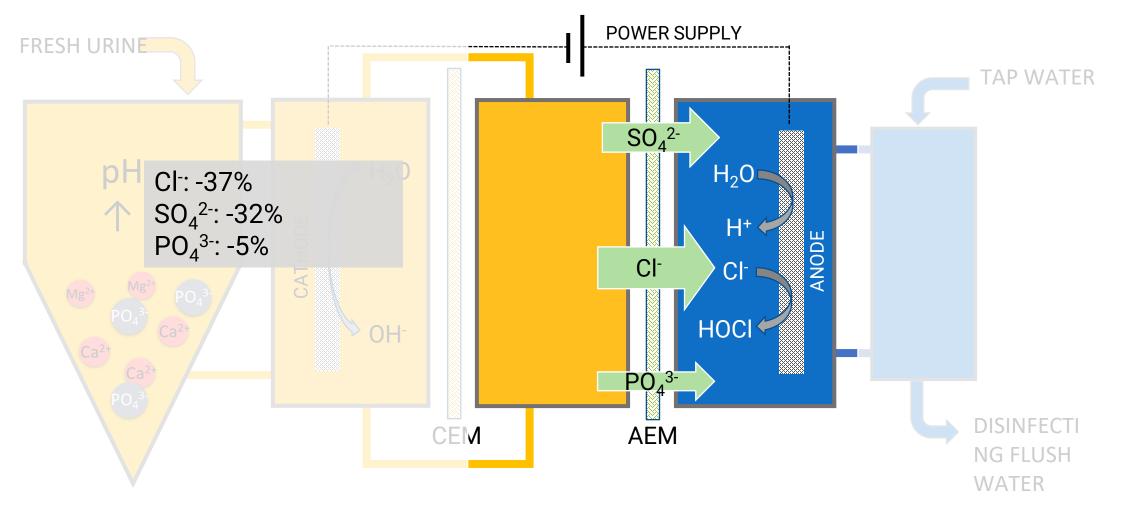
Changes in urine composition





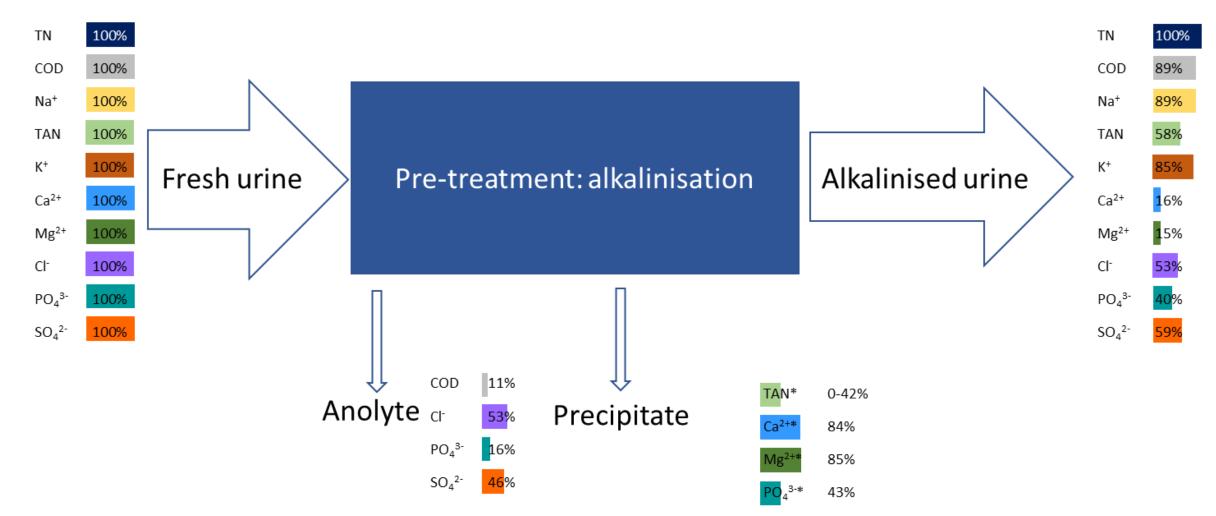
Changes in urine composition

Changes in urine composition



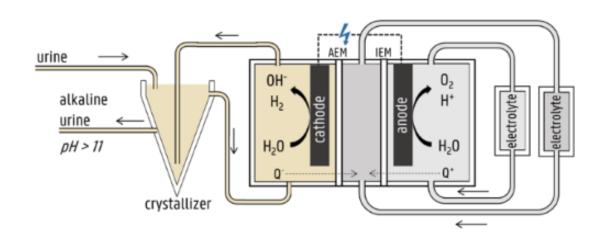
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Changes in urine composition

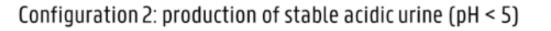


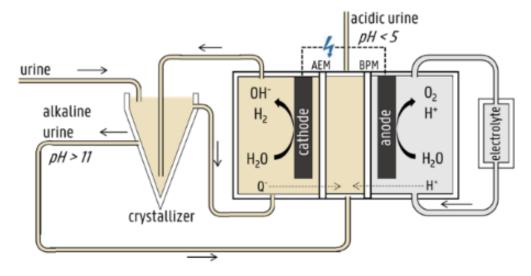
Trade-off testing: urine acidification

Acidification: only stable when part is acidified (remainder: alkalinized urine)



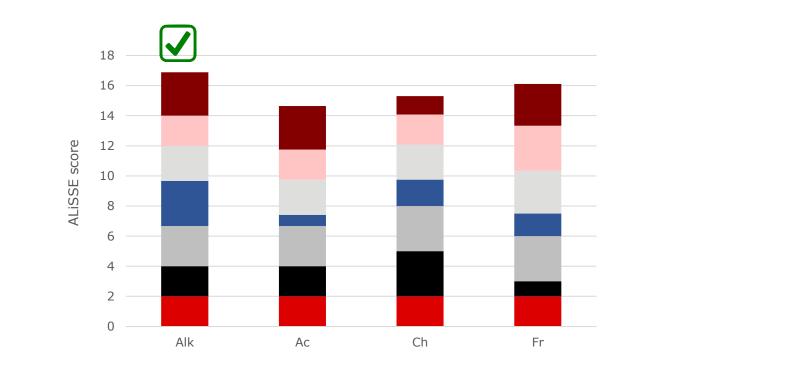
Configuration 1: production of stable alkaline urine (pH > 11)







Advanced Life Support System Evaluator (ALiSSE)



■ Physical mass ■ Energy and power ■ LSS crew time ■ Efficiency ■ Risk for human ■ Reliability ■ Sustainability

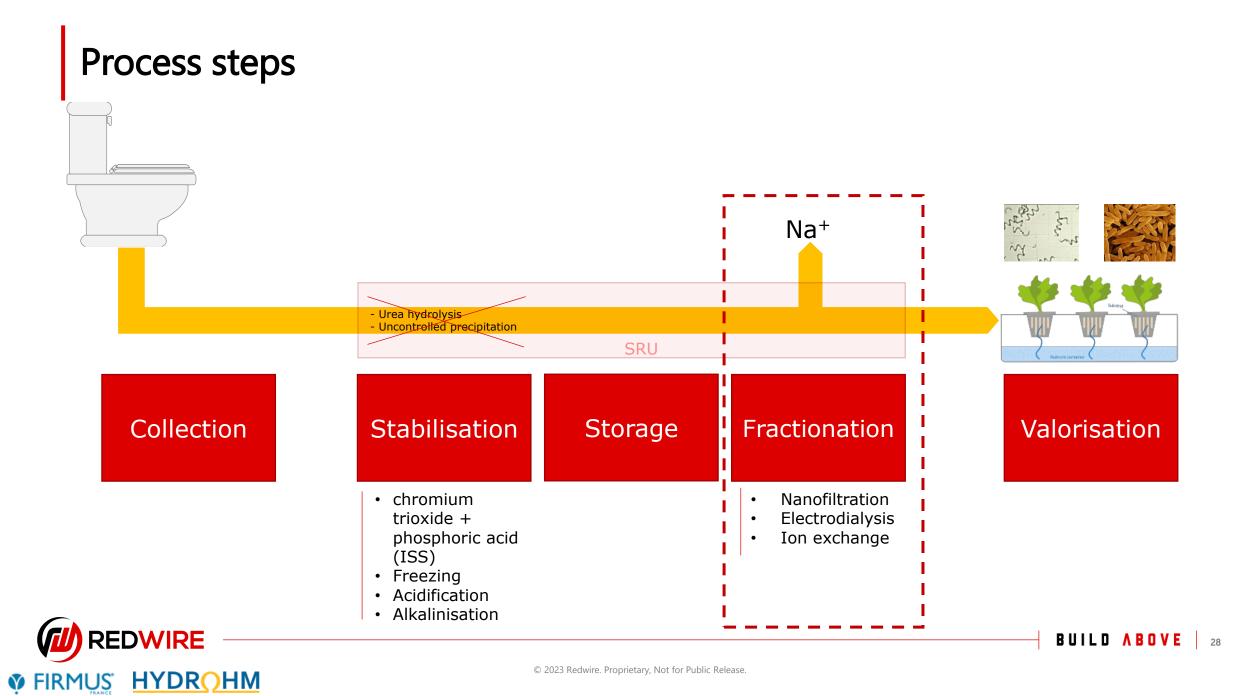


3. Technology Trade-off

3.2 Urine Fractionation

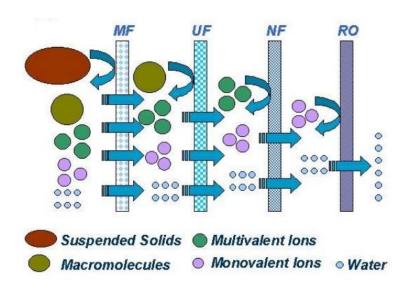






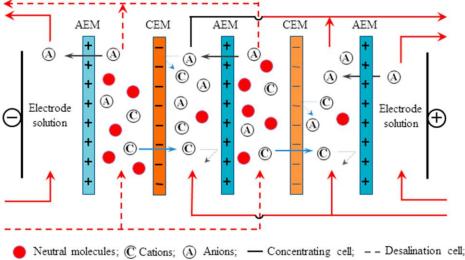
Principles of the technologies

Nanofiltration



- Cut-off threshold : 0,1-1 nm, 150/1000 g/mol
- Separation based on molecules size and electric charge
- Applied pressure: 4-20 Bar
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 FIRMUS' HYDROHM

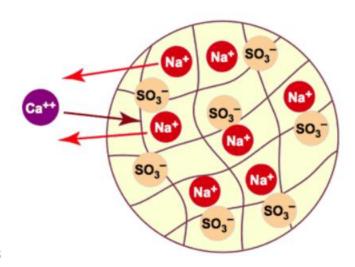
Electrodialysis



CEM: Cation exchange membrane; AEM: Anion exchange membrane;

- Ion extraction from a solution
- Current applied between anode and cathode
- Ion migration through selective membrane

Ion Exchange



- Reversible replacement of an ion by another
- Organic polymer resin (polystyrene sulfonate)
- Porous, large surface area
 - BUILD **ABOVE** 29

Urine fractionation – Nanofiltration testing

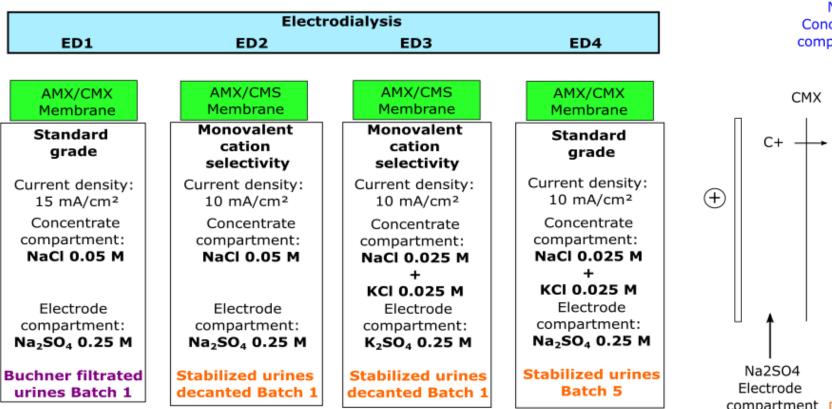
Nanofiltration Test 1 Test 2 Test 3 Test 4 Test 5 Test 6 Ceramic Membrane **Organic Membrane** KOCH MPF34 AMS A3014 **INOPOR LC1** Suez DL Suez DL ALSYS NF Plane organic Acidifed urines Tubular ceramic Plane organic Acidifed urines Tubular ceramic Thin-film composite Thin-film composite pH = 2.5Membrane: ZrO2 Membrane: TiO2 pH = 8.8pH range: 0-14 pH range: 0-12 Support: a-Al2O3 Plane organic Plane organic Support: a-Al2O3 Surface: 28 cm² Thin-film composite Surface: 28 cm² Thin-film composite pH range: 0-14 pH range: 0.3-13.4 RO water theoric flux: Surface: 38 cm² Water theoric flux: pH range: 3-9 pH range: 3-9 Surface: 34 cm² 2 L/h.m².bar 2.3 L/h.m².bar Surface: 28 cm² Water theoric flux: Surface: 28 cm² Water theoric flux: 200 Da 400 Da MgSO4 2000 ppm in MgSO4 2000 ppm in 9 L/h.m².bar >20 L/h.m².bar water theoric flux: 1000 Da 200 Da water theoric flux: Buchner filtrated 8.4 L/h.m².bar Stabilized urines 8.4 L/h.m².bar decanted Batch 2 urines Batch 1 150-300 Da Buchner filtrated 150-300 Da Stabilized urines urines Batch 1 non-decanted Batch 1 Stabilized urines Acidified urines decanted Batch 3 decanted Batch 2 acidified pH = 8.8 pH = 2.5

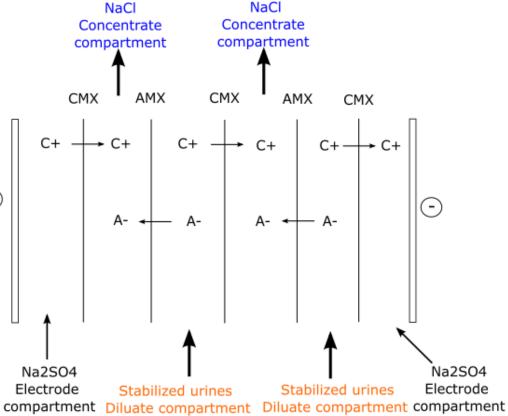


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Urine fractionation – Electrodialysis tests



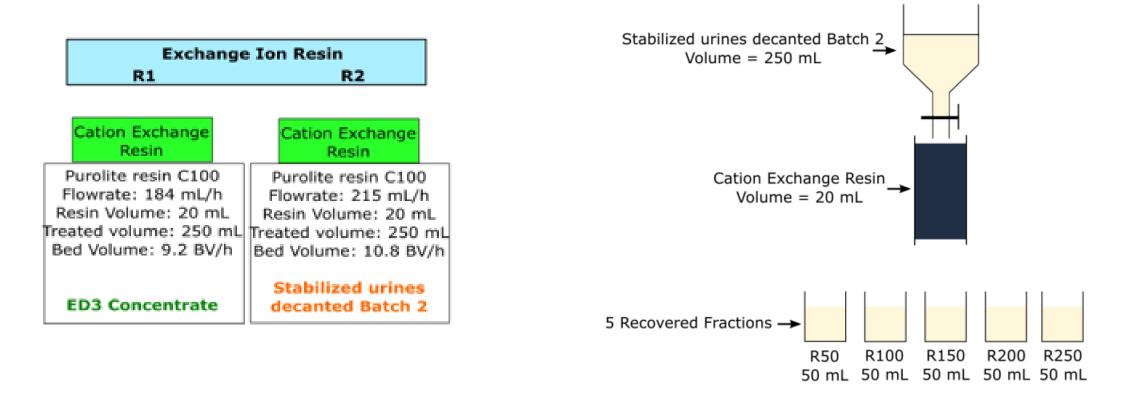


Description of electrodialysis module



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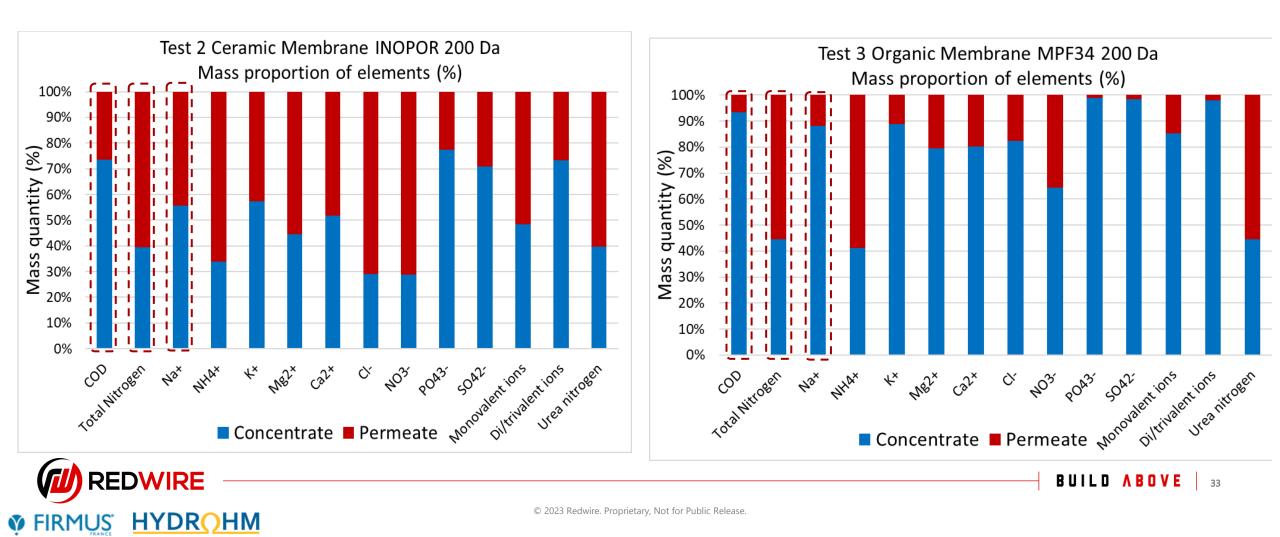
Urine fractionation – Ion Exchange Resin tests





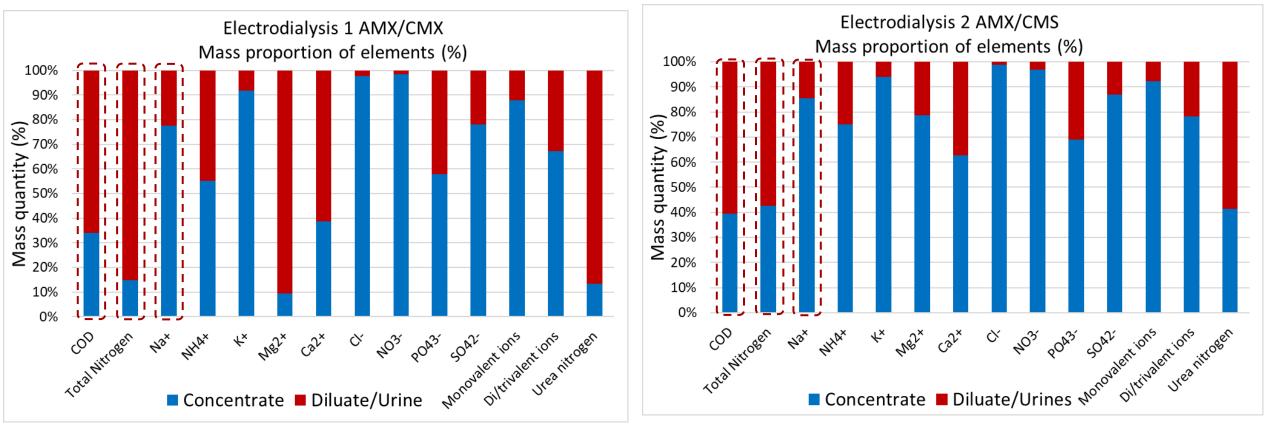
Urine fractionation – Nanofiltration tests results

• No separation salts/COD



Urine fractionation – Electrodialysis tests results

- Majority of monovalent salts go on the concentrate.
- About minimum 60 % of COD and total nitrogen remains on the urine.



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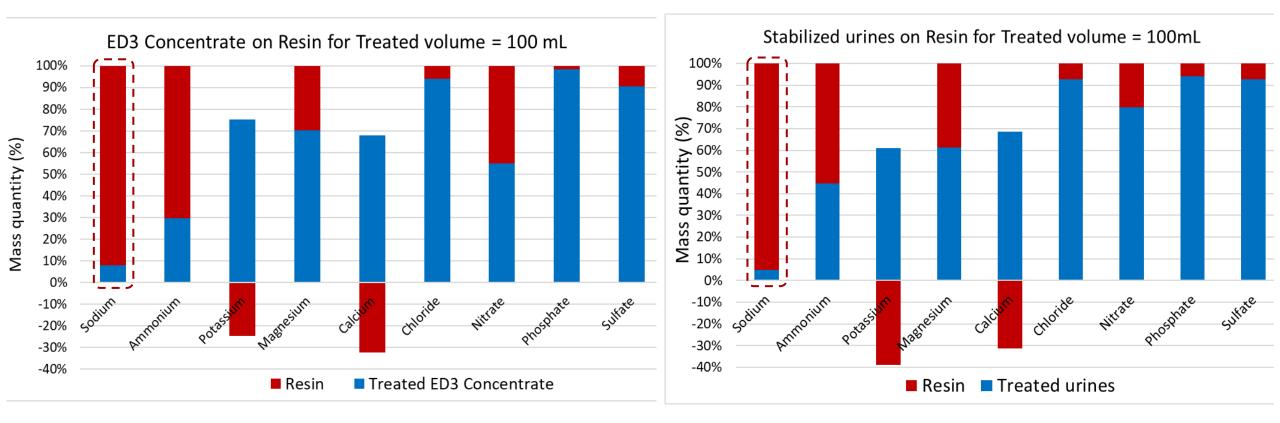
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Urine fractionation – Ion exchange resin tests results

 More than 90 % of sodium is removed from the treated solution (ED3 concentrate and stabilized urine)

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Urine fractionation

Combination of ED and IX for feasibility tests

Scenario 1: IX then ED

- IX to remove sodium from the urine
- ED to separate COD/total nitrogen from salts

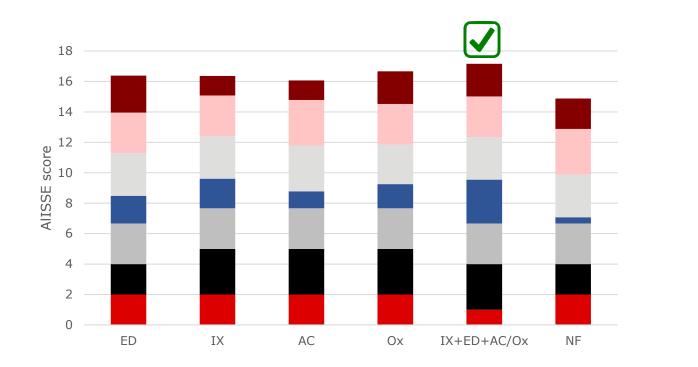
Scenario 2: ED then IX

- ED to separate COD/total nitrogen from salts
- IX to separate sodium from other salts





Advanced Life Support System Evaluator (ALiSSE)



■ Physical mass ■ Energy and power ■ LSS crew time ■ Efficiency ■ Risk for human ■ Reliability ■ Sustainability







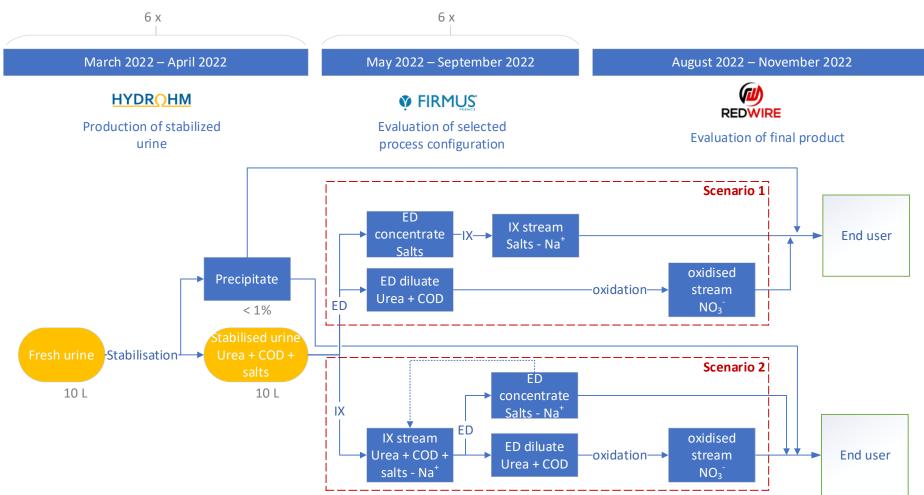




Scenarios and general test plan

• Scenario 2 as baseline:

- Best selective Na⁺
 removal from urine
 (primary objective)
- Higher ALiSSE score than scenario 1 for sustainability and power consumption
- Scenario 1 as back-up (if issue with COD and IX)





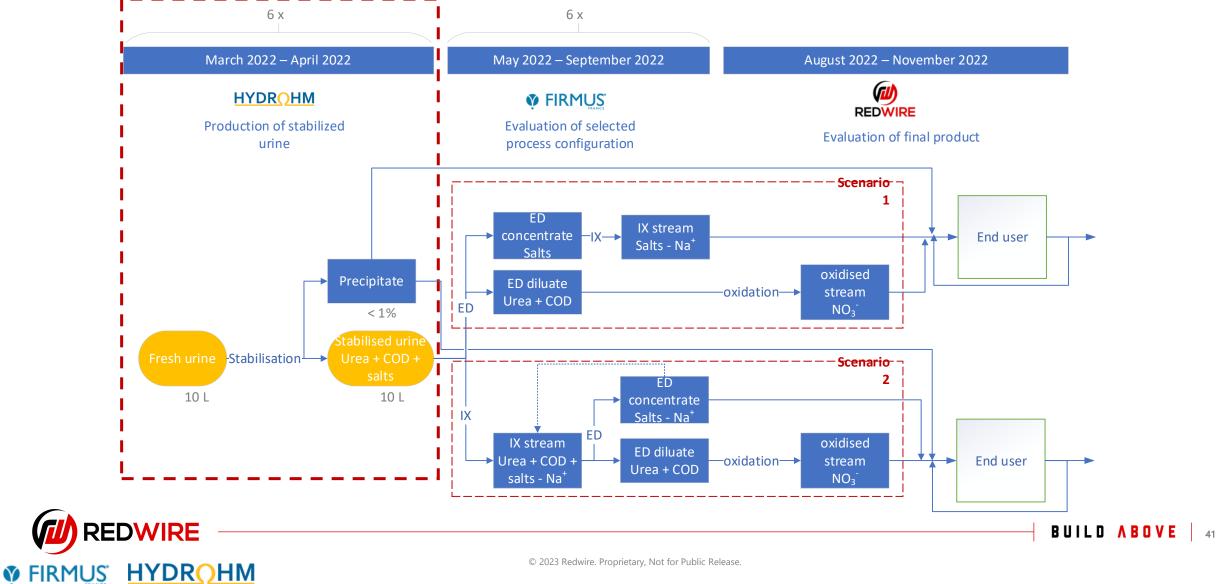
4. Feasibility tests

4.1 Urine Collection and Stabilization





4.1 Urine collection and stabilisation



Urine collection and stabilisation

- Urine collection: same approach as trade-off testing
- Urine stabilization: electrochemical alkalinization
 - Between March 2022 and June 2022
 - 6 batches for feasibility testing
 - Stability of batches (TN139.3.2)
 - By comparing composition
 HYDROHM/FIRMUS
 - Minor differences

DWIRE

V FIRMUS

HYDROHM

- Slight decrease in pH (0,2-0,7 pH units) (CO₂ dissolution from headspace)
- No substantial increase in TAN or TAN/TN

| Parameter | Unit | BATCH 1 | BATCH 2 | ВАТСН З | BATCH 4 | BATCH 5 | BATCH 6 |
|--------------------------------------|---------------|---------|---------|---------|---------|---------|---------|
| рН | - | 11.82 | 12.26 | 11.94 | 12.09 | 12.19 | 12.25 |
| EC | [mS/cm] | 10.50 | 10.26 | 9.93 | 10.75 | 10.38 | 9.34 |
| COD | [mg COD/L] | 4730 | 3960 | 4240 | 4380 | 4010 | 3680 |
| TN | [mg TN/L] | 5400 | 4470 | 5500 | 5250 | 4260 | 3790 |
| Cl- | [mg/L] | 1311 | 1180 | 1084 | 1096 | 870 | 722 |
| NO ₂ - | [mg/L] | 5 | 4 | 4 | 1 | 4 | 3 |
| NO ₃ - | [mg/L] | 11 | 14 | 21 | 17 | 7 | 6 |
| PO ₄ ³⁻ | [mg/L] | 528 | 430 | 507 | 479 | 426 | 478 |
| SO ₄ ²⁻ | [mg/L] | 590 | 470 | 516 | 484 | 444 | 479 |
| Na ⁺ | [mg/L] | 1662 | 1562 | 1537 | 1685 | 1626 | 1493 |
| NH ₄ ⁺ | [mg/L] | 534 | 403 | 410 | 421 | 492 | 423 |
| K + | [mg/L] | 1960 | 1643 | 1724 | 1988 | 1872 | 1555 |
| Ca ²⁺ | [mg/L] | 10 | 11 | 75 | 55 | 40 | 38 |
| Mg ²⁺ | [mg/L] | 7 | 10 | 13 | 10 | 8 | 7 |



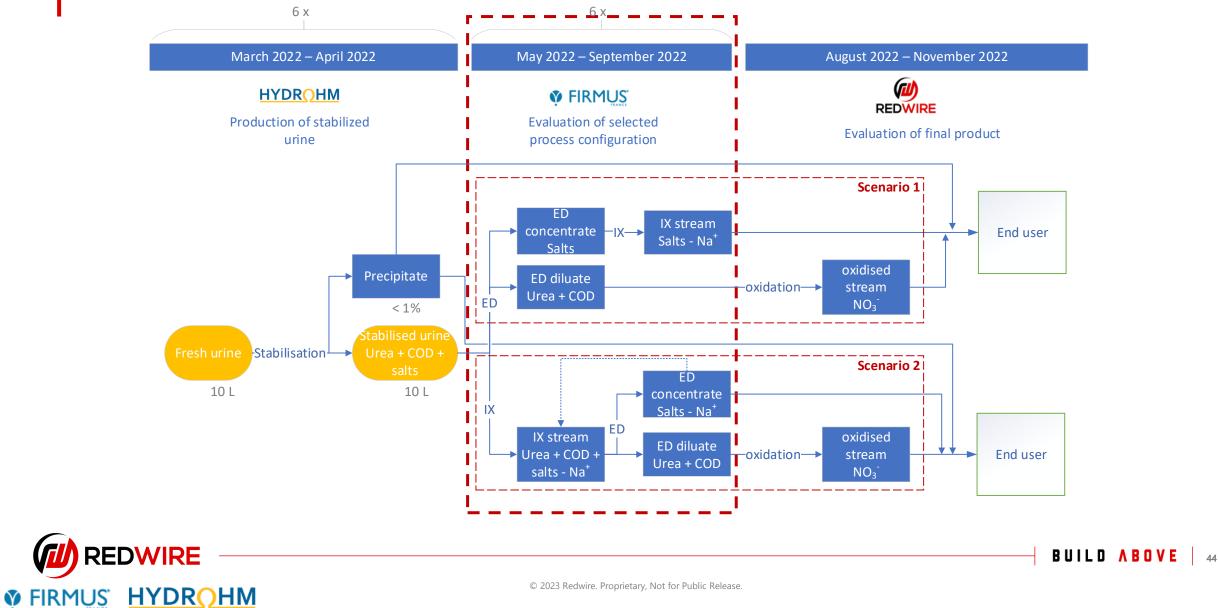
4. Feasibility tests

4.2 Urine Fractionation



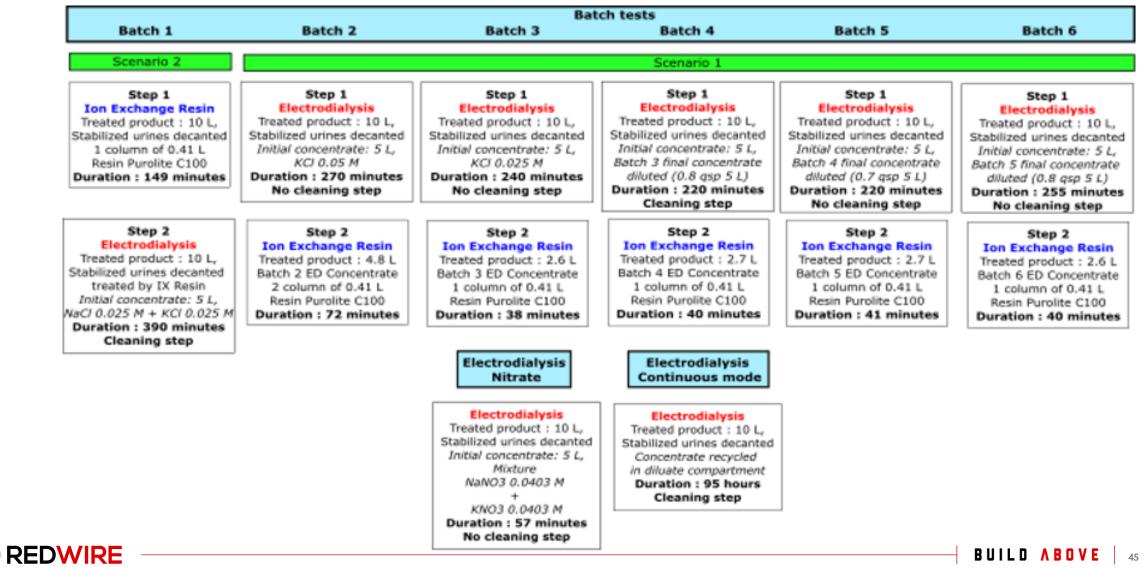


4.2 Urine fractionation



4.2 Urine fractionation

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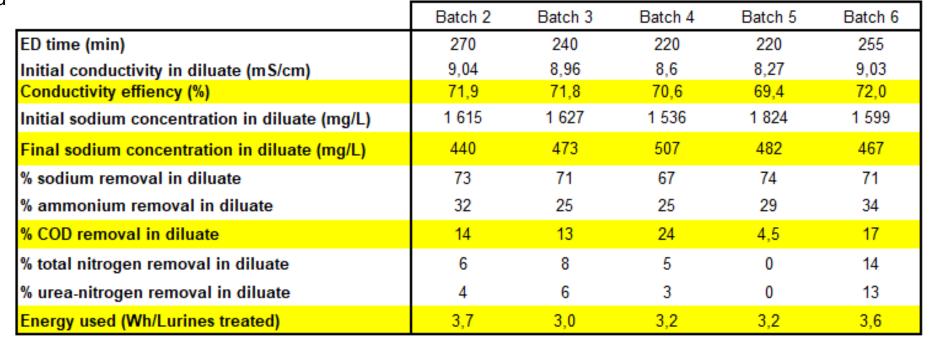


Electrodialysis:

- 6 Batches realized
- Similar results for the 6 Batches
- About 70 % of sodium removed
- Final sodium concentration about 470 mg/L
- Energy used : about
- 3.2 Wh/Lurines treated

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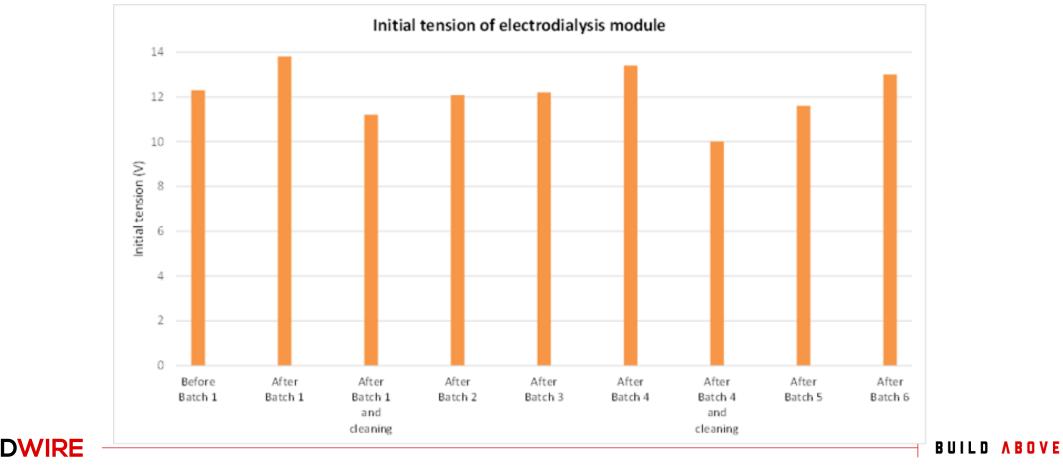
Electrodialysis tests results

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• Initial tension does not increase significantly after 6 Batches.

FIRMUS HYDROHM

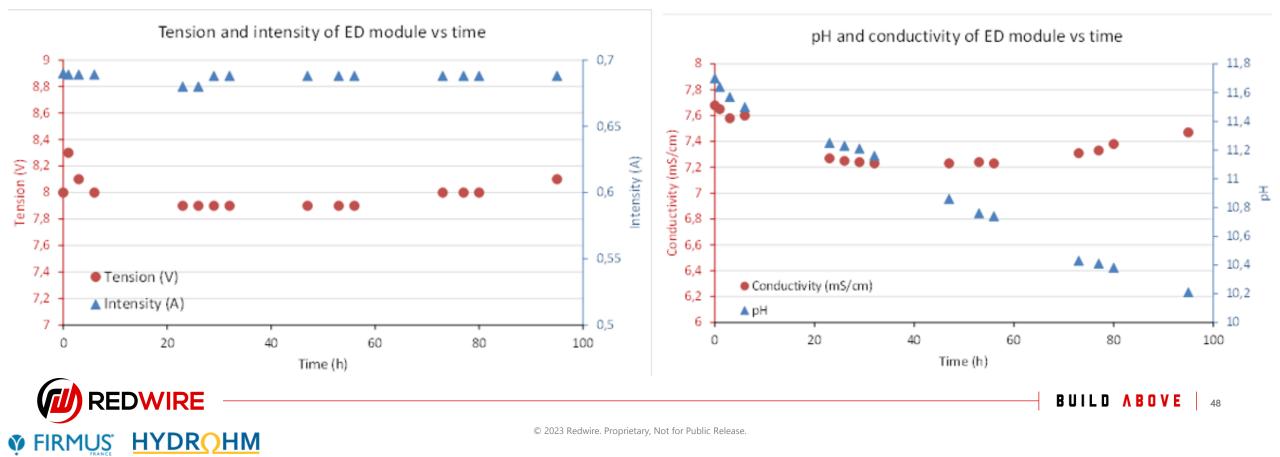
• Cleaning step allows to recover initial values of initial tension during salt tests



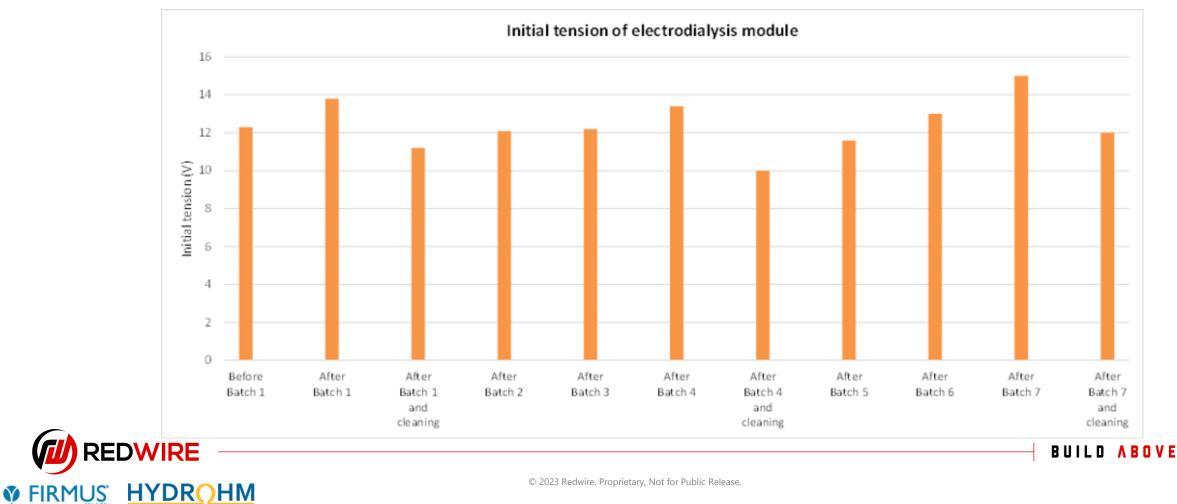
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- ED module in mode "recycling" during 95 h (4 days)
- Tension, conductivity and current intensity remain stable
- pH decreases from 11.8 to 10.2 (dissolution of air-CO₂ in urine)



- Increase of 2 V for salt tests' initial tension
- After cleaning step: Recovery of initial tension value before ED test



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4.2 Urine fractionation – IX step

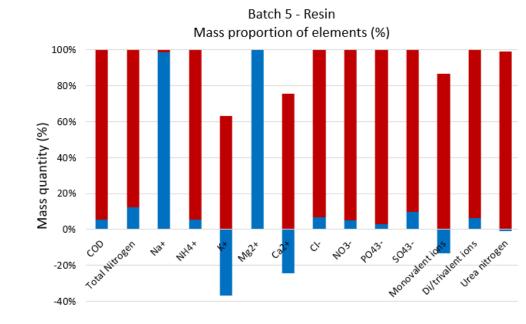
• 98 % of sodium removal

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- No adsorption of ammonium and total nitrogen
- Final potassium concentration : ~7 500 mg/L
- Final sodium concentration : ~50 mg/L



Resin

Diluate (urines)

| | Batch 2 | Batch 3 | Batch 4 | Batch 5 | Batch 6 |
|--------------------------------------|---------------|---------------|---------------|--------------|---------------|
| Treated volume (L) | 4,8 | 2,6 | 2,7 | 2,7 | 2,6 |
| % COD removal | Released/20 % | Released/18 % | Abdorbed/38 % | Adsorbed/5 % | Adsorbed/11 % |
| Final potassium concentration (mg/L) | 8 909 | 7 885 | 7 549 | 7 015 | 7 623 |
| Initial sodium concentration (mg/L) | 2 797 | 2 588 | 2 671 | 2 481 | 2 712 |
| Final sodium concentration (mg/L) | 126 | 48 | 42 | 37 | 62 |
| % sodium removal | 95 | 98 | 98 | 99 | 98 |
| % ammonium removal | 46 | 37 | 0,8 | 5,4 | 37 |
| % total nitrogen removal | 15,4 | 8,9 | 13,6 | 12,5 | 5,4 |
| % urea-nitrogen removal | 0 | 0 | 9,7 | 0 | 0 |
| | | | | | |

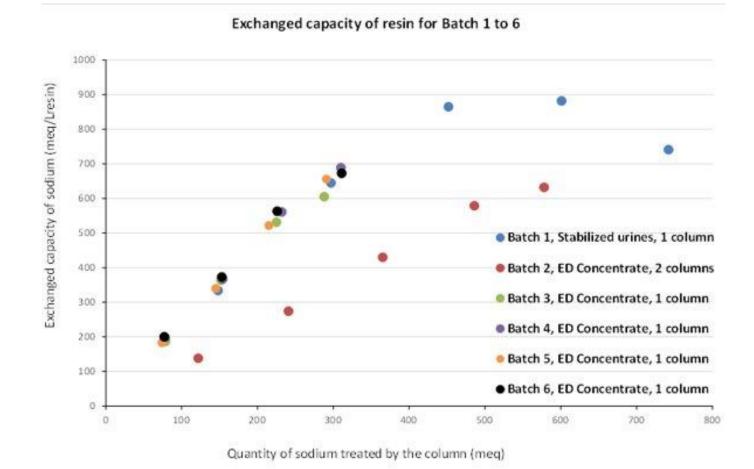


4.2 Urine fractionation – IX step

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• Optimal regeneration on 6 batches: no decrease of resin's exchange capacity



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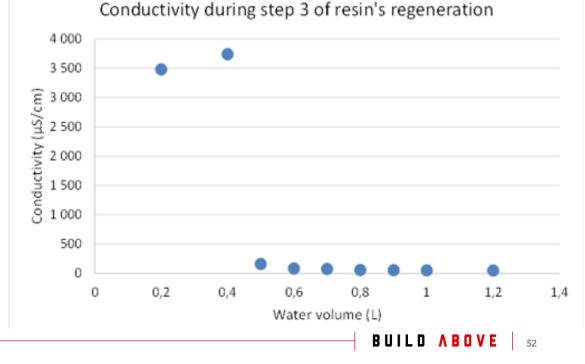
BUILD ABOVE

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4.2 Urine fractionation – IX step

| | BV/h | Flowrate | Time | Volume | BV |
|--|------|----------|-------|--------|------------|
| | | (L/h) | (min) | (L) | (L/Lresin) |
| Optimization step 1 Regeneration KCI 106 g/L | 4 | 1,6 | 22 | 0,6 | 1,5 |
| Optimization step 2 Slow rinse DM water | 2 | 0,8 | 22 | 0,3 | 0,7 |
| Optimization step 3 Final rinse DM water | 15 | 6,2 | 10 | 1,0 | 2,4 |

- Resin's regeneration : 3 steps
- Optimization of third step: 0.6 L instead of 1 L
- Globally, resin's regeneration requires 1.5 L of water





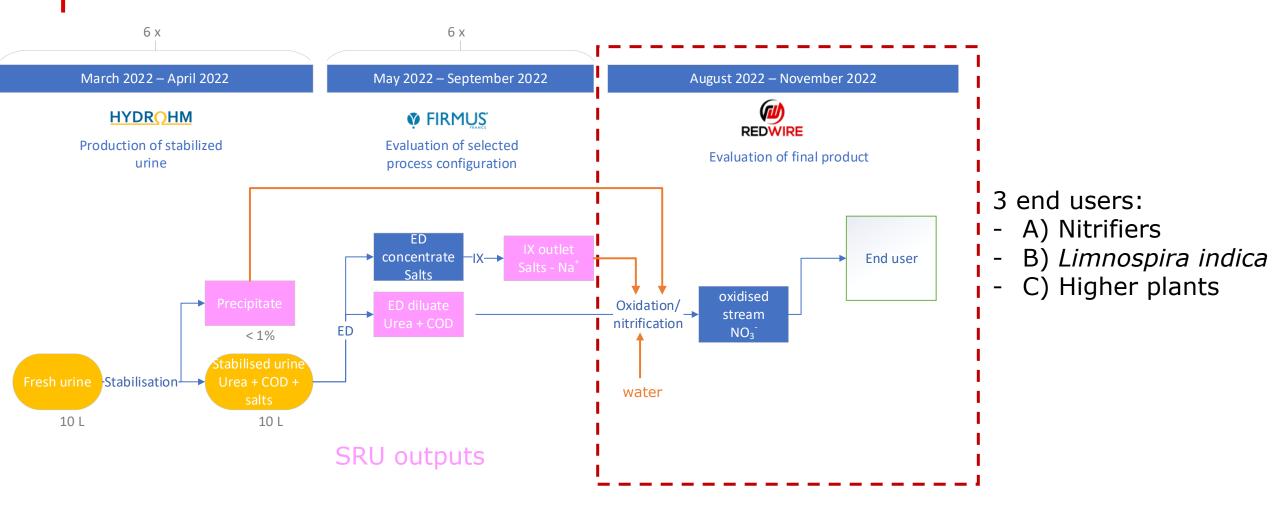
4. Feasibility tests

4.3 Urine Valorization



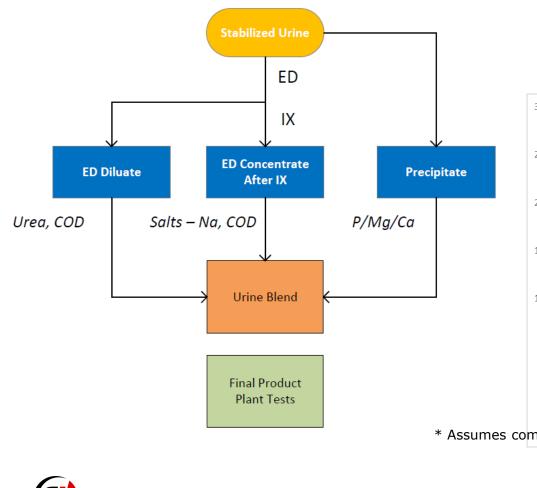


4.3 Urine valorisation



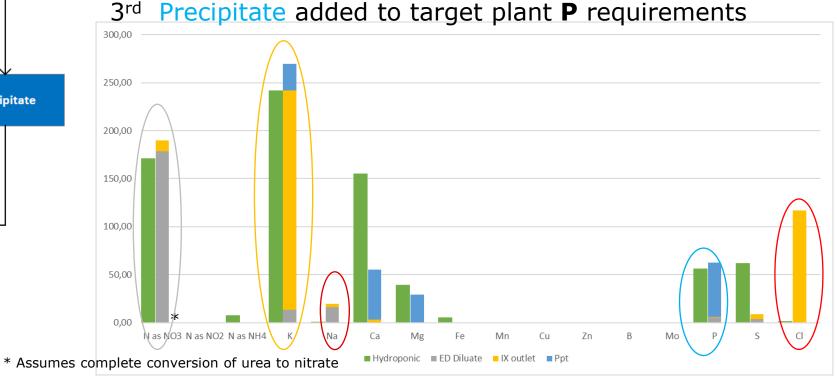


4.3 Urine valorisation: nitrification to produce final product for plant toxicity tests



1st ED Diluate diluted to target plant **N** requirements

2nd IX outlet diluted to target plant **K** requirements



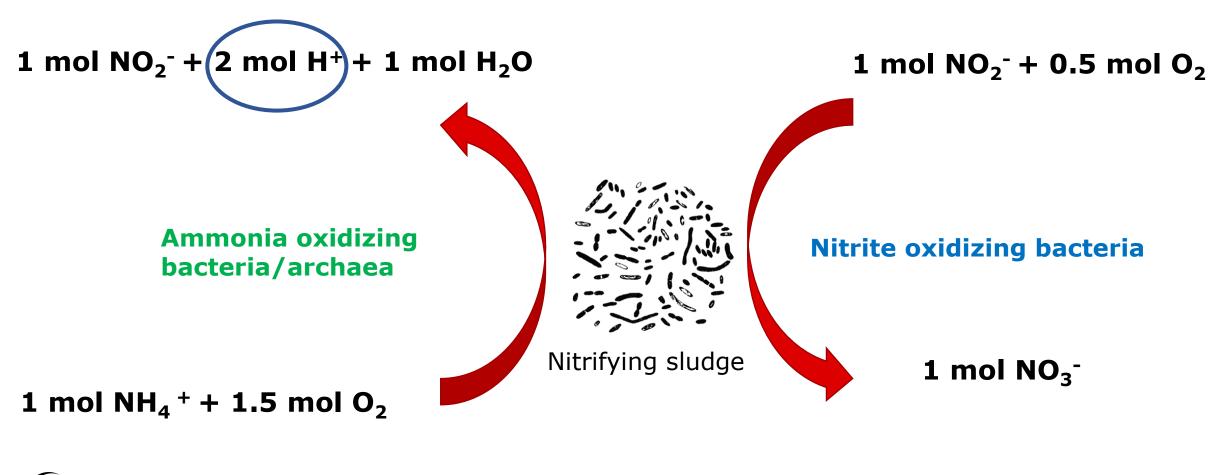
interface meeting #3| November 202

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Na and Cl remain high



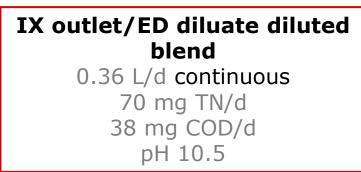
4.3 Urine valorisation: nitrification principle



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4.3 Urine valorisation: nitrification reactor

Inputs



- IX outlet to remove excess COD

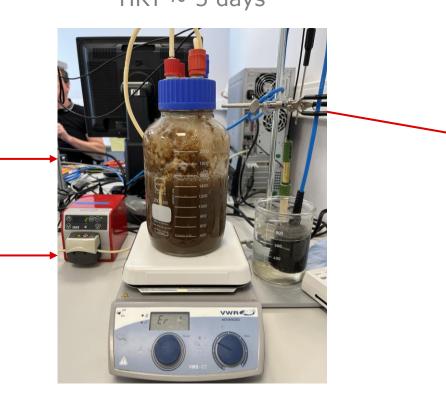
- Dilute blend before nitrification for speed

Stabilization precipitate

0,24 g/d *added to bioreactor 3 x per week Provides some alkalinity P/Ca/Mg/K



Continuous Fed Batch Operation (Continuous feeding – Effluent decanted 3x/week) 1-2L volume during operation HRT ~ 5 days

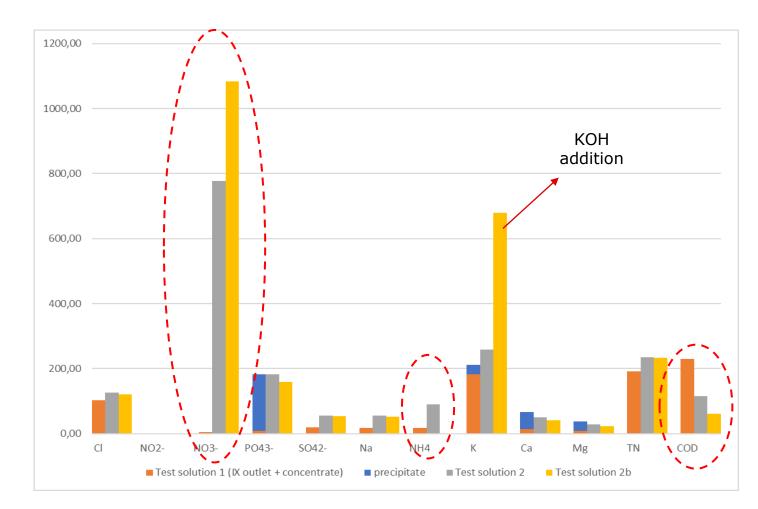


Outputs

Decanted effluent: (partially) nitrified urine blend 0.34 L/d discontinuous Monitor: ammonia/Nitrate/Nitrite/ pH Full analysis only for final products

4.3 Urine valorisation: nitrification products for toxicity tests

- Partially nitrified product collected during reactor operation
 - 70% Nitrate/30% ammonia
 - 50% COD removal
- Fully nitrified product batch produced by addition of base (KOH) to reactor
 - 100% nitrate
 - 73% COD removal





4.3 Urine valorisation: Higher plants - test plan

| | Media to be tested |
|-------------|--|
| Control (+) | Demi water – recommended control kit procedure |
| Control (+) | Hydroponic media (model media for urine blend) |
| Exp media 1 | Effluent from partial nitrification: ammonium+ nitrate |
| Exp media 2 | Effluent from full nitrification: nitrate + KOH |

| | Plants to be tested |
|-----|---------------------------------|
| SOS | Sorghum saccharatum (Sorgho) |
| LES | Lepidium sativum (garden cress) |
| SIA | Sinapis alba (mustard) |



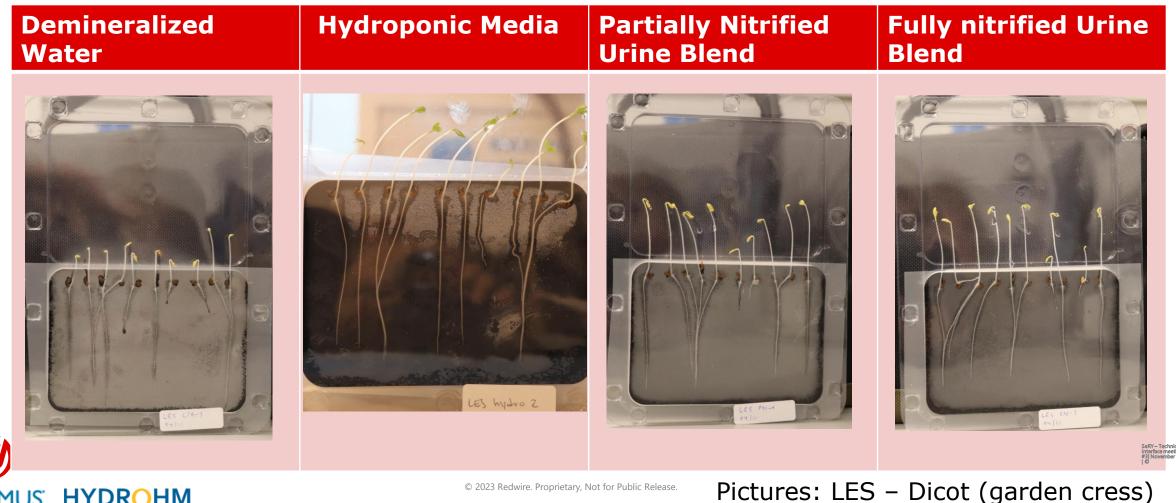


4.3 Urine valorisation: Higher plants- use of Phytotoxkit

- Test plates filled with filter paper spiked with the test solution, seeds on top, incubate 3 days
- Data: Count number of germinated seeds, measure length of roots and shoots

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4.3 Urine valorisation: Higher plants - results

| Inhibition vs DM water control | % Inhibition Germination | | | % Inhibition Root Growth | | | % Inhibition Shoot Growth | | |
|---|--------------------------|-----|-----|--------------------------|-----|-----|---------------------------|-----|-----|
| | LES | SIA | SOS | LES | SIA | SOS | LES | SIA | SOS |
| Hydroponic Media | 0 | 0 | 0 | -8 | 3 | -11 | -85 | -75 | -29 |
| Nitrification effluent – partial nitrification WK46 | 0 | 10 | 3-0 | -2 | 10 | 5 | -85 | -40 | 0 |
| Nitrification effluent – full nitrification WK46 | 0 | 3 | 3-4 | 2 | 18 | 15 | -76 | -53 | 3 |

- % Inhibition = (length control length urine)/length control * 100 where length is the lengths of root/shoot
- A negative value indicates a positive effect compared to the control
- No inhibitory effect from hydroponic media control, shoot growth appears to be better
- (Partially) nitrified urine seems to boost shoot growth such as hydroponic media, but negative effect on germination and root growth
- No clear difference between partial and full nitrification





4.3 Urine valorisation: Limnospira - test plan

| | Media to be tested |
|-------------|---|
| Control (+) | Zarrouk Medium |
| Test 1 | Partially nitrified effluent : ammonium+ nitrate |
| Test 2 | Fully nitrified effluent: nitrate + KOH |
| Test 3 | Urine blend (ED diluate + IX outlet) – without dilution and without nitrification |
| Test 4 | IX outlet |
| Test 5 | ED diluate |
| Test 6 | Ammonium chloride |

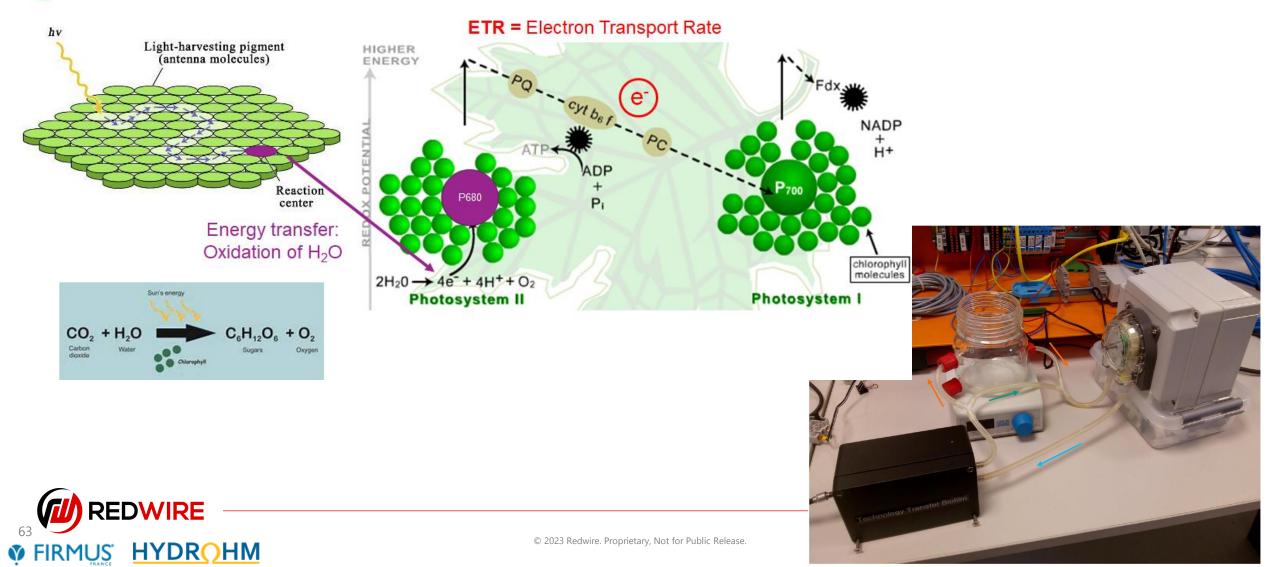
Note: The tests were done on 100 mL *L. indica* culture





4.3 Urine valorisation: Limnospira - PAM (Pulse-amplitude modulated fluorescence)

Y_m = Maximum Yield = Maximum available reaction centers



4.3 Urine valorisation: Limnospira - test results

| Number | Test solution | COD (mg/L in culture) | Ammonia (mg/L in culture) | Effect on ETR/Ymax |
|--------|---|-----------------------|----------------------------|--------------------------------------|
| Test 1 | Partially nitrified effluent | (contamination) | 8-15 | Positive |
| Test 2 | Fully nitrified effluent | 6-10-14-21 | 0 | Positive |
| Test 3 | Mix of ED diluate and IX outlet (same ratio as urine blend) | 224-416 | 19-36 | Negative |
| Test 4 | IX outlet | 59 | 5 | Positive |
| Test 5 | ED diluate | 408-617 | 34-51 BUT 150-300 measured | Negative |
| Test 6 | Ammonium chloride | 0 | 54-99-151 | Negative from 100 mg/L approx. |

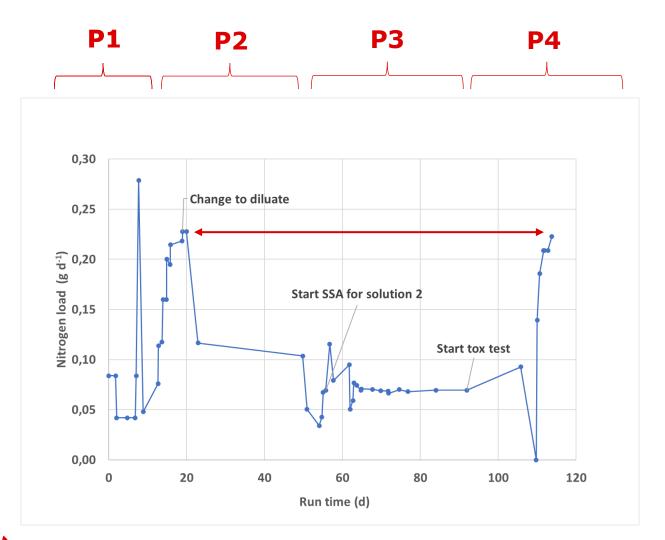
- No toxicity when product is diluted/(partially) nitrified
- Toxicity of ED diluate (non diluted)
- Probable source of toxicity: ammonia, COD





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4.3 Urine valorisation: Nitrification toxicity



REDWIRE

HYDROHM

FIRM

Nitrite accumulation as indication of toxicity

P1 – Baseline – ammonium/acetate

 ✓ No nitrite accumulation observed under ammonium/acetate feeding

P2 – Transition to Diluate – Crash&Recovery

- Concurrent with change in feed
- Nitrite quickly accumulated
- Too sudden transition/Nutrient limitation?
- ✓ Acute toxicity observed
- ✓ Activity recovered

P3 – Production of (partially) nitrified effluent

- ✓ Feed solution 1 + ppt
- ✓ Steady state achieved
- ✓ Stable partial + complete nitrification

BUILD ABOVE

P4 – nitrifier toxicity test

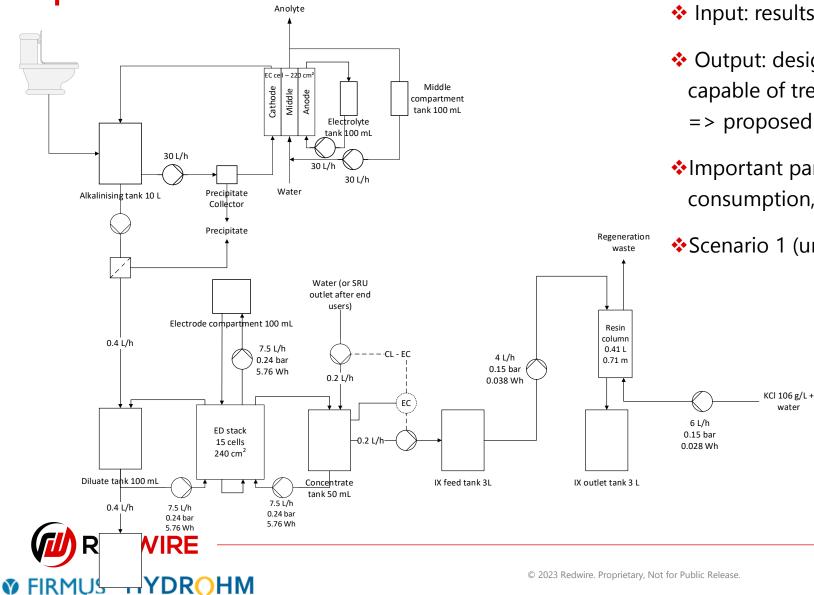
- ✓ no nitrite accumulation
- ✓ Able to reach control load

5. Proposed scenario





Proposed scenario



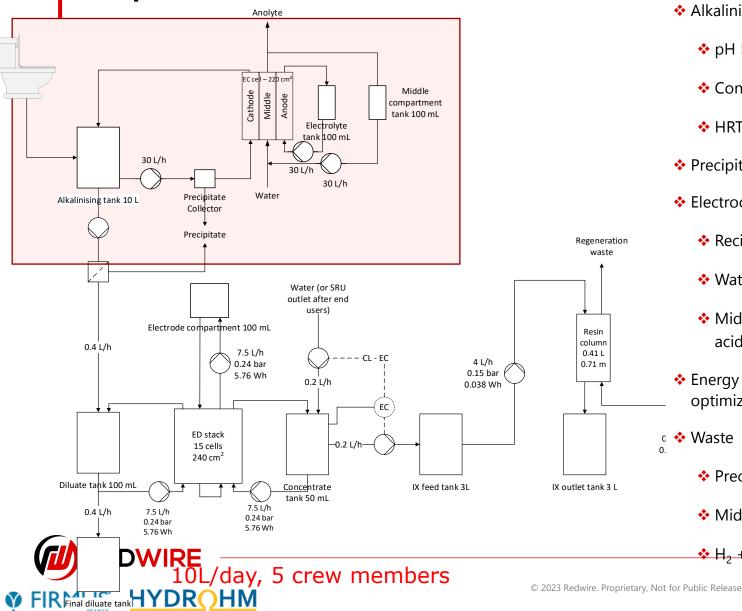
- Input: results from feasibility tests
- Output: design a salinity reduction unit (SRU)
 capable of treating 10 L urine/day (5 crew members)
 => proposed scenario
- Important parameters: physical mass, energy, power consumption, sustainability

BUILD ABOVE

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Scenario 1 (urine stabilisation = ED = IX = SRU)

Proposed scenario: alkalinisation



- Urine is collected in alkalinising tank
- Alkalinising tank (=crystallizer in our setup) functions as buffer
 - ✤ pH > 11,5
 - Continuous flow to ED
 - HRT~1 day (10L of urine per day)
- Precipitate collector to retain precipitate (no settling in space)
- Electrochemical cell 220 cm² (margin: factor 5)
 - Recirculation over cathode to keep pH > 11,5
 - ◆ Water oxidation at anode ⇔ URIDIS: chloride oxidation (safety!)
 - * Middle compartment: water or liquous waste stream as input \rightarrow turned into acid (waste or product)

BUILD ABOVE

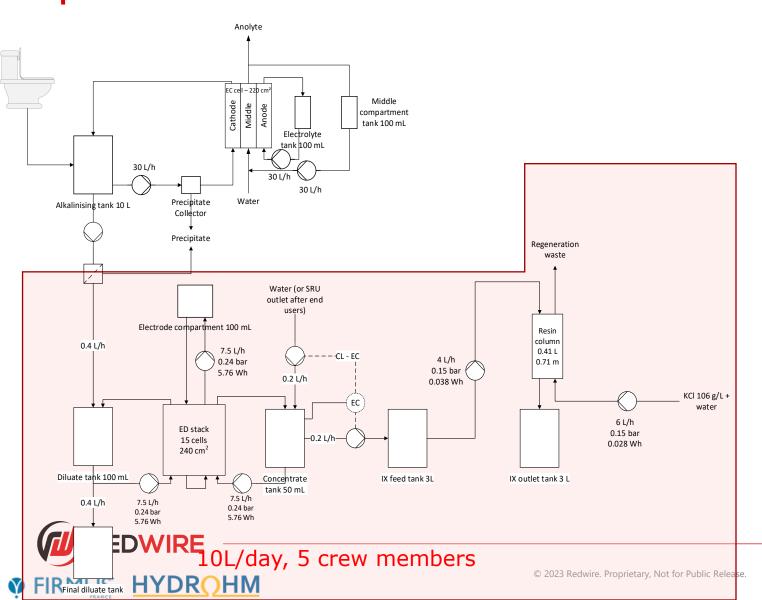
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- Energy input: 0,45-0,6 kWh (=electrode power consumption of lab setup, not optimized cell design)
- 🛛 💠 Waste

♦ H₂ + O₂

- Precipitate = resource of P
- Middle compartment electrolyte = acid

Proposed scenario: fractionation



- 0.4 L urine treated by hour
- ED stack: 15 cells, 240 cm²
- Water to "deconcentrate" the concentrate compartment
- IX processed by batch of 2.7 L in 40 minutes

BUILD ABOVE

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- IX feed tank: 3 L
- About 13 hours to fill IX tank
- About 55 minutes to regenerate resin
- Only 1 column is required

6. Conclusions and future work





Conclusions

- Demonstration of the adequacy of the selected technologies
 - Alkalinisation enables urine stabilisation without chemical inputs
 - ED separates TN (94%) and COD (86%) from salts (72% Na), although ED concentrate contains some COD
 - IX removes selectively sodium (98%) by replacement with potassium
- From fresh urine, ~ 72% sodium removal, considering ED diluate and IX outlet as the final products
- Final products = precipitate rich in divalent cations (Ca²⁺ and Mg²⁺) and phosphate, ED diluate rich in nitrogen and COD, and IX outlet rich in potassium
- Main consumables: water (recover from hydroponics), KCl for resin regeneration
- No toxicity of the diluted/nitrified final products on *Limnospira indica* but negative impact on higher plants
- Proposed scenario: SRU running continuously and in an automated way, with a focus on space constraints



Future work

- Implementation of the proposed scenario: integration of the sub-systems, automation, continuous mode
- Modification of the technologies towards a space design (e.g., gas management) => test unit on ISS to validate higher TRL
- Further knowledge acquisition with regards to COD behavior on the resins
- Improvement of COD oxidation
- Alternative source of potassium for resin regeneration : in-situ resource? (ISRU)
- Optimisation of final products





Thank you