ULTRAPURE MICRO 2018

OVIVO

TEXAS INSTRUMENTS

End-of-Pipe Semiconductor Wastewater Reclaim

OUTLINE

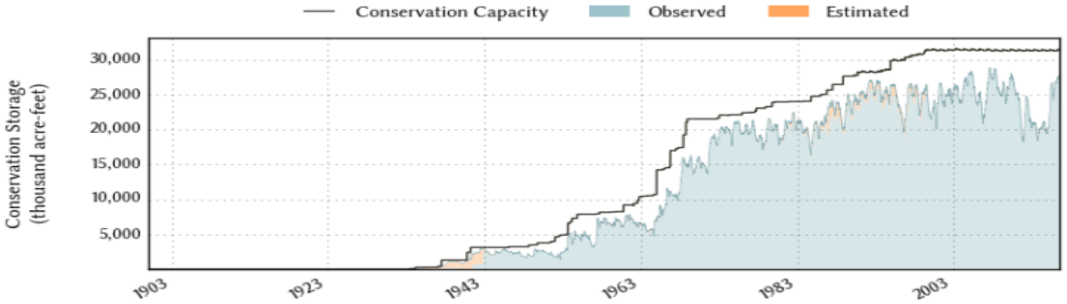
- A CALL TO ACTION
- APPROACH
- RESULTS
  - PILOT CONSTRUCTION
  - TEST PLAN
  - PILOT PERFORMANCE
- CONCLUSIONS/TAKEAWAYS

A CALL TO ACTION

- North Texas Water

**Surface water is the primary source for Texas (80+%)

**Reservoir Capacity in recent drought dropped from 80 o 60% in only 3 years



The Risks

- Regional water constraints are a rising risk to our ability to manufacture wafers (population, infrastructure degradation and capacity, climate change)
- Water costs are continuing to increase (up 8-10% YOY)
- Need to look at the value of water as well as the cost of water
- What do we do if the North Texas situation worsens?

- The Response
  - Develop water balances for all sites
  - Incentivize and prioritize water reduction inside existing cost reduction funding, tracking, best practice structure
  - Further alignment with site water champions as well as project execution and savings validation
  - After 5 years of consumption reduction and reuse enhancement:
    - Water consumption per chip produced reduced by 37%
    - All "low hanging fruit" achieved and easy to recover reuse streams captured
  - New Picture:
    - Makeup (80% for DIW/UPW , 18% for Cooling Towers/Scrubbers , 2% All other uses)
    - Outflows (80% through IWW, 15% through evaporation, 5% through sanitary)
    - **What if we see end-of-pipe IWW as a resource? What if the entirety of it can be a source of new/makeup water? What is the cost? What are the risks?

APPROACH

- Options
  - Wait until situation worsens and technologies are even more mainstream
  - Move product to areas without geographical risk
  - Develop a system with trial and error (bottoms-up)
  - Develop a system using Systems Engineering Process (tops-down)

- Objectives
  1. Understand and quantify the variability in water streams (Can our wastewater be reclaimed?)
  2. Pilot technologies and systems capable of altering water quality to acceptable limits (What would it take?)
  3. Quantify and develop risk mitigation strategies for waste water reuse in semi manufacturing processes (Can we do it reliably?)
  4. Reduce city water consumption and optimize total lifecycle cost for all water use systems (Can we do it cost effectively?)

- Why will this work?
  - This is a formalized process focused on stakeholder requirements definition and life cycle considerations
    - Upfront risk management
    - Requirements definition (traceable and verifiable)
    - Adaptable and Iterative
    - Make use if Integrated Product Teams and lessons learned
    - Find errors before the system is built (optimize cost and schedule)
  - End-of-pipe reclaim for makeup to DIW Plant (using vendors with expertise)

RESULTS

PILOT CONSTRUCTION

PILOT CONSTRUCTION

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PILOT CONSTRUCTION

PILOT SYSTEM DELIVERED TO RFAB

COMPRISES (QTY 2) 40-FT SHIPPING CONTAINERS

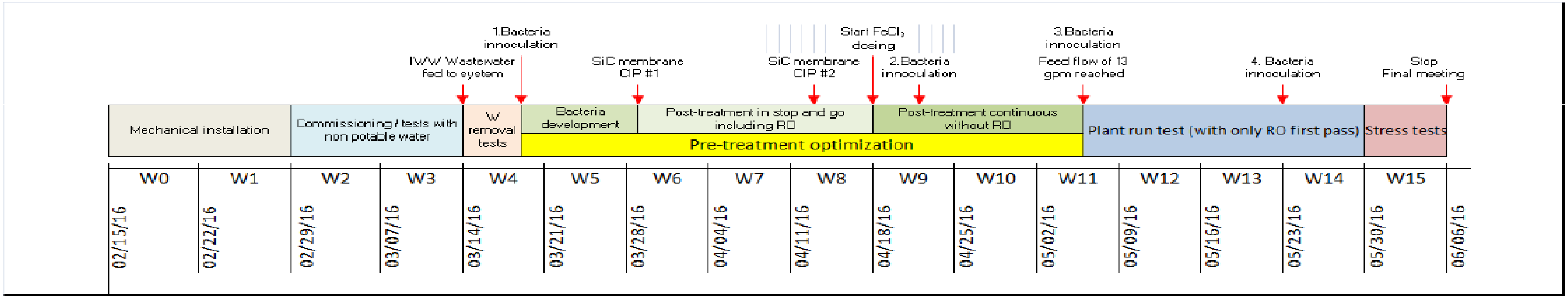
OVIVO ON-SITE LABORATORY

RESULTS

TEST PLAN

RESULTS - TEST PLAN

- **Example Test Items:**
  - Spike each unit processes to test deviations (outside normal variation) – Stress Test
  - Conduct bacteria dip slide samples and microscope inspection for bio population health
  - Perform post-pilot consumable destructive testing?
  - Quantify performance and maintenance required for probes and analyzers
  - Quantify benefit of upfront segregation (ion exchange breakthrough curves)
  - Track man-hours for pilot and lab operation by unit process/equipment
  - Test the pilot configuration for 1st pass RO Reject Recycle (back to front of pilot) during the trial period
  - Conduct a final inspection for all tanks, pumps, piping, etc. prior to full decommissioning. Checking for advanced fouling, consumables breakthrough, etc.



- Pilot Test Duration (90 days) with end-of-pipe wastewater from a semiconductor fab
- The fab effluent contained varying concentrations of chemicals applied during manufacturing process:
    - Hydrogen peroxide (H_2O_2)
    - Tungsten (W)
    - Ammonia (NH_4)
    - Fluorides (F)
    - Organic compounds including:
        - Isopropanol (IPA)
        - Tetramethylammonium hydroxide (TMAH)
        - Acetone
    - High salinity and hardness
    - Fine and very fine particles (e.g. SiO_2)

RESULTS

PILOT PERFORMANCE

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► FAB EFFLUENT QUALITY – 3 MONTHS DATA

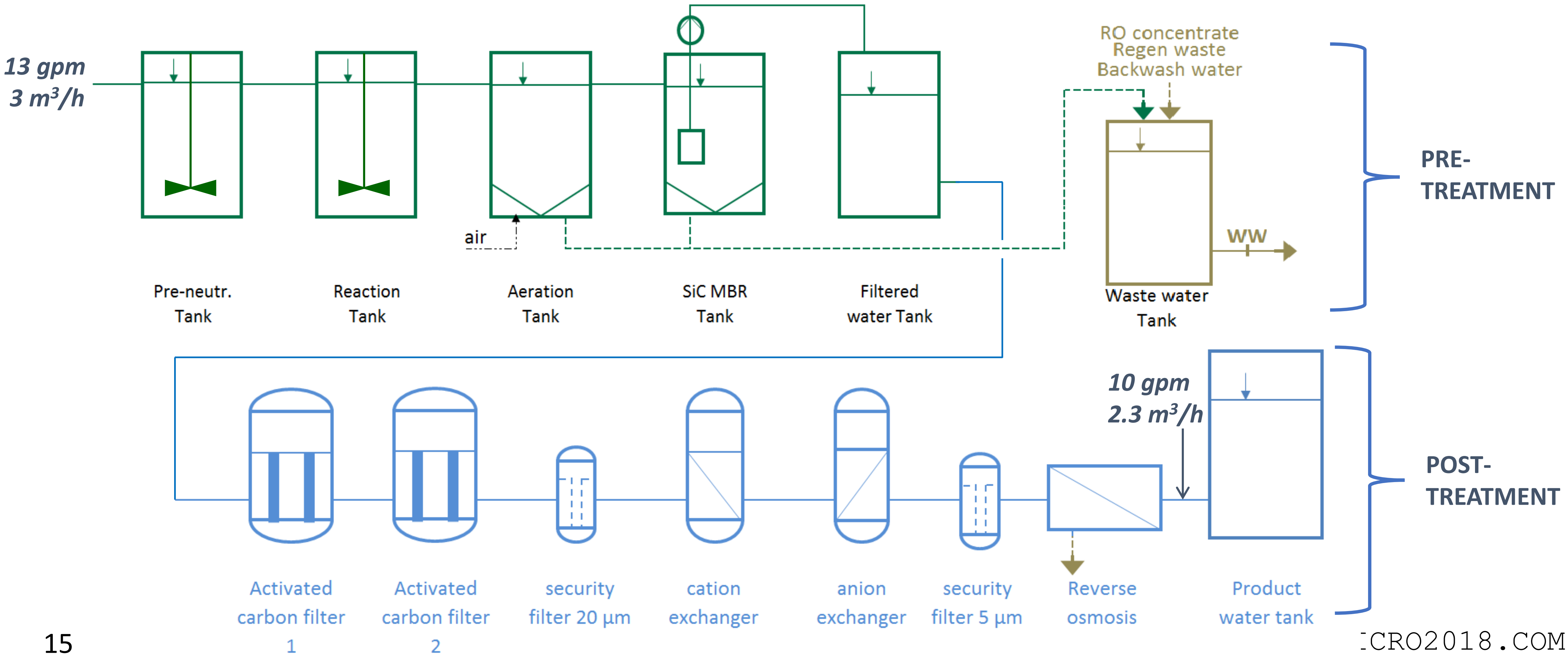
March 4th to June 3rd 2016

Parameter	unit	N° Samples	min	avg	max
Temperature	°F	101	72.3	**79.2**	84.2
Temperature (°C)	°C	95	22.4	**26.2**	29
TSS	ppm	35	0.2	**1.8**	6.4
Turbidity	NFU	77	2.4	**9.6**	17.3
Micro-Bio	CFU/mL	12	0	**7.6**	40
ORP	mV	103	117	**214**	360
H2O2	ppm	76	< 0.5	**110**	400
Conductivity	µS/cm	105	909	**1259**	8000
pH		103	6	**7.9**	8.8
Cations					
TH	ppm CaCO3	102	17.46	**25.5**	43.49
Ca	ppm	102	5.9	**8.7**	15.1
Mg	ppm	102	0.6	**0.9**	1.6
Sr	ppm	102	0.06	**0.79**	1.60
Ba	ppm	102	< 0.01	**< 0.01**	< 0.01
Na	ppm	97	4.6	**64.2**	1400
K	ppm	102	0.8	**1.8**	11.5
NH4-N	ppm	104	61	**107.6**	169
NH4	ppm	105	78.4	**136.3**	217.3
Total cations	meq/l	100	7.6	**10.9**	68.9

Parameter	unit	N° Samples	min	avg	max
TOC	ppm	104	13.2	**20.7**	35.9
IPA	ppm	102	0.10	**5.0**	30.12
Acetone	ppm	99	0.05	**0.17**	2.1
TMAH	ppm	-	5	**10**	15
Oxygen demand					
COD	mg/l	16	31	**60.8**	110
BOD	mg/l	0	0	**0**	0
Anions					
Alkalinity	ppm CaCO3	103	14.55	**47.1**	112.5
F	ppm	103	18	**41.8**	102
Cl	ppm	103	28	**90.4**	360
NO3-N	ppm	103	0.2	**1.2**	19
NO3	ppm	104	0.0	**5.5**	84.1
SO4	ppm	103	11	**205.2**	> 3000
PO4-P	ppm	103	1	**3.8**	19.6
PO4	ppm	104	0.0	**11.5**	60.1
Total anions	meq/l	101	1.0	**10.3**	68.9
Dissolved SiO2	ppm	102	4.8	**20.6**	27.4

Parameter	unit	N° Samples	min	avg	max
Metals					
Hg	ppm	102	< 0.01	**< 0.01**	< 0.01
W	ppm	102	2.1	**4.8**	11
Al	ppm	102	< 0.01	**0.02**	0.2
Cr	ppm	102	< 0.01	**< 0.01**	< 0.01
Cu	ppm	102	< 0.01	**0.01**	0.1
Fe	ppm	102	< 0.01	**0.02**	0.54
Se	ppm	96	< 0.01	**0.01**	0.06
Zn	ppm	102	< 0.01	**0.05**	4.6
Si	ppm	102	2.2	**10.6**	15.7
As	ppm	97	< 0.01	**< 0.01**	< 0.01
B	ppm	101	< 0.01	**0.03**	0.38
Ni	ppm	102	< 0.01	**< 0.01**	< 0.01
Ag	ppm	102	< 0.01	**< 0.01**	< 0.01
Ti	ppm	102	< 0.01	**0.01**	0.02
Mn	ppm	97	< 0.01	**0.0**	0.02
Co	ppm	97	< 0.01	**< 0.01**	< 0.01

RECLAIM SYSTEM OVERVIEW



FAB EFFLUENT (MEASURED VALUES)		
Conductivity	µS/cm	909 - 8000
pH		6 - 8.8
H_2O_2	ppm	0.5 - 400
TOC	ppm	13 - 36

100%

FEED DIVERSION POINT

Two-valves diversion system with following analyzers:

- Conductivity
- pH
- (TOC)
- (H_2O_2)

≈ 92%

RECLAIM PLANT (DESIGN LIMITS)		
Conductivity	µS/cm	< 1500
pH		4 - 10
H_2O_2	ppm	< 600
TOC	ppm	< 40

≈8%

Diverted wastewater stream to the city WW

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PLANT FEED WATER (AVERAGE VALUES)		
Conductivity	µS/cm	1145
pH		7.9
H_2O_2	ppm	106
TOC	ppm	20.5
IPA	ppm	4.8
Acetone	ppm	0.17
TMAH	ppm	10
F	ppm	41.4
NH_4-N	ppm	106.9
W	ppm	4.7
Dissolved Silica	ppm	20.3
TSS	ppm	1.8
Turbidity	NFU	9.7

WCF > 97%

PRE-TREATMENT

Nutralization/Reaction tanks: pH adjustment and flocculant dosing ($FeCl_3$)

Aeration tank: filled with bio-media providing maximum surface area for bacteria to colonize

MBR filtration tank: SiC (0.1µm) membranes, physical barrier to suspended solids (solids in the fab WW, bacteria, flocs)

UF concentrate discharged to city WW (or filterpress)

WCF > 85%

POST-TREATMENT

Activated carbon filter: Populated with specfic bacteria for further TOC reduction

Cation/anion exchanger: SAC, WBA/SBA: removal of salinity and other; "pH bacteria barrier".

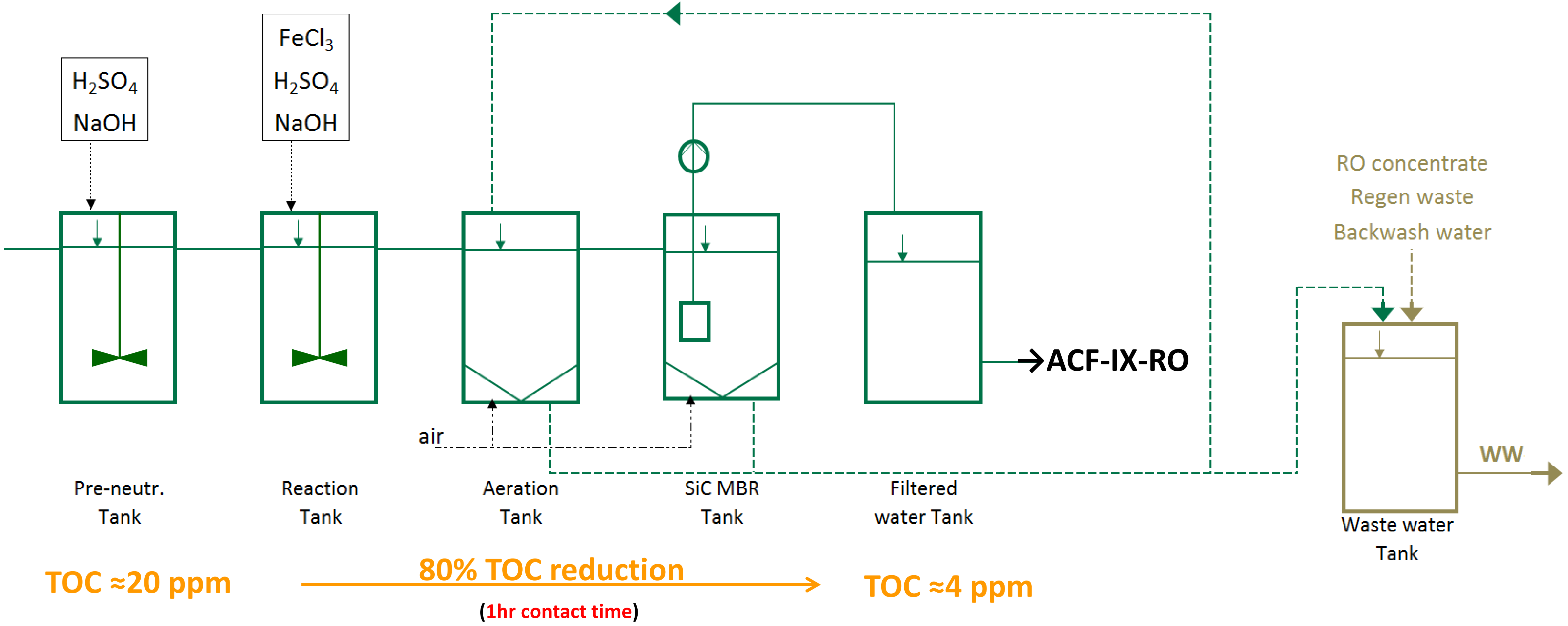
Reverse osmosis: Final reduction of TOC, salinity, other. Safety role in case of lower upstream steps performance.

ACF BW waste, IX regen. waste, RO reject: discharged to city WW or recirculated

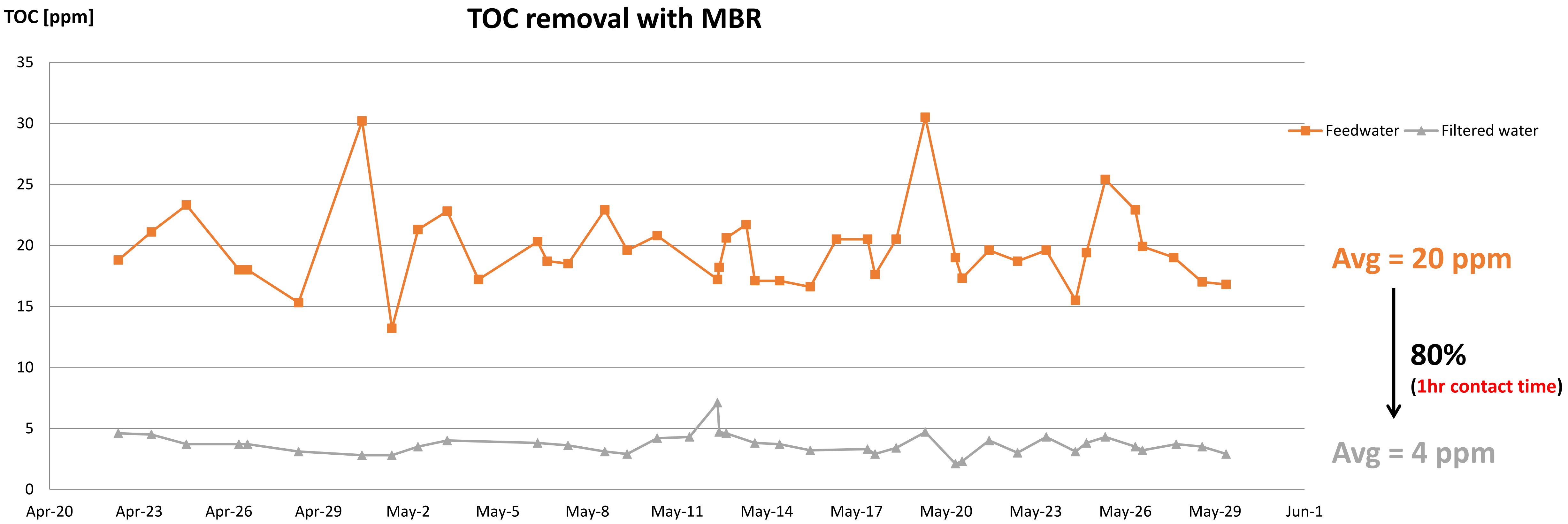
OVERALL WCF > 80%

RECLAIM SYSTEM PRODUCT (GUARANTEED VALUES)		
Conductivity	µS/cm	<10
pH		≈7
H_2O_2	ppm	< 1
TOC	ppm	<0.5
IPA	ppm	<0.05
Acetone	ppm	<0.05
TMAH	ppm	<0.1
F	ppm	<0.1
NH_4-N	ppm	<0.1
W	ppm	< 0.05
Dissolved Silica	ppm	< 0.5
TSS	ppm	< 1
Turbidity	NFU	-

PRE-TREATMENT



PRE-TREATMENT



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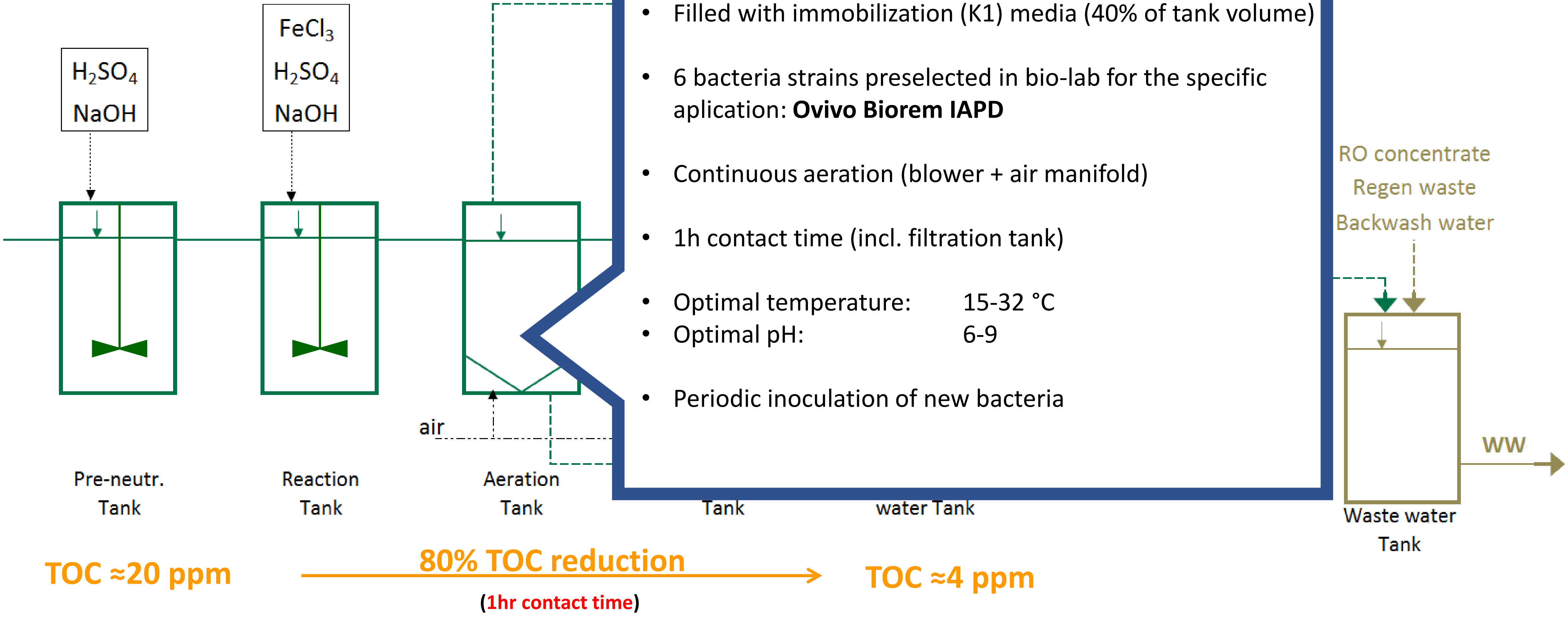
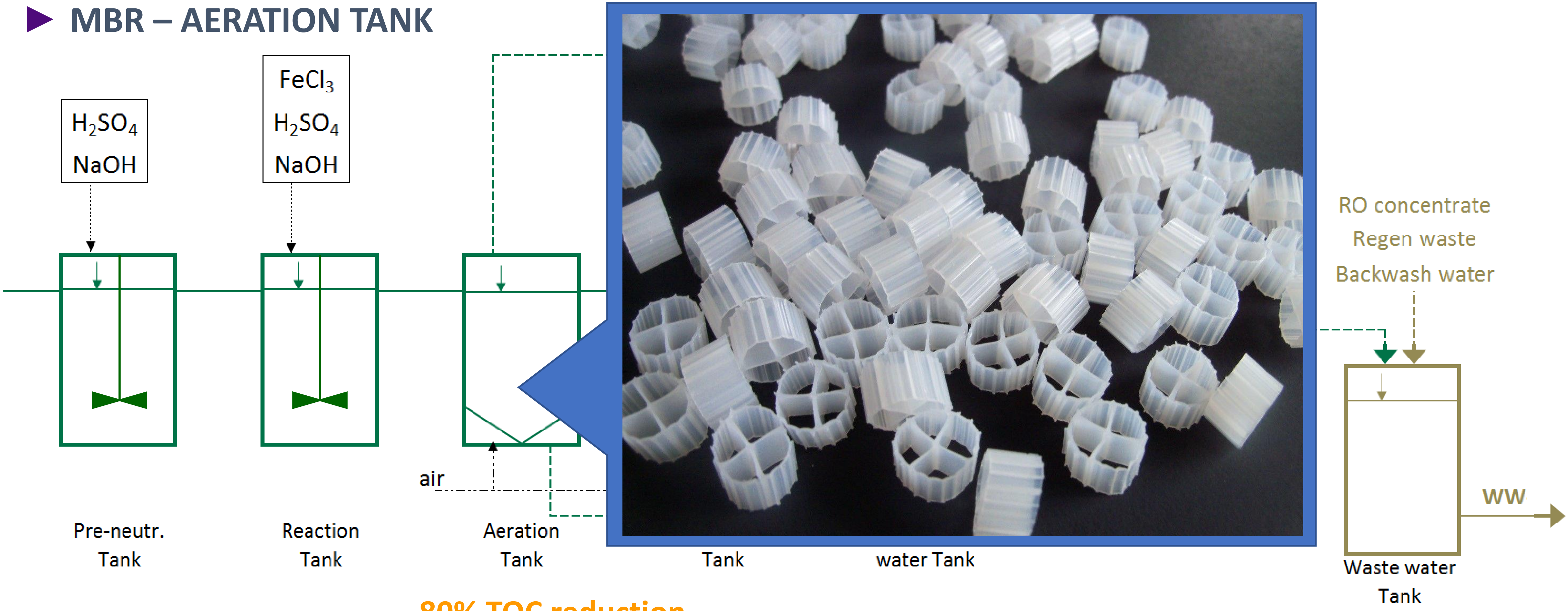
MBR – AERATION TANK



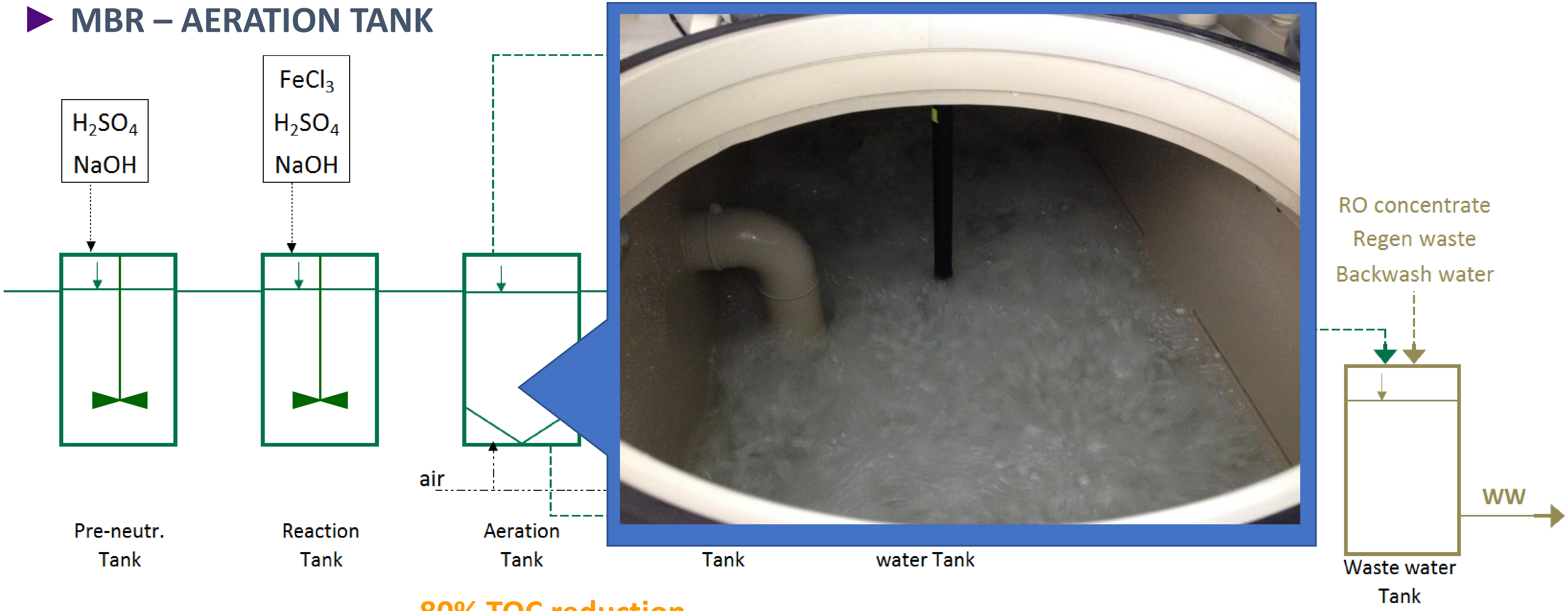
- Filled with immobilization (K1) media (40% of tank volume)
- 6 bacteria strains preselected in bio-lab for the specific aplication: **Ovivo Biorem IAPD**
- Continuous aeration (blower + air manifold)
- 1h contact time (incl. filtration tank)
- Optimal temperature: 15-32 °C
- Optimal pH: 6-9
- Periodic inoculation of new bacteria

MBR – AERATION TANK



TOC ≈20 ppm → **80% TOC reduction** (1hr contact time) → TOC ≈4 ppm

MBR – AERATION TANK



TOC ≈20 ppm → **80% TOC reduction** (**1hr contact time**) → **TOC ≈4 ppm**

► MBR – OVIVO BIOREM IAPD

- Specific mixture of six bacteria strains preselected in bio-lab in Switzerland

- The bacteria mixtured is tailored to the specific wastewater to:
    - maximize digestion of unwanted organics present in the wastewater
    - minimize digestion time (later corresponding to the contact time / MBR tanks sizes)
    - minimize bacteria mortality

- The bio-lab with a long time experience in bacteria selection has a library of bacteria strains with pre-tested performance

- The final choice of bacteria includes a test with sample of actual wastewater to be treated

▶ MBR – OVIVO BIOREM IAPD PERFORMACE: TOC SPECIATION BY LC-OCD

				NOM							SOM														
	DOC Dissolved	HOC* Hydrophob.	CDOC Hydrophil.	BIO-polymers	DON (Norg)	% Proteins in BIOpol.**	Building Blocks	LMW Acids	LMW Neutrals	Urea	X1 110 min	X2 88,9 min	X3 94 min	X4 103,4 min	X5 114,7 min	X6 117,3 min	X7 121,7 min	X8 129,3 min	X9 590 min	X10 161,4 min	X11 190,5 min	DN	CDON	NO3-	NH_4^+
Project: ovivo_43	ppb-C	ppb-C	ppb-C	ppb-C	ppb-N	% BIOpol.	ppb-C	ppb-C	ppb-C	ppb-C	ppb-C	ppb-C	ppb-C	ppb-C	ppb-C	ppb-C	ppb-C	ppb-C	ppb-C	ppb-C	ppb-C	ppb-N	ppb-N	ppb-N	ppb-N
Feed water	% DOC	% DOC	% DOC	% DOC	--	--	% DOC	% DOC	% DOC	% DOC	% DOC	% DOC	% DOC	% DOC	% DOC	% DOC	% DOC	% DOC	% DOC	% DOC	% DOC	--	--	--	--
	25572	< 5	25572	11	< 5		246	353	1493	< 5	3314	10	< 5	3028	< 5	3858	< 5	< 5	481	12776	< 5	>> 58901	2437	692	>> 11289
MBR product	100%	--	100,0%	0,0%	--	--	1,0%	1,4%	5,8%	–	13,0%	0,0%	--	11,8%	--	15,1%	--	--	1,9%	50,0%	–	--	--	--	--
	3662	< 2	3662	197	< 2		317	301	471	11	1896	< 2	13	< 2	< 2	< 2	89	< 2	351	17	< 2	>> 19763	127	655	>> 4619
Bio-ACF product	100%	--	100,0%	5,4%	--	--	8,6%	8,2%	12,9%	0,3%	51,8%	--	0,4%	--	--	--	2,4%	--	9,6%	0,5%	–	--	--	--	--
	418	< 1	418	199	< 1		12	24	119	15	13	7	1	< 1	< 1	< 1	13	6	< 1	< 1	8	>> 7796	35	392	>> 1627
	100%	--	100,0%	47,6%	--	--	2,8%	5,7%	28,3%	3,6%	3,2%	1,8%	0,4%	--	--	--	3,1%	1,4%	--	--	2,0%	--	--	--	--
Sample D	585	107	479	102	< 1		15	35	190	23	34	13	< 1	31	7	< 1	< 1	14	< 1	5	11	325	130	167	29
	100%	18,2%	81,8%	17,4%	--	--	2,6%	6,0%	32,4%	3,9%	5,8%	2,2%	--	5,3%	1,2%	--	--	2,3%	--	0,9%	1,9%	--	--	--	--
Sample E	203	55	149	4	< 1		< 1	7	39	20	9	1	< 1	38	4	< 1	1	10	< 1	7	8	55	42	< 1	13
	100%	27,0%	73,0%	2,0%	--	--	--	3,3%	19,2%	10,0%	4,3%	0,5%	--	18,9%	2,1%	--	0,6%	4,7%	--	3,3%	4,1%	--	--	--	--
RO product	156	60	95	3	< 1		< 1	10	30	16	10	2	< 1	< 1	6	< 1	4	1	< 1	4	11	51	34	< 1	17
	100%	38,7%	61,3%	1,8%	--	--	--	6,5%	19,6%	10,0%	6,2%	1,1%	--	--	3,5%	--	2,4%	0,9%	--	2,3%	7,1%	--	--	--	--

IPA

TMAH

TOC reduction rates:

- MBR: 85%
- Bio-ACF 98.4%
- whole reclaim plant incl. RO 99.4%

Library matches:

X1:	MeOH / Acetamide / EtOH
X2:	Glucose / Fructose
X3:	Glycerine / DMSO
X4:	1,2 Propandiol / 1,3 Propandiol
X5:	Acetone
X6:	IPA
X7:	y-Butyrolactone / t-BuOH
X8:	MEK
X9:	Benzotriazole
X10:	TMA / TMAH
X11:	Pyridine

Evaluation of H_2O_2 toxicity

Strain (N°)	0 ppm	500 ppm	750 ppm	1'000 ppm	2'000 ppm	10'000 ppm
1	0-10 % killed	10-20 % killed	20-50% killed	> 90-99 % killed	> 90-99 % killed	> 90-99 % killed
2	0-10 % killed	20-50% killed	50-90 %	> 90-99 % killed	> 90-99 % killed	> 90-99 % killed
3	0-10 % killed	0-10 % killed	0-10 % killed	0-10 % killed	0-10 % killed	10-20 % killed
4	0-10 % killed	0-10 % killed	0-10 % killed	0-10 % killed	20-50% killed	> 90-99 % killed
5	0-10 % killed	20-50% killed	50-90 %	> 90-99 % killed	> 90-99 % killed	> 90-99 % killed
6	0-10 % killed	0-10 % killed	0-10 % killed	0-10 % killed	0-10 % killed	> 90-99 % killed

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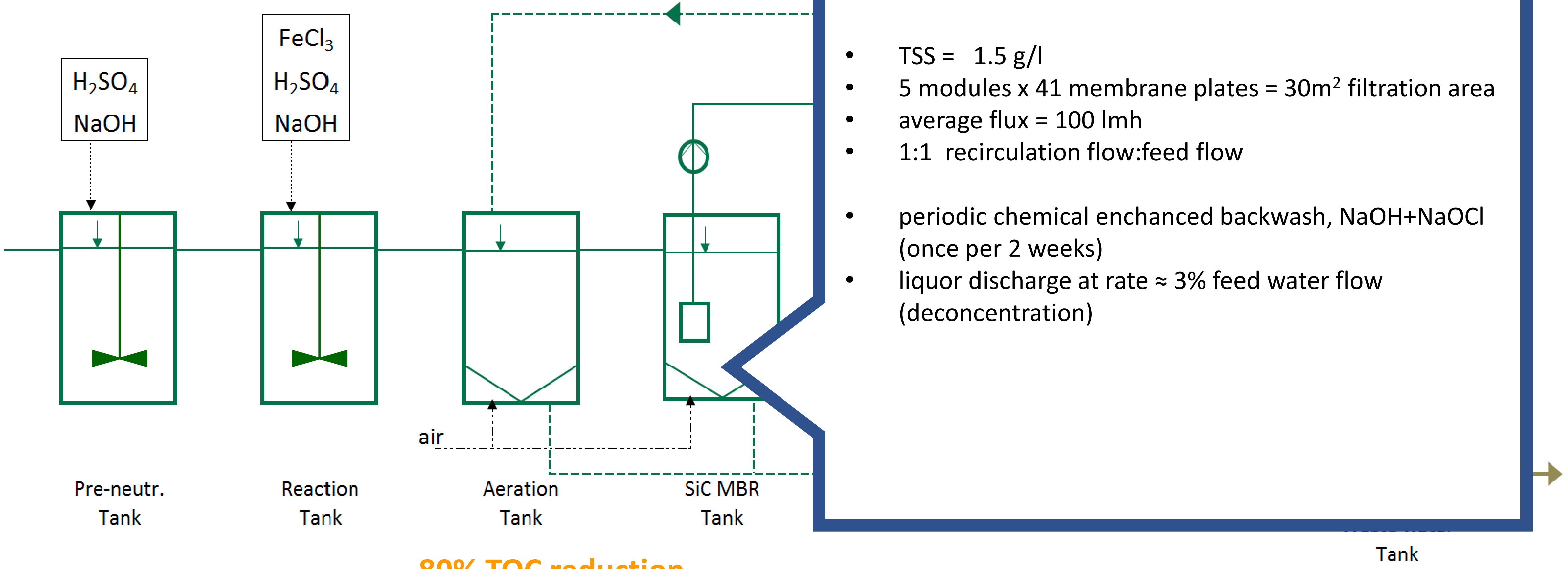
▶ SiC FILTRATION TANK

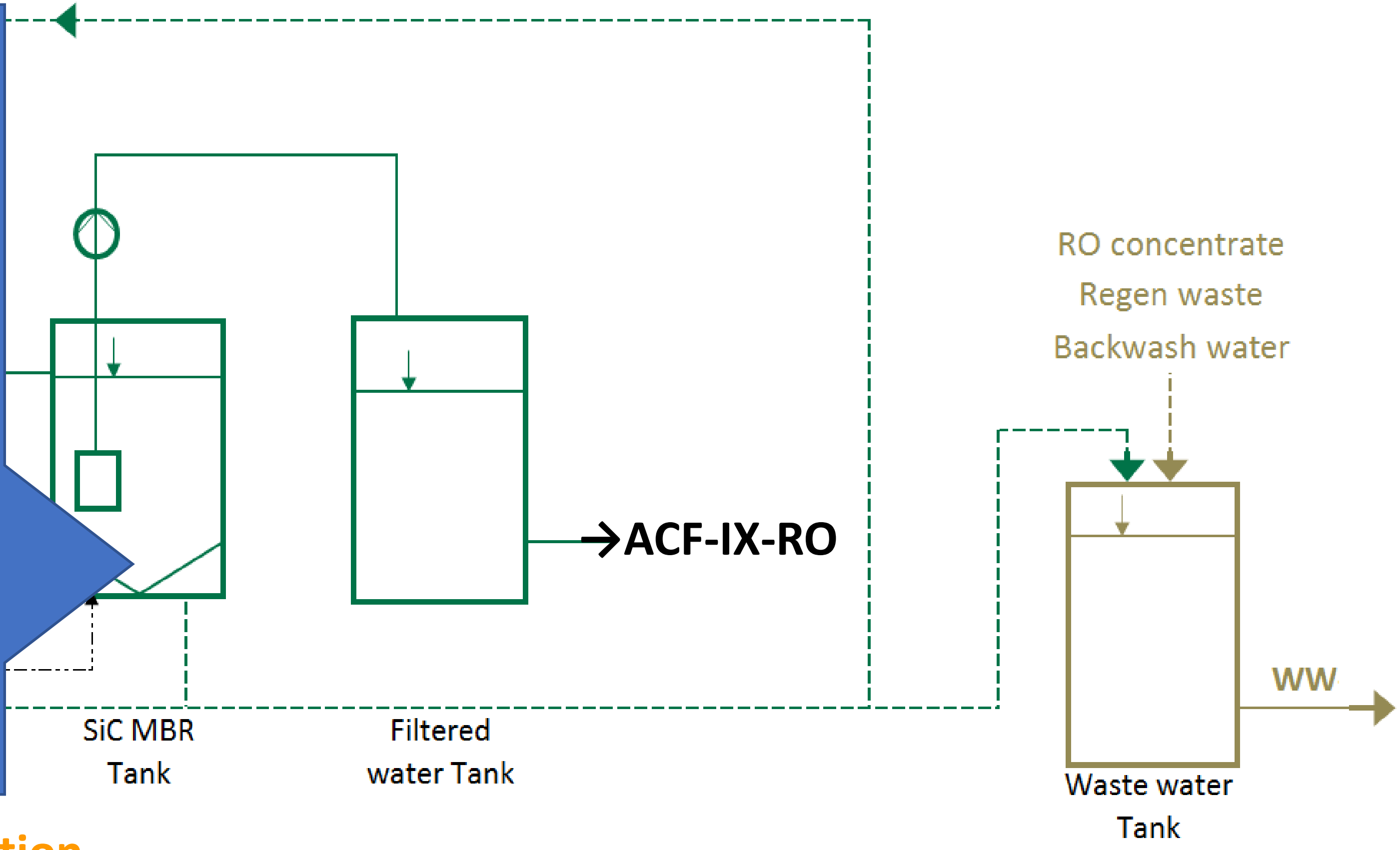


- TSS = 1.5 g/l
- 5 modules x 41 membrane plates = 30m^2 filtration area
- average flux = 100 lmh
- 1:1 recirculation flow:feed flow

- periodic chemical enchanced backwash, NaOH+NaOCl (once per 2 weeks)
- liquor discharge at rate ≈ 3% feed water flow (deconcentration)

TOC ≈20 ppm → **80% TOC reduction** (**1hr contact time**) → **TOC ≈4 ppm**

SiC FILTRATION TANK



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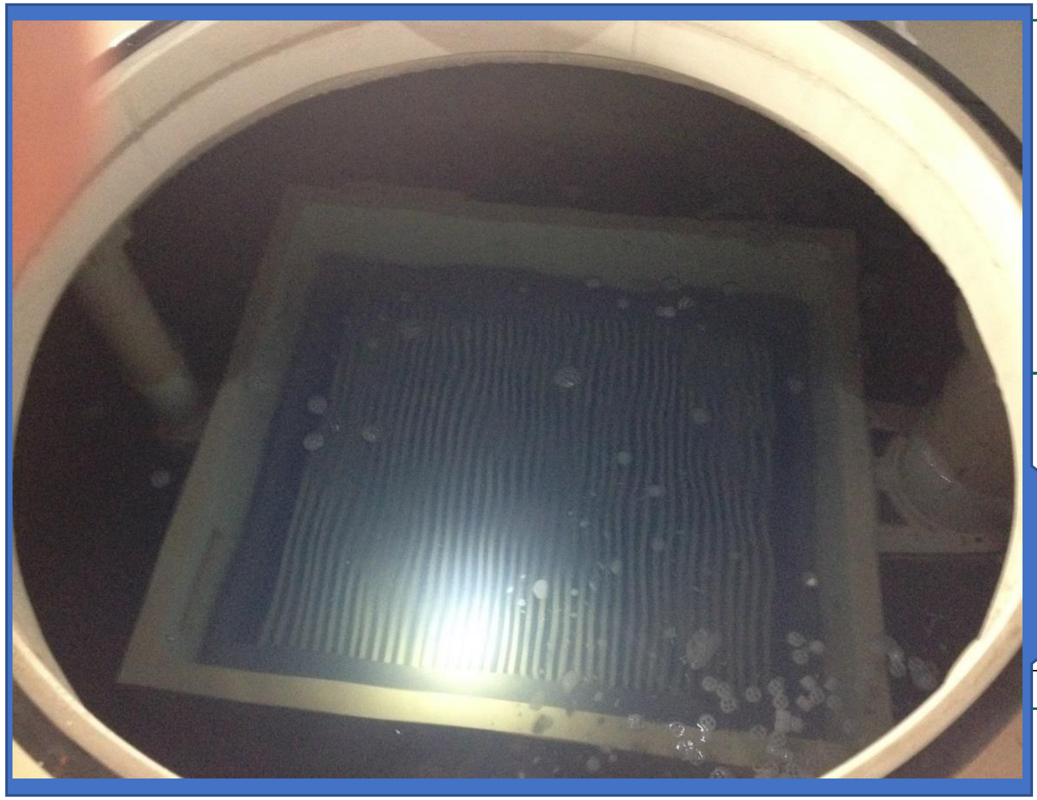
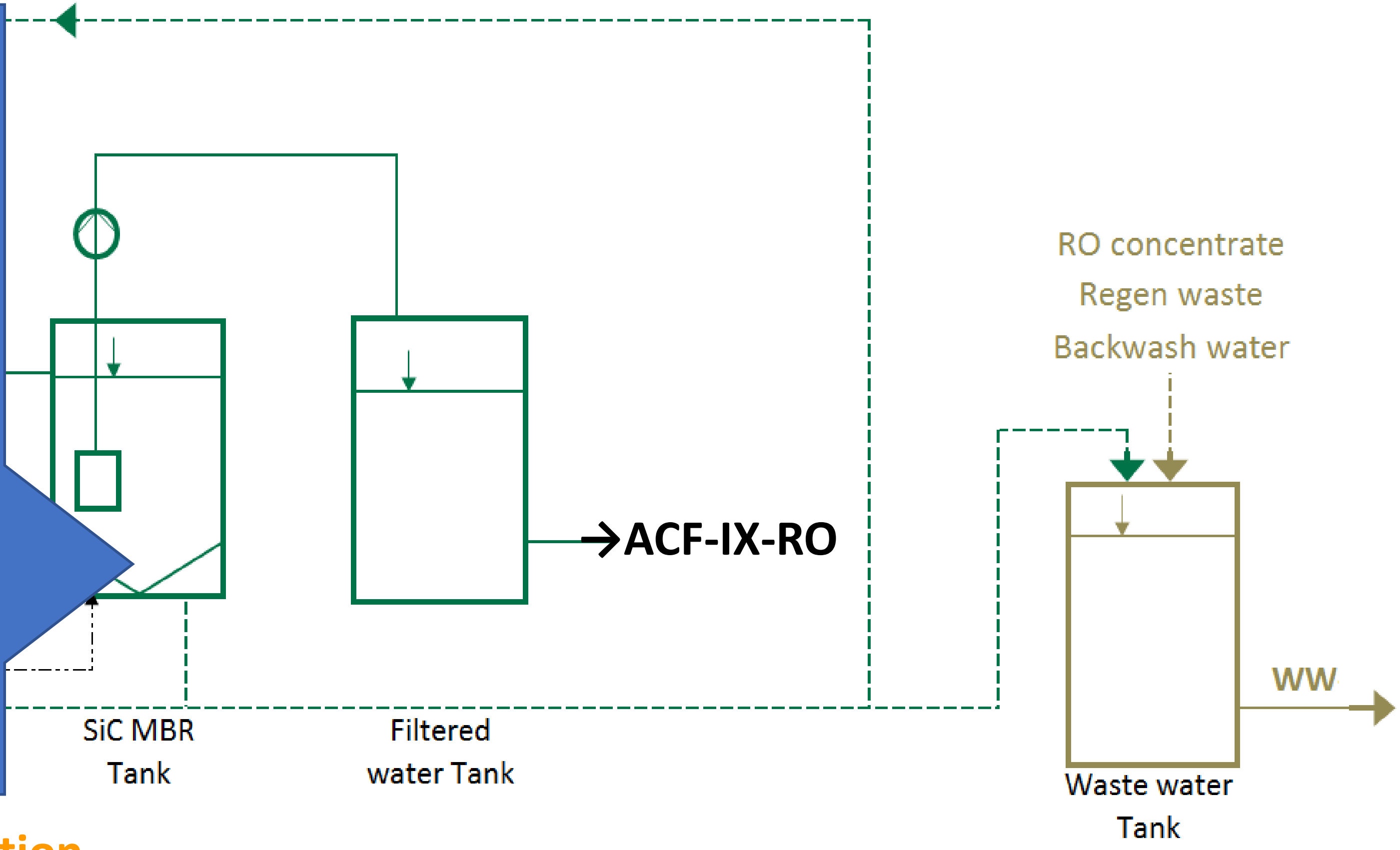
SiC FILTRATION TANK





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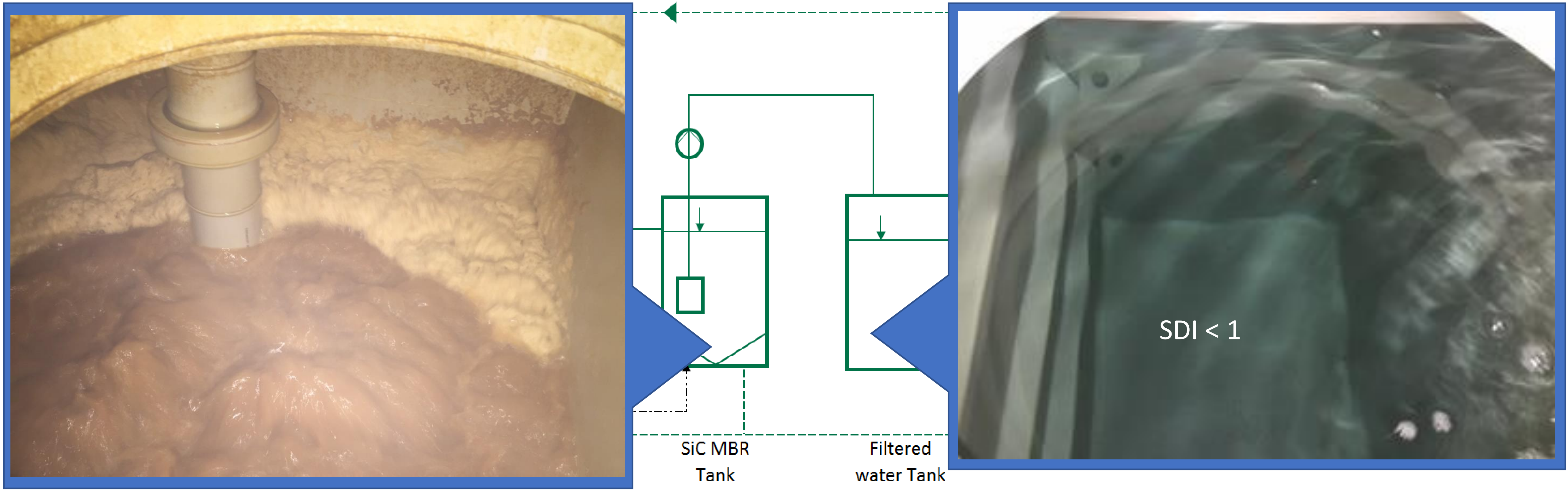
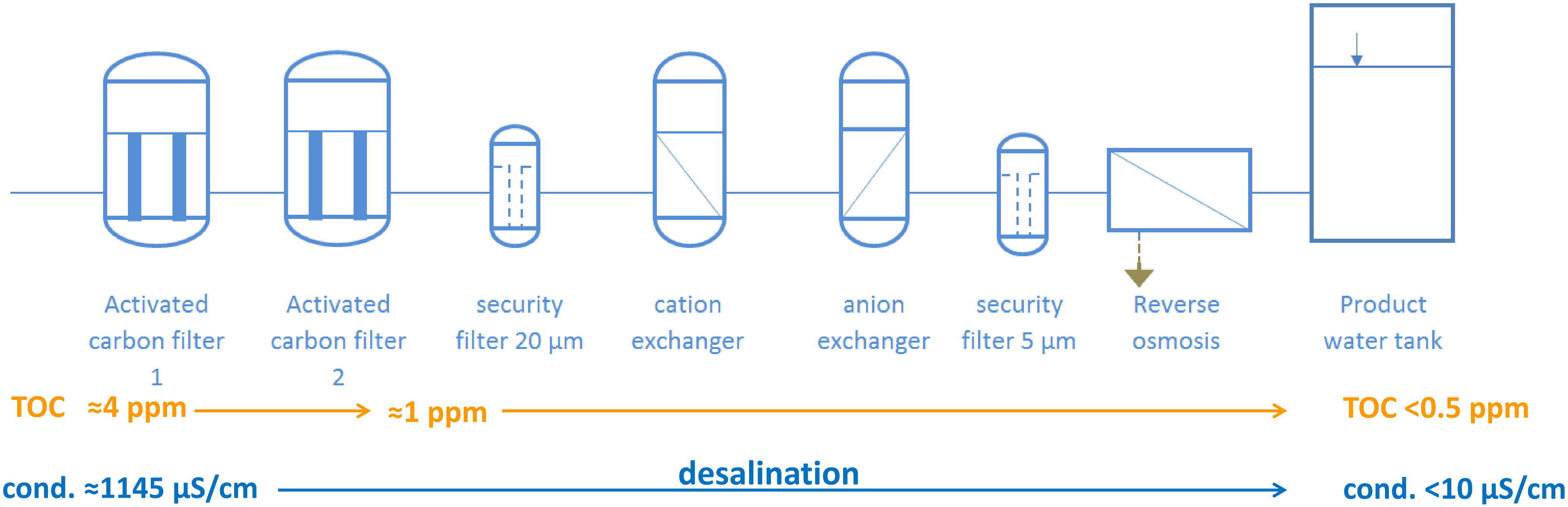
▶ SiC FILTRATION TANK AND FILTERED WATER TANK

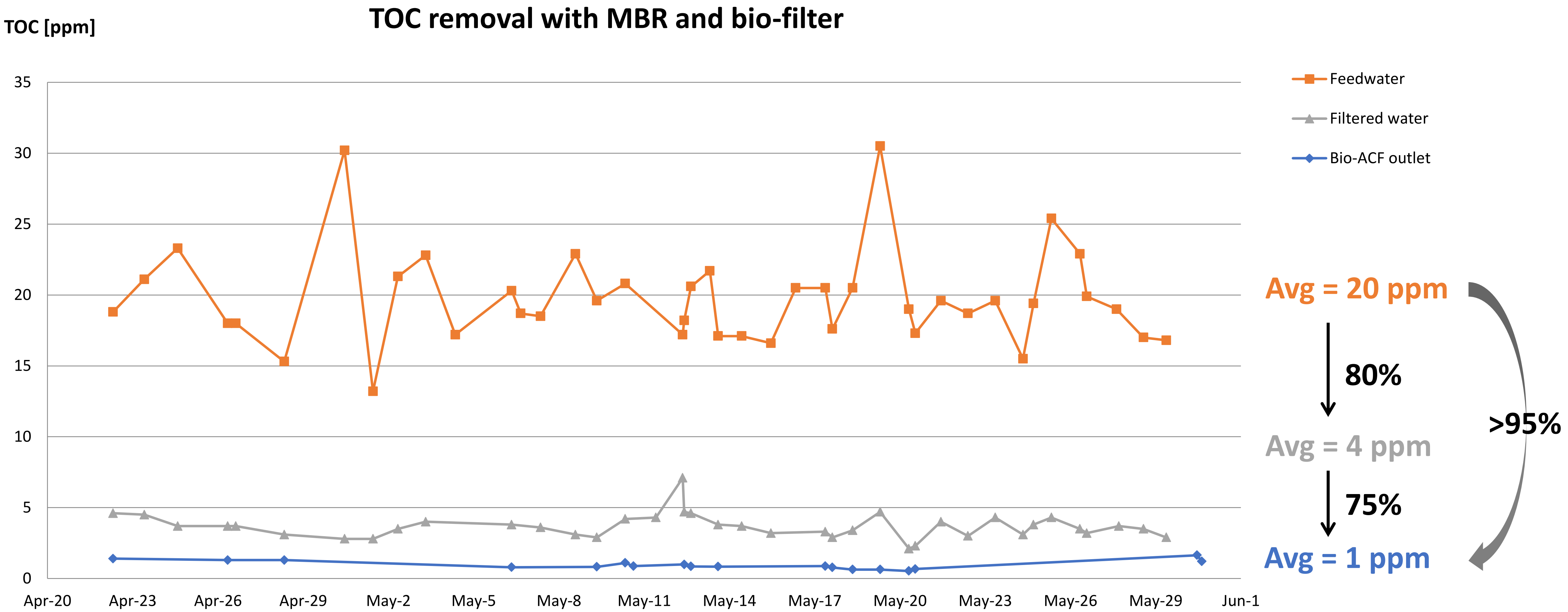


TOC ≈20 ppm → **80% TOC reduction** (**1hr contact time**) → TOC ≈4 ppm

POST-TREATMENT



▶ POST-TREATMENT – BIO-ACF

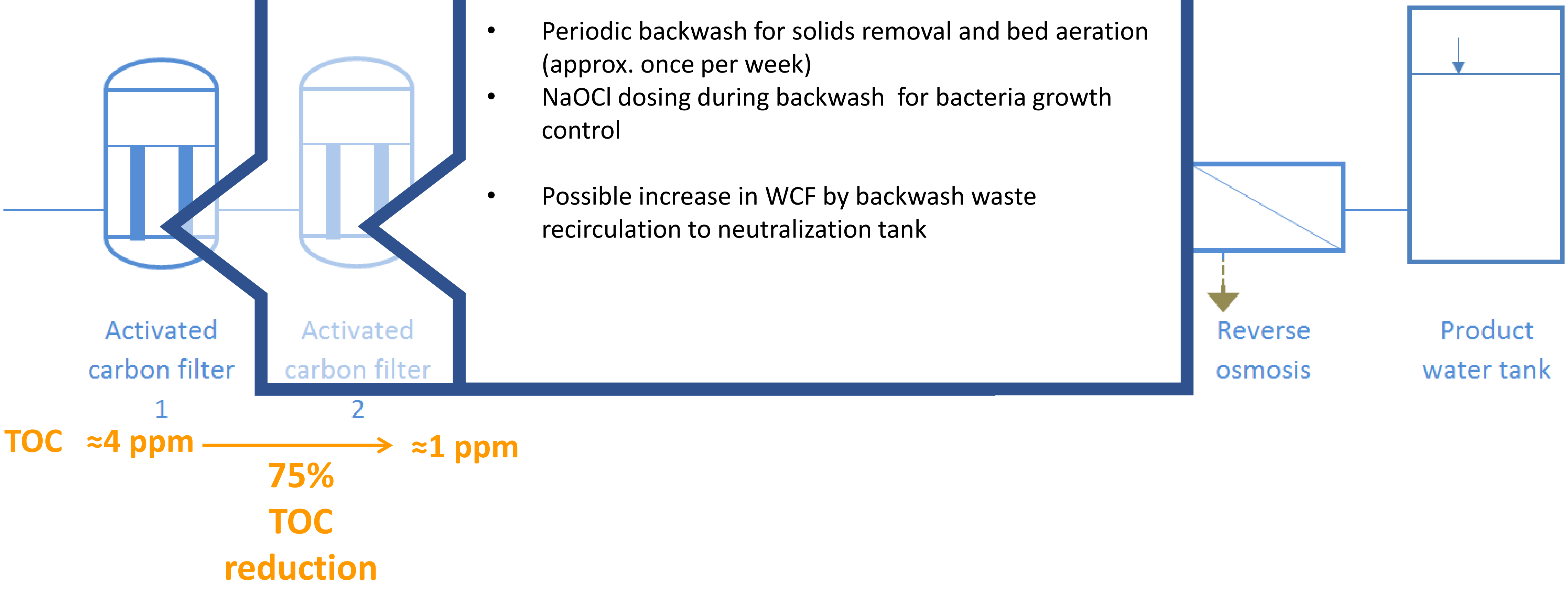


▶ POST-TREATMENT

- Bio-activated carbon filters
- Populated with the same bacteria strains as in the MBR
- 15 min contact time

- Periodic backwash for solids removal and bed aeration (approx. once per week)
- NaOCl dosing during backwash for bacteria growth control

- Possible increase in WCF by backwash waste recirculation to neutralization tank



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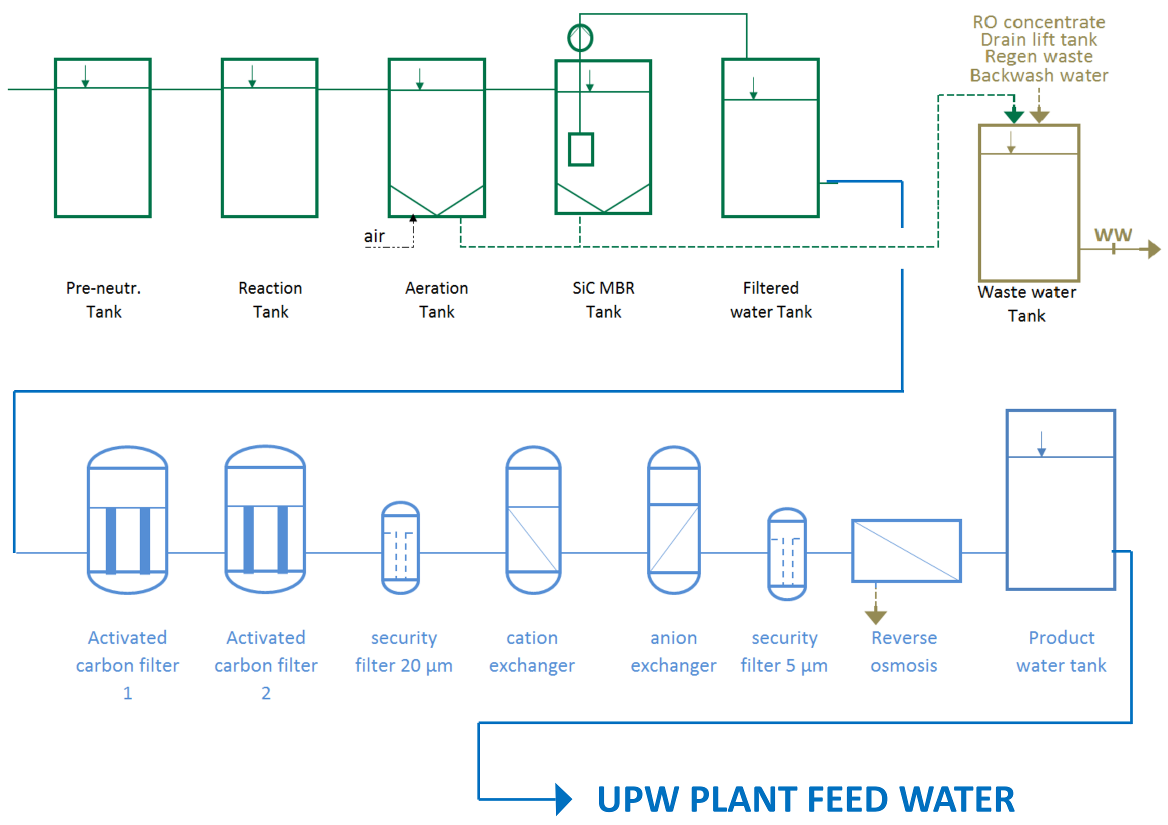
SUMMARY



MBR:

- Removal of critical organics (low molecular weight polar molecules, e.g. IPA, acetone) and TMAH
- TOC reduction at MBR > 80%
- Pre-treatment WCF > 97%
- Reduced footprint thanks to short contact times achieved and high SiC membranes flux (less membrane area required)

OVERALL PLANT:

- Fab waste treated to quality superior to city water
- Overall plant TOC reduction > 97%
- Overall plant WCF > 80%
- Cost effective and ecologically friendly solution

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OPERATING COST

OPERATING COST [USD / volume product]		
Electric energy		11.1 %
Regeneration agents / chemical dosing		
H2SO4 90%		24.9 %
NaOH 50%		29.9 %
FeCl3 40% (feed)		8.0 %
NaOCl 14% (MBR + FWT + ACF)		0.1 %
Ovivo Biorem IAPD		9.6 %
Consumables [Lifetime in years]		
IX resins	**[6]**	3.9 %
Activated carbon	**[3]**	2.4 %
UF Ovivo SiC membranes	**[6]**	8.3 %
RO-Modules (+ sec. filters)	**[6]**	1.7 %
TOTAL		100 %

**The IWW Reuse system operating cost being based mostly on chemicals is much more variable in relation to fab loadings than city water cost is

- DIW Plants
  - Increased RO efficiency and membrane life
  - Increased time between mixed bed regenerations and resin life
  - Improved carbon life and potential to extend backwashes
  - Improved delivered water quality and plant stability
- Scrubbers
  - Eliminates scale-forming potential
  - Increased consumables life (packing and mist eliminators)
- Cooling Towers
  - Reduces scale forming potential and allows towers to run at higher cycles consuming less water
  - Prolongs the life of cooling tower structure and fill

**The IWW Reuse System itself can also be optimized and lessons learned from future Pilots fed back to the final design and operation

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PRIMARY CONCLUSIONS/TAKEAWAYS

Lessons Learned

- By having MBR & IEX before RO's, the RO's see no biofouling throughout full lifetime of operation
- Testing at sufficient equipment scale has direct application to predicting expected maintenance performance
- End of pilot stress testing can better define diversion criteria and quantify process sensitivity to interruption or change in wastewater quality
- Design pilot not to match exact the expectation of a full-scale solution, design for flexibility, potential, and testability (Double Pass RO that can also be operated as a Single Pass)

What is novel about this pilot program?

- Validated with real-time cost tracking that reclaimed water could be produced cheaper than city water (payback validation to aid in funding a long-term project)
- SiC UF & MBR for robust operation and sustained higher flux
- Test protocol and onsite lab (combo of online analyzer and manual samples)
- Specifically developed bio strains started 6 months before the pilot arrived (no pilot operational delay for bio strain development)
- System Engineering Approach (functional, tops-down requirements development)

Conclusions

- End-of-pipe reclaim is feasible and offers the greatest opportunity for water recovery
- Proved wastewater (as is) can be turned into city water or better with a water conversion efficiency between 70-85%
- Validated a robust measurement and diversion scheme and stress tested the system to ensure reliability and resilience
- Proved reuse water could be produced at 30-45% the cost of city water at today's rates

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