

From Lab to Pilot Scale

The Path to Commercialization of the membrane processes

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Me-Sep Team

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Our mission

- ⊗ Support of the membrane business with:
 - ☒ Consultancy
 - ☒ Development projects
 - ☒ HF membrane potting
 - ☒ Customized equipment for R&D and membrane manufacturing
 - ☒ Contract manufacturing
 - ☒ Distribution: Porometer, Philos, Convergence

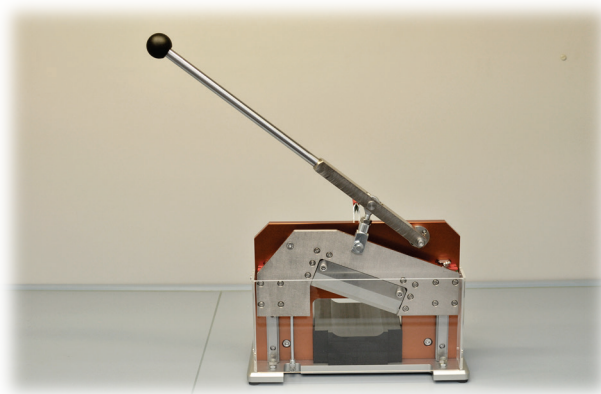


Custom-made equipment

- ⊗ Potting centrifuges
- ⊗ Stationary potting systems
- ⊗ Glue mixing and dispensing systems
- ⊗ Module cutting systems
- ⊗ Integrity testers



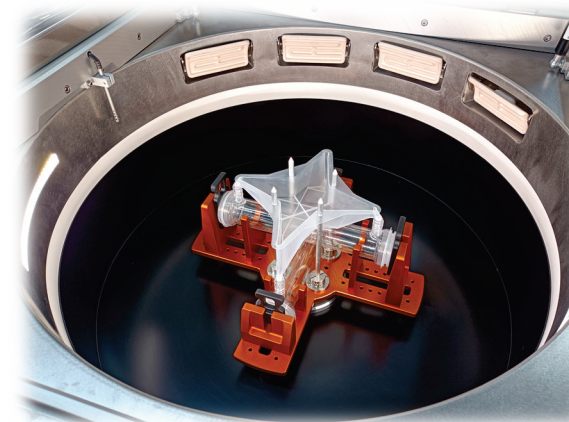
Potting centrifuge for 500 x 3' modules



Guillotine for PUR modules

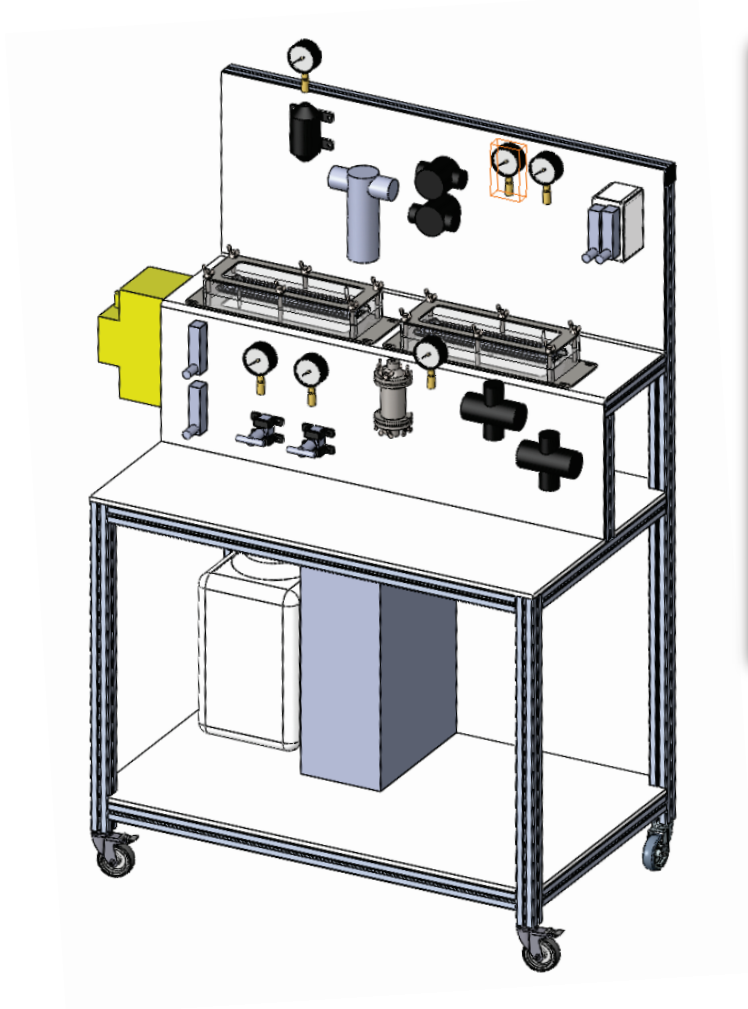


2K Glue dispenser head



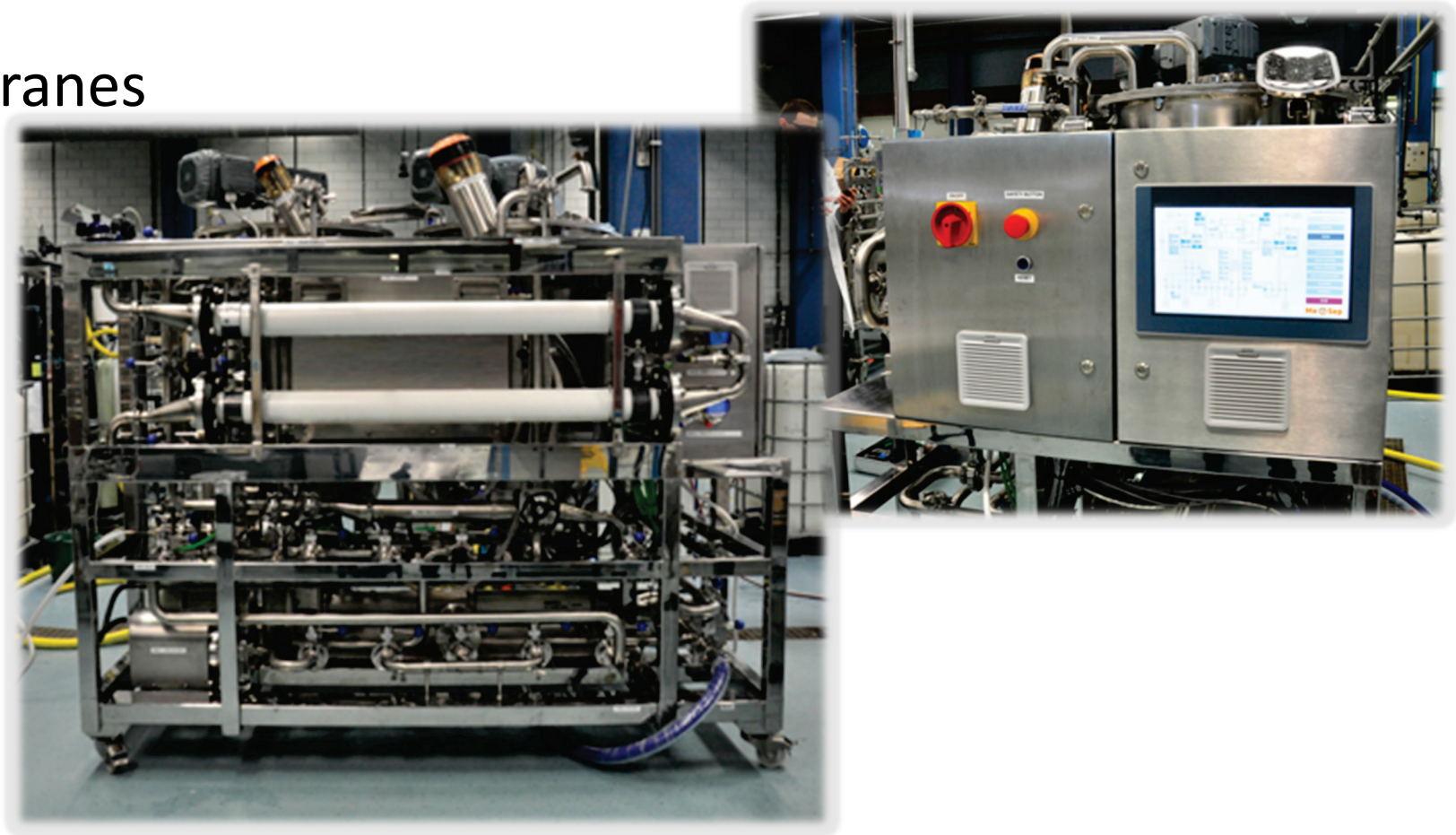
R&D modules in centrifuge

Lab Scale Filtration Set-ups

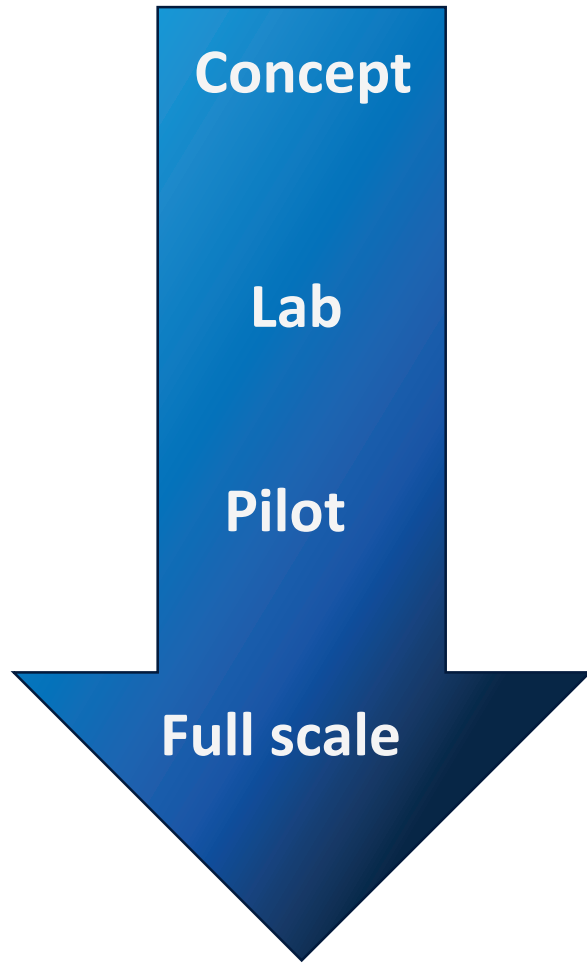


Pilot Scale Filtration Units

- ⊗ Custom-made pilot scale filtration units
- ⊗ Selection of the membranes
- ⊗ Membrane tests
- ⊗ Process optimization



Introduction



Me-Sep tasks in MEDZERO:

WP5 T3:

- ⊗ Technology development for water waste treatment

WP5 T5:

- ⊗ Technology selection and definition of measurable parameters

WP6 T4:

- ⊗ Concept of pilot installation

WP6 T5:

- ⊗ Concept of full scale installation

Waste water quality

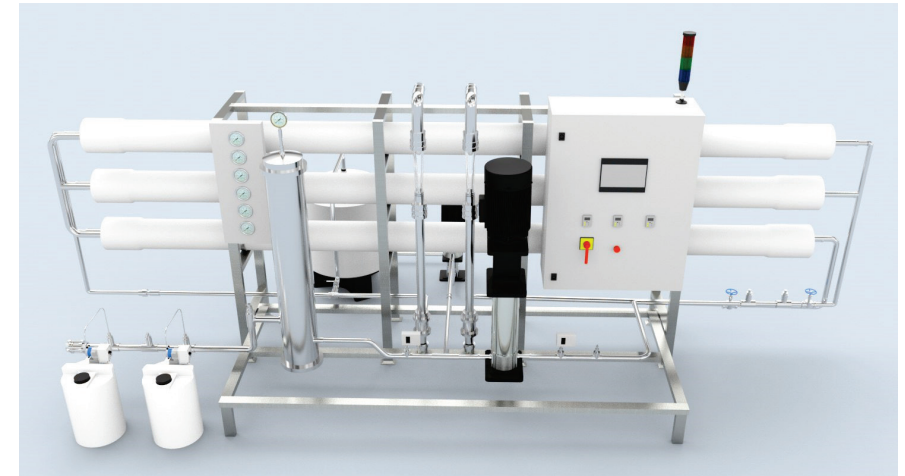
Representative example of wastewater quality after biological treatment and filtration with 0.45 um filter

Parameter	Conc.
TOC [mg/L]	16,7
DOC [mg/L]	7,5
IC [mg/L]	28,2
TN [mg/L]	151
ON [mg/L]	5,6
NH ₄ -N [mg/L]	3
NO _x -N [mg/L]	142,4
NO ₃ -N [mg/L]	140
NO ₂ -N [mg/L]	2,4
oPO ₄ -P [mg/L]	6,8

- ⊗ Nitrogen in form of NO₃⁻ is the main concern
- ⊗ Organic carbon, inorganic carbon and phosphorus should also be removed

Suitable membrane technology

- ⊛ Membrane technologies such as RO or NF should manage to remove inorganic and organic impurities
 - ⊛ Brackish water RO membranes
 - ⊛ Monovalent ion removal: above 99%
 - ⊛ Permeability: 2-4 LMHBar
 - ⊛ Operating pressure: up to 15.5 bar
 - ⊛ Recovery: 15 to 50%
 - ⊛ TFC
 - ⊛ Spiral wound modules
 - ⊛ Tap water RO membranes
 - ⊛ Monovalent ion removal: above 95%
 - ⊛ Permeability: 8-12 LMHBar
 - ⊛ Operating pressure: up to 8 bar
 - ⊛ Recovery: 15 to 50%
 - ⊛ TFC
 - ⊛ Spiral wound modules
 - ⊛ NF membranes
 - ⊛ Divalent ion removal: above 98%
 - ⊛ Monovalent ion removal: 30-80%
 - ⊛ Permeability: 4-10 LMHBar
 - ⊛ Operating pressure: up to 10 bar
 - ⊛ Recovery: 15 to 50%
 - ⊛ TFC or LBL
 - ⊛ Spiral wound modules or hollow fibers



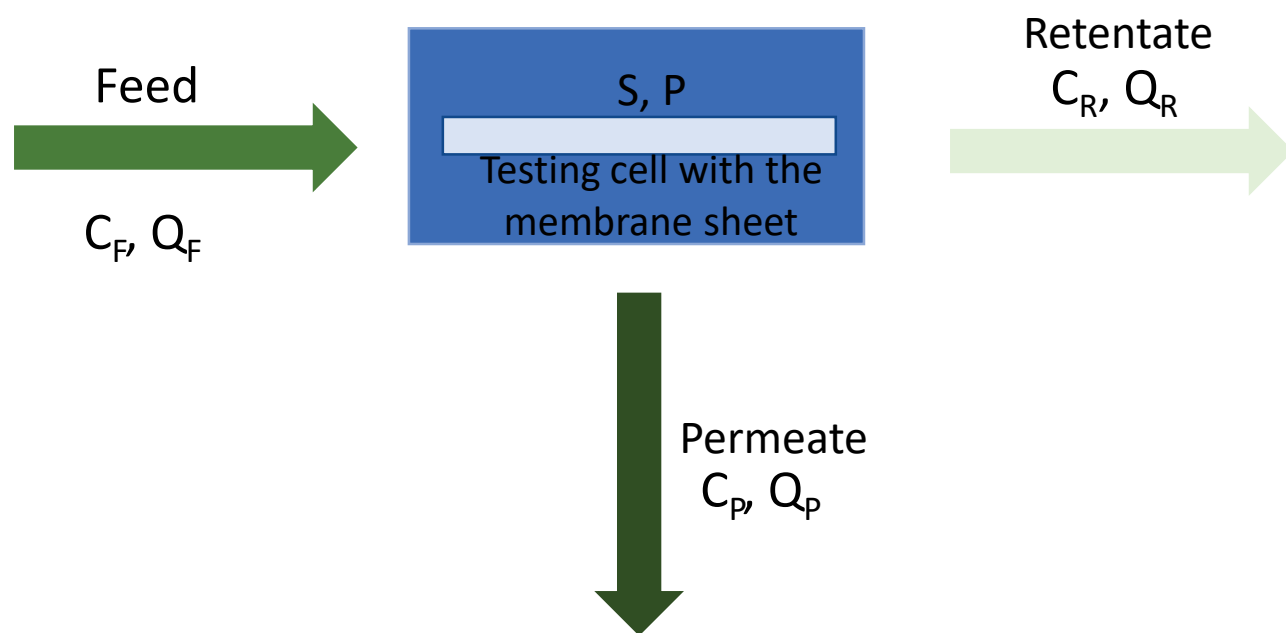
Selection of the membranes for the test

Membrane module selection for the tests:

Supplier	Model	Permeate flow rate [LPH]	Permeability [LMHBar]	Length [cm]	Diameter [cm]	Surface area [m ²]	Features
DuPont	BW30-2540	133	3,3	100	6	2,6	BW-standard
TriSep	2540-ACM4-TSF	125	5,1	100	6	2,6	BW-standard
DuPont	XLE-2540	133	7,4	100	6	2,6	TW-low energy
LG	BW-2540 UES	125	9,1	100	6	2	TW-antifouling
TriSep	2540-XN45 -TSF	83,5	4,6	100	6	2,4	NF-standard

MBR setup can produce about 10L of wastewater per week so tests on modules are impossible – lab scale tests on FS membranes

Measuring principle

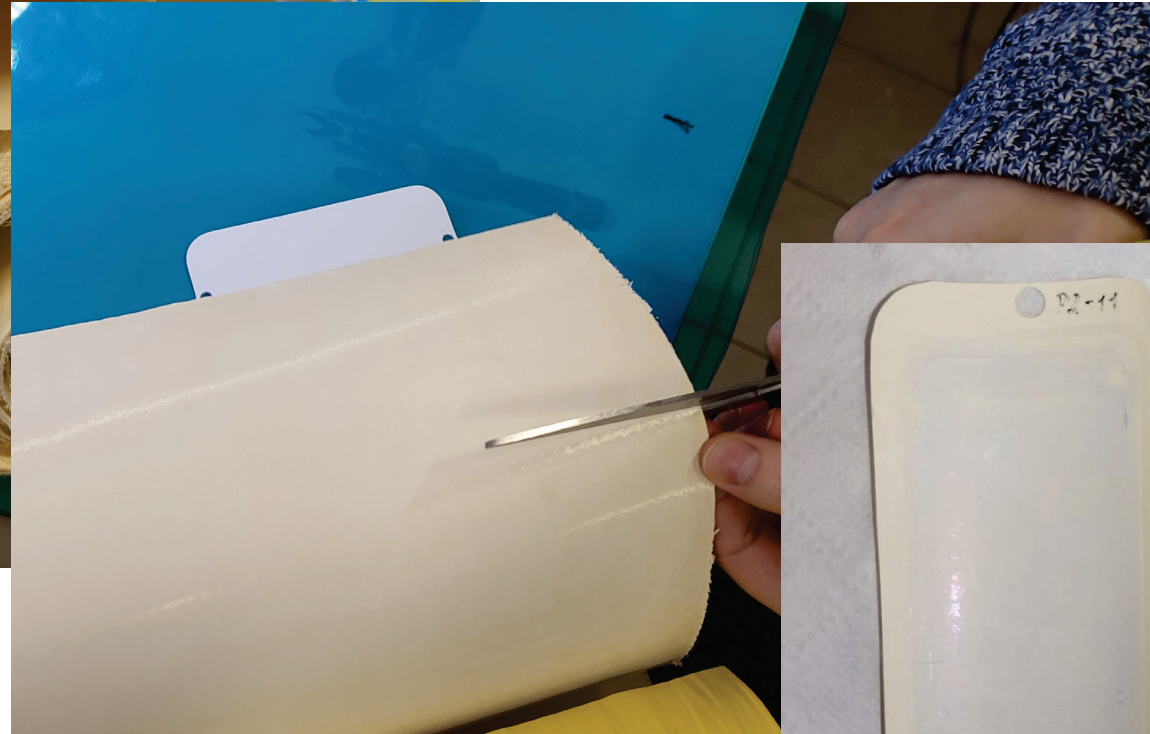
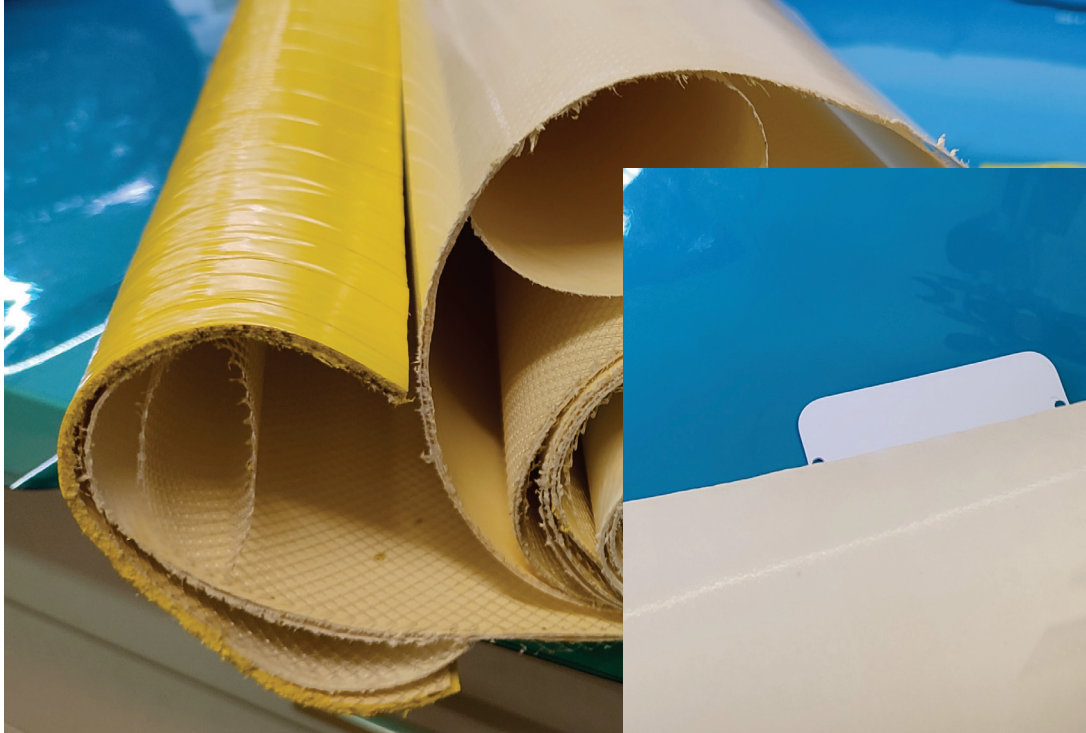


$$R = \frac{C_F - C_P}{C_F} \cdot 100\% \quad \text{Rejection}$$

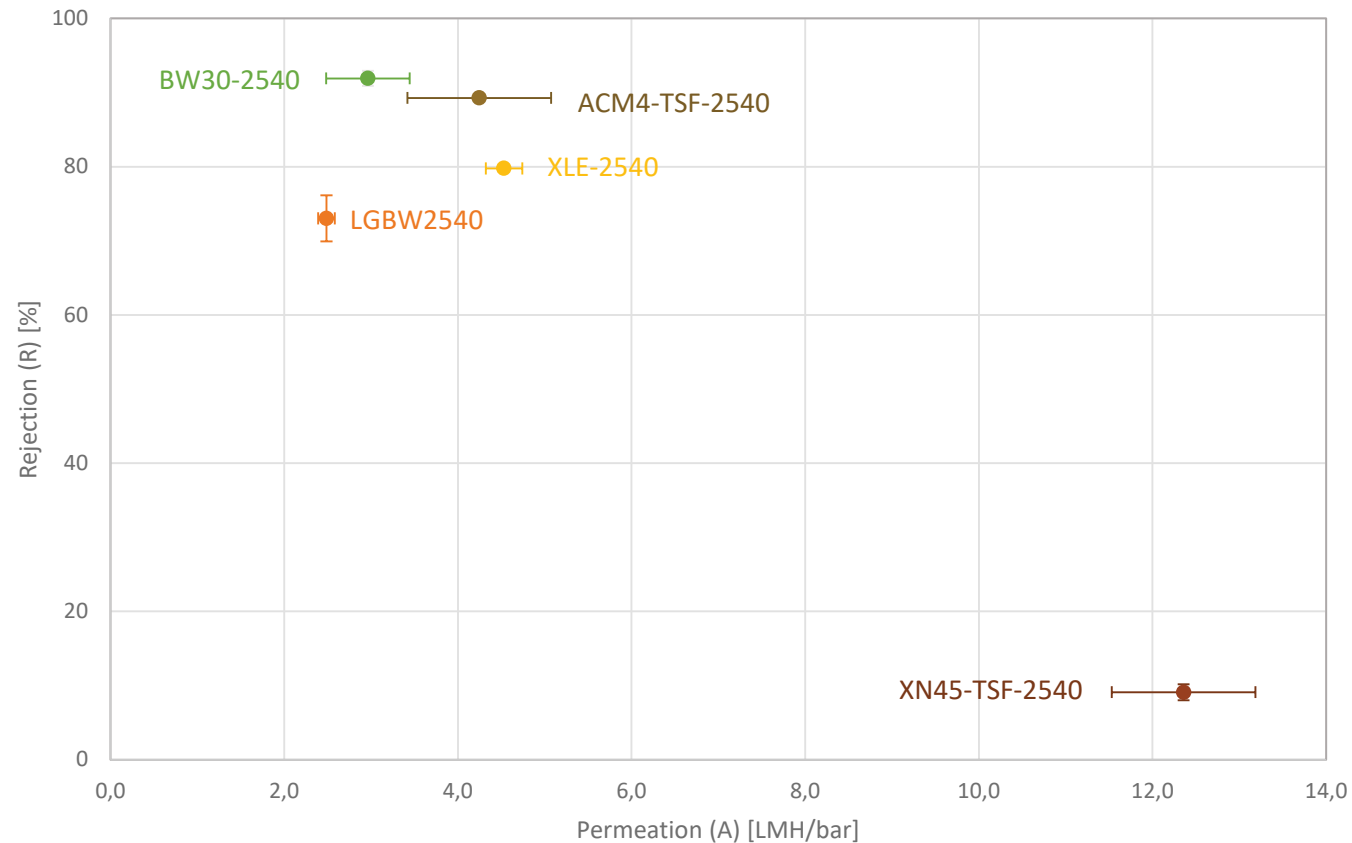
$$A = \frac{\Delta V}{S \Delta t \Delta P} [\text{LMHBar}] \quad \text{Permeability}$$

$$\text{Rec.} = \frac{Q_P}{C_F} \cdot 100\% [\%] \quad \text{Recovery}$$

Extraction of the membranes

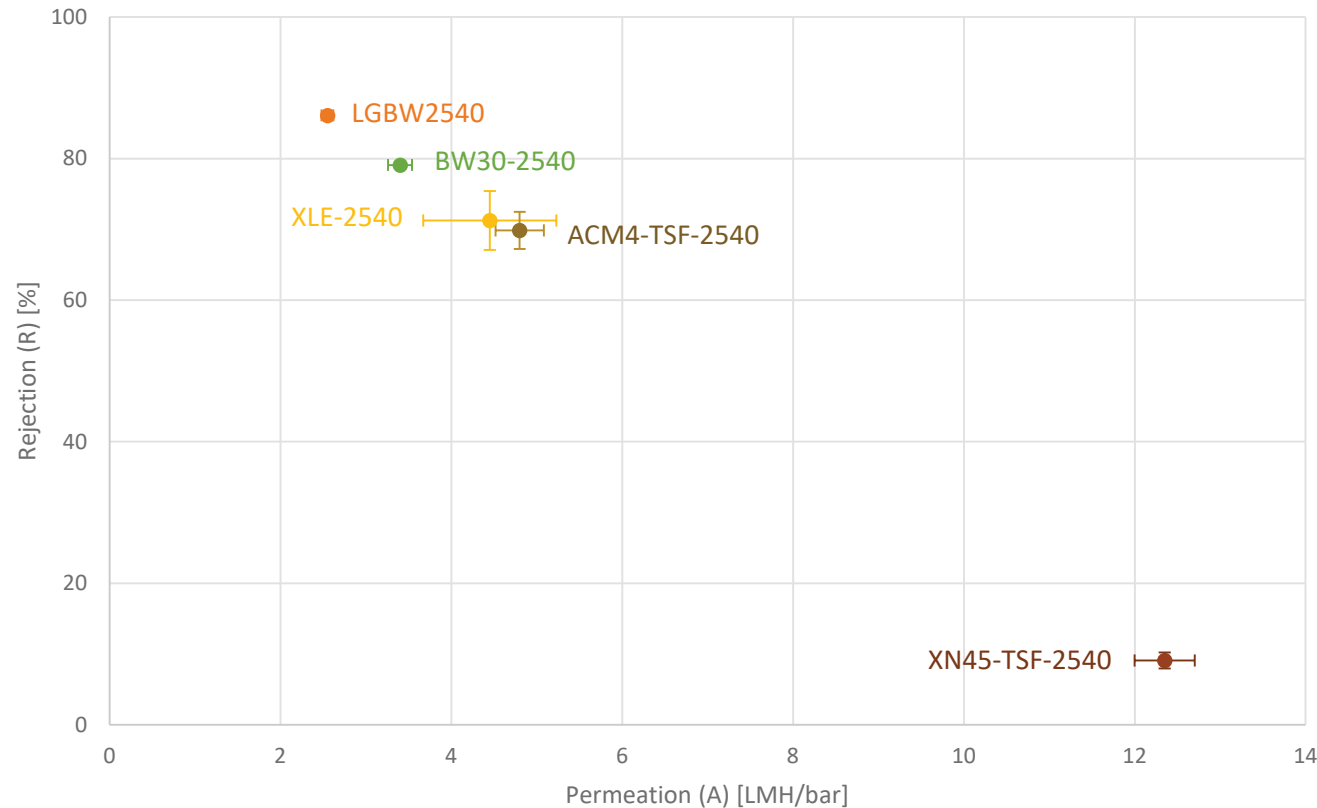


Results – model salt NaCl



Temp = 25 °C, P = 16 bar, C_{Feed} : 1 g/L NaNO₃, Feed flow rate = 9 LPH, n = 2, Recovery = 6 %

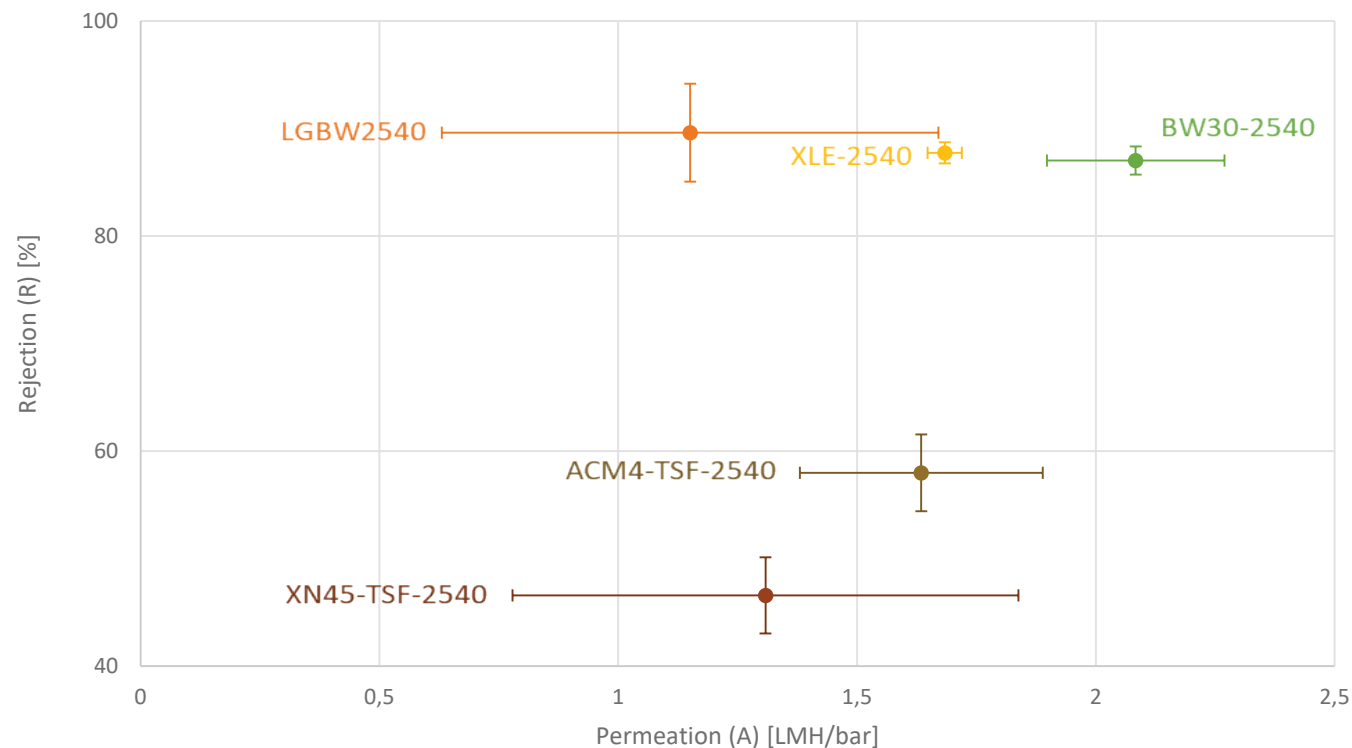
Results – model salt NaNO_3



Temp = 25 °C, P = 16 bar, C_{Feed} : 1 g/L NaNO_3 , Feed flow rate = 9 LPH, n= 2, Recovery= 3% (7%*)

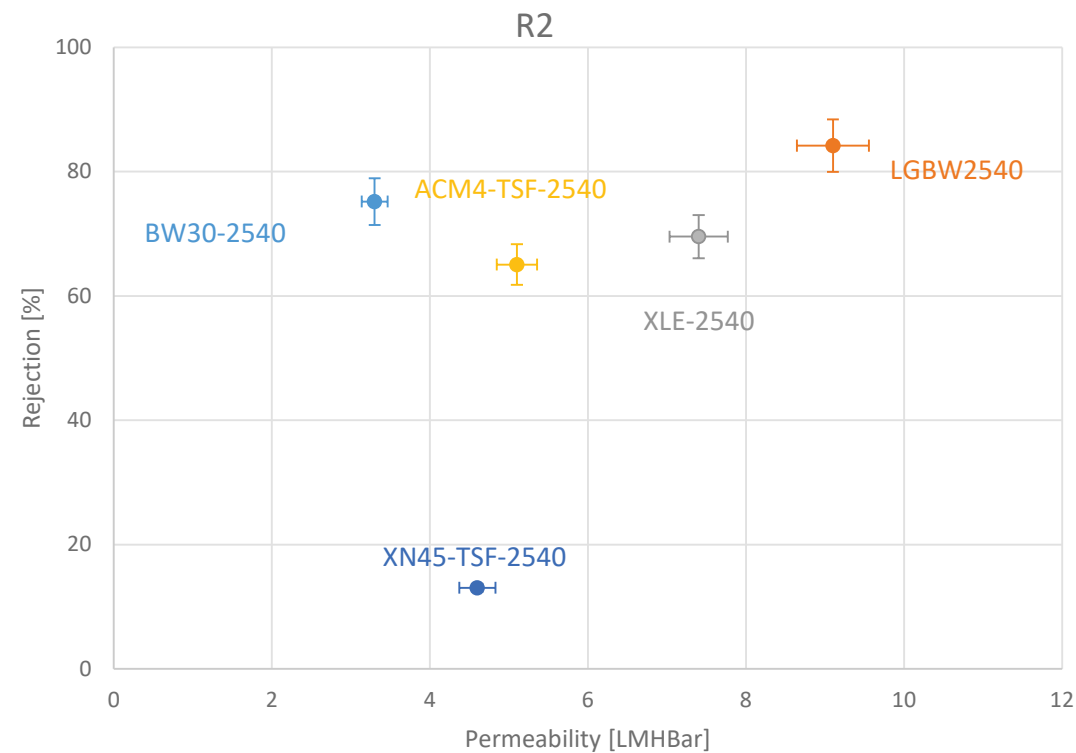
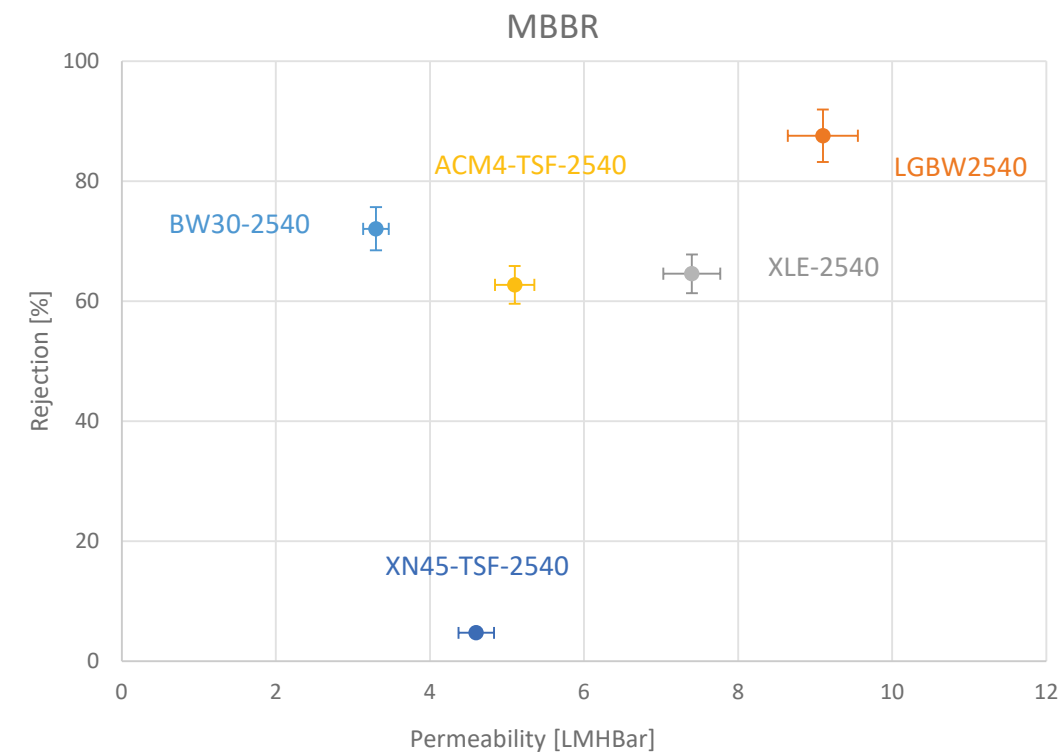
* For XN45 – TSF - 2540

Results – Raw water

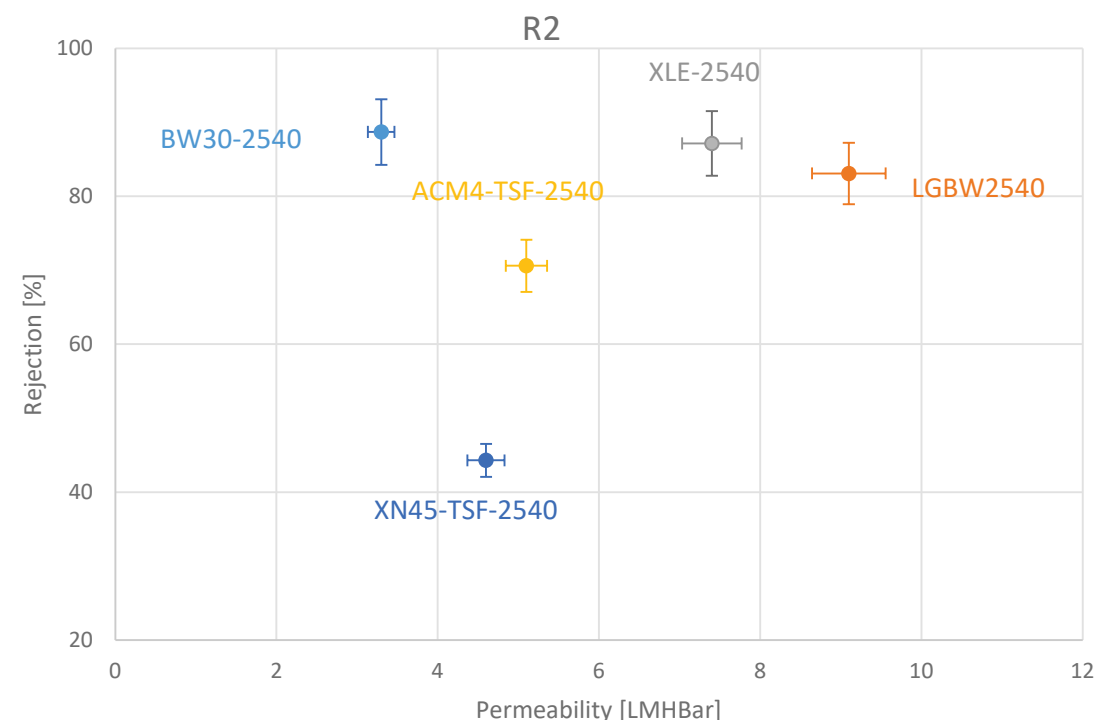
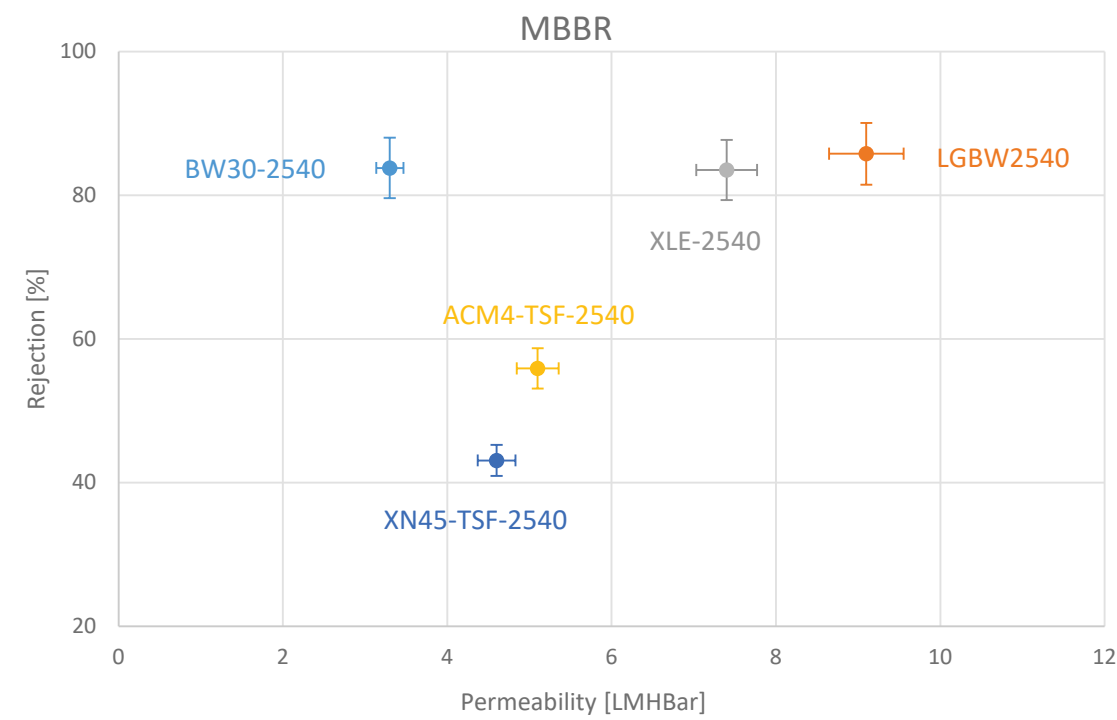


Temp = 25 °C, P = 16 bar, C_{Feed} : Raw water, Feed flow rate = 9 LPH, n = 4, Recovery = 2%

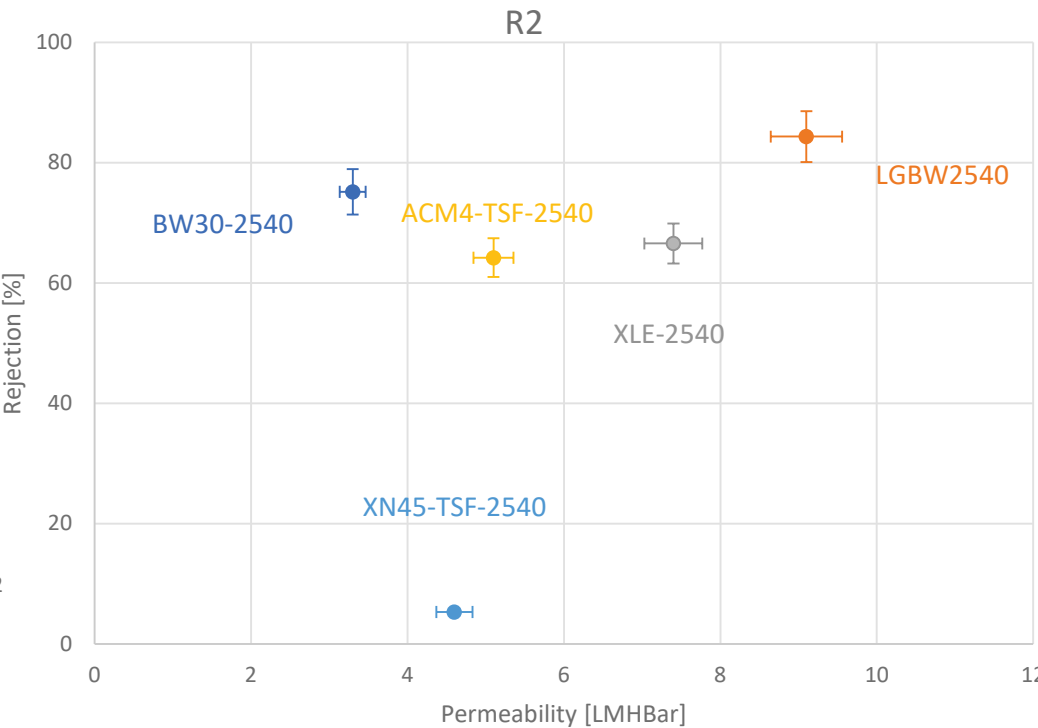
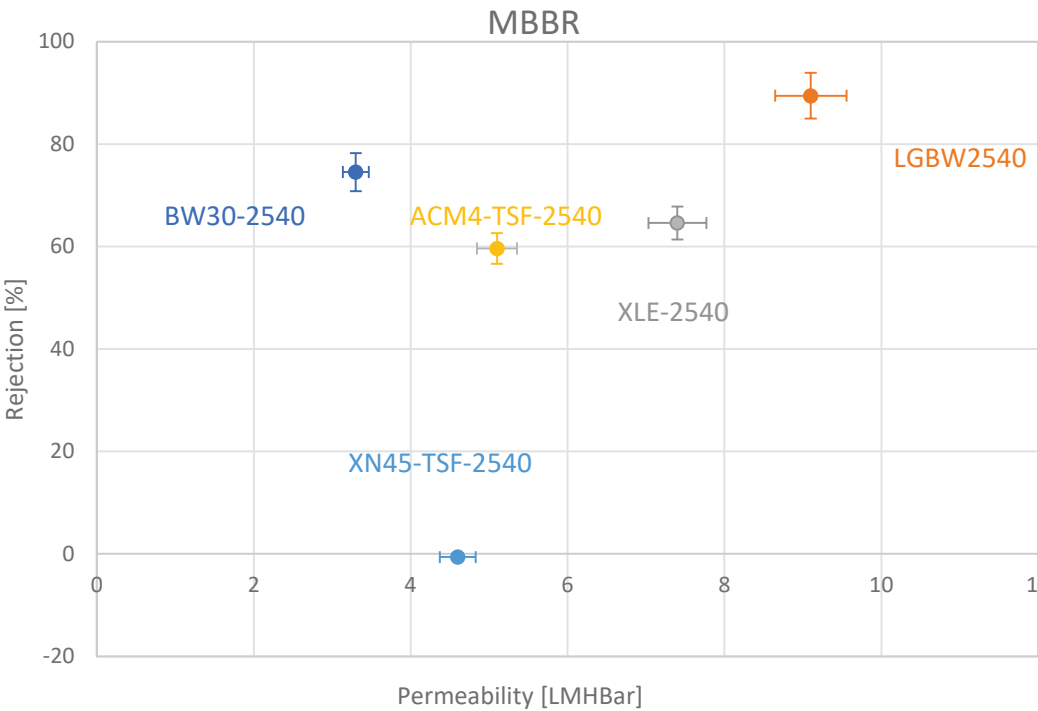
Results – TN



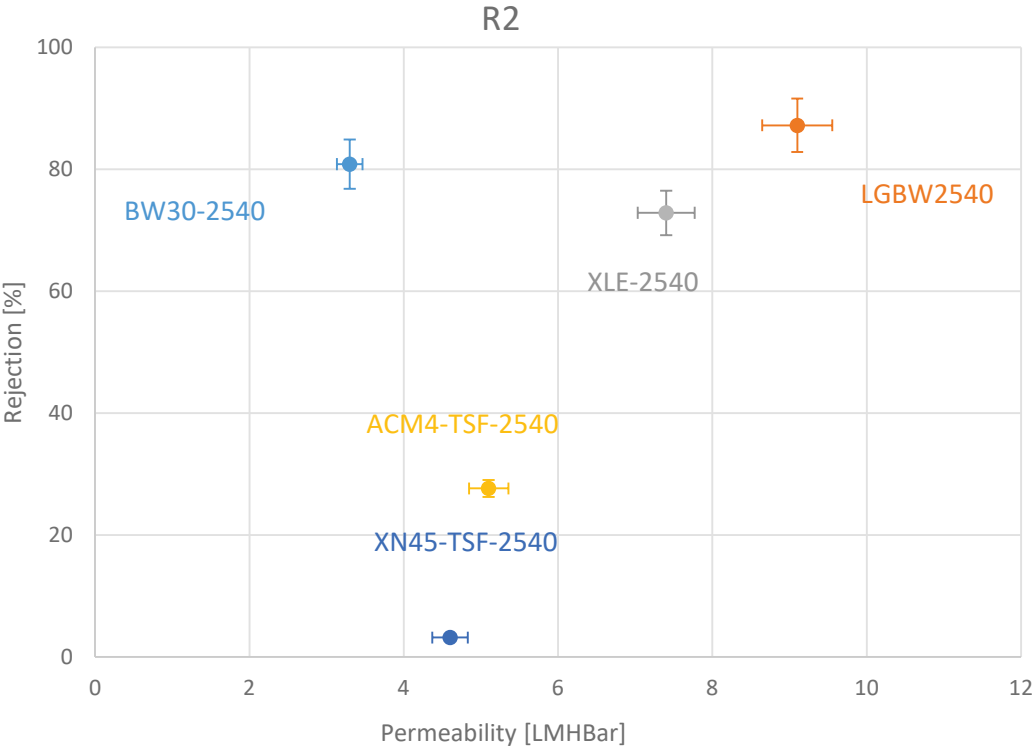
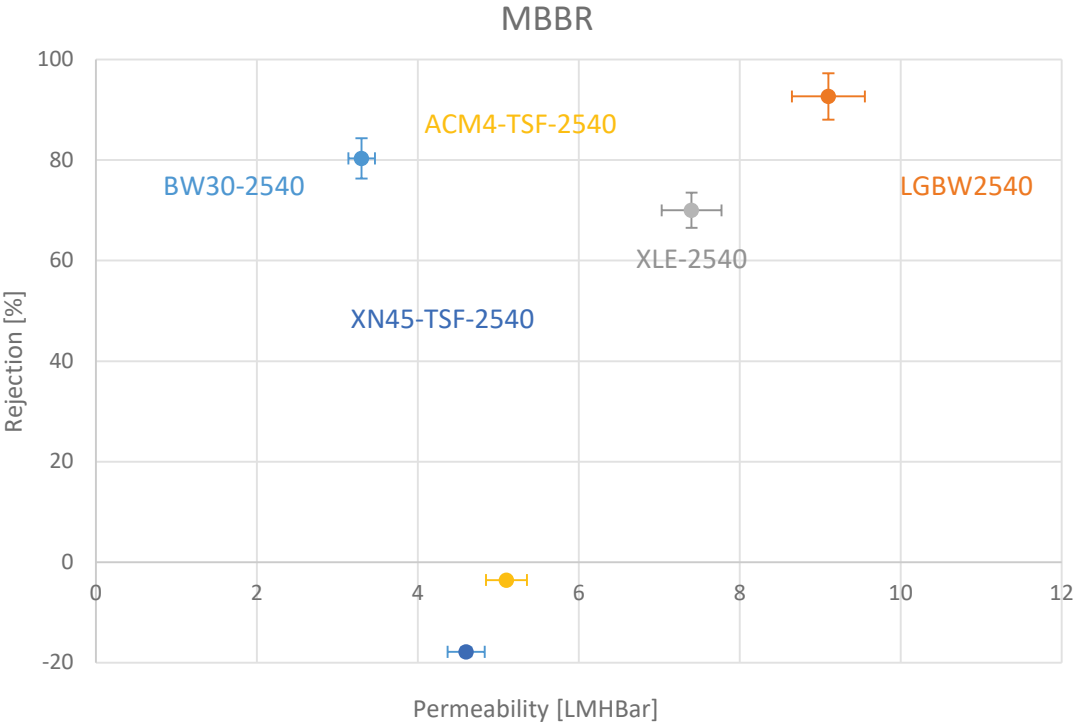
Results – TOC



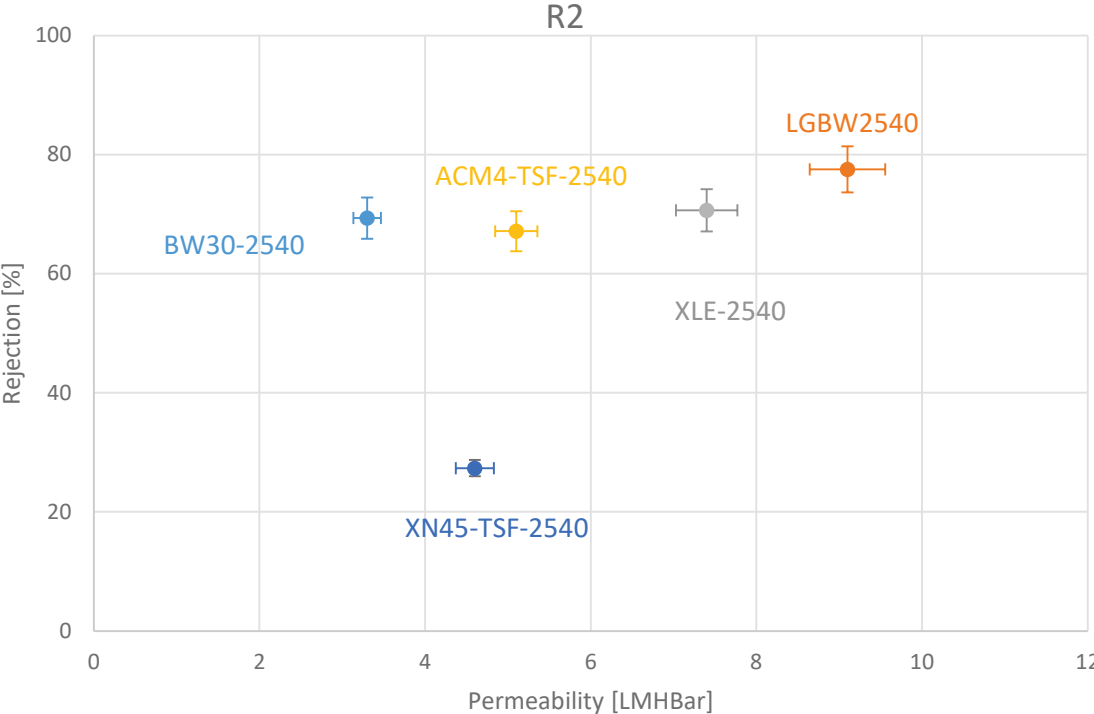
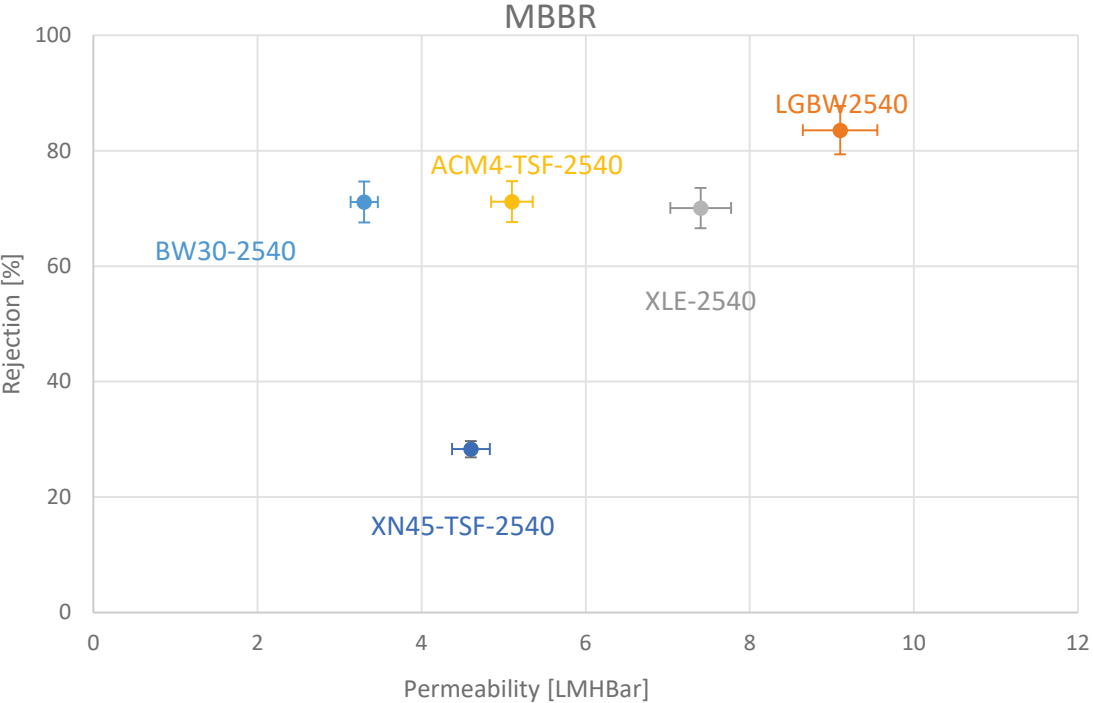
Results – NO3-N



Results – NO2-N



Results – NH4-N



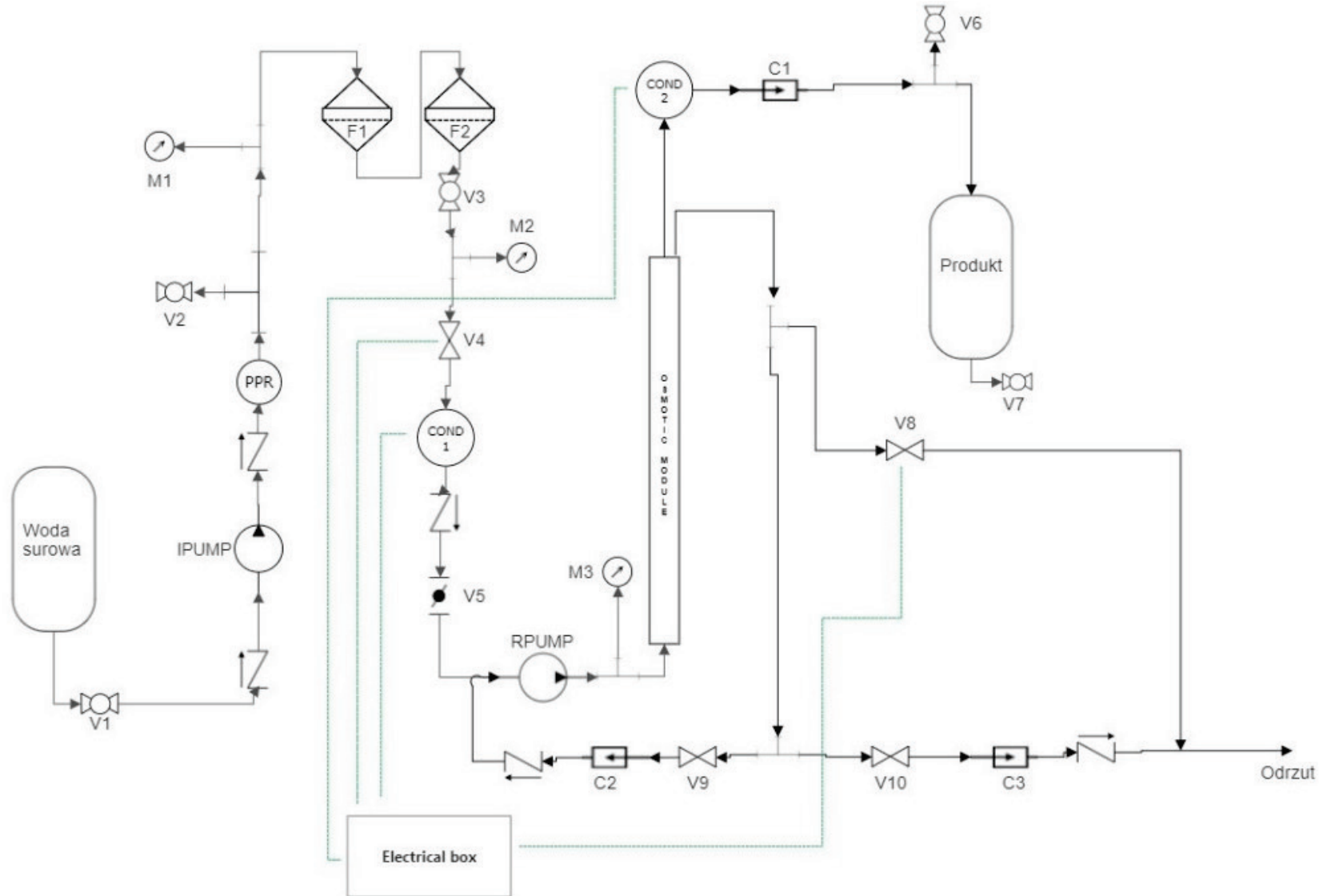
Conclusions (module selection)

- Commercial BW and TW membranes have similar rejection and permeability for model NaNO_3 solution
- NF membrane (TriSep XN45–TSF-2540) is not suitable for rejection of NaNO_3
- TriSep membranes (XN45–TSF-2540 and ACM4-TSF-2540) have poor rejection (based on conductivity) but also when rejection based on TN, TOC, NO_3^- , NO_2^- and NH_4^+ are taken into account
- **Membrane Dupont Filtec BW30-2540 and LG BW-2540 UE gives highest rejection and gives similar performance in all solutions tested therefore were selected for further tests on modules.**

Next steps

- **2 membranes qualified for further test**
 - Dupont Filmtec BW30-2540
 - LG BW-2540 UE
- Further tests on the modules
 - Long term performance
 - Fouling
 - Influence of the recovery
 - Tests of the selected modules with model solutions
 - Tests of the selected modules with waste water 2m³

Testing setup scheme



Pilot scale set up for 2.6m² modules



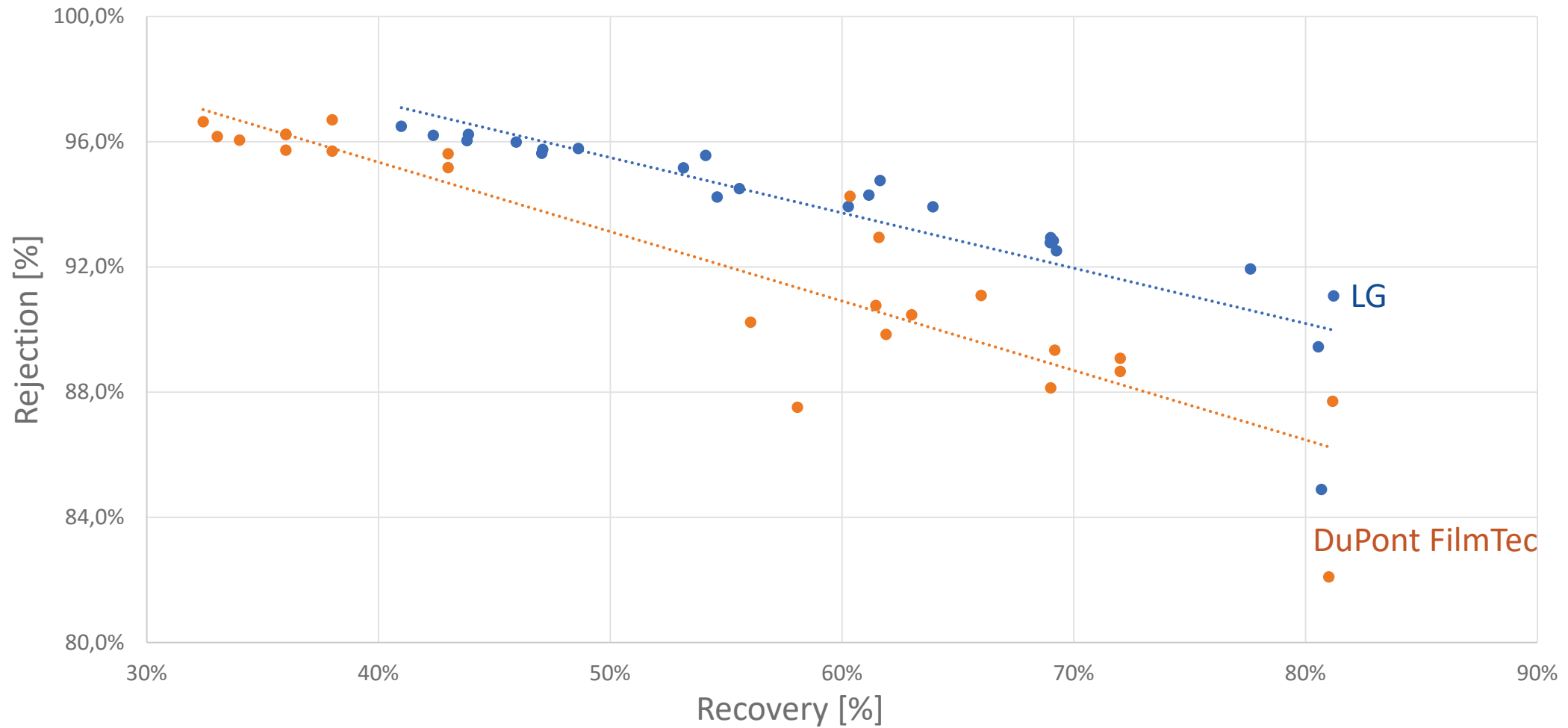
Aufbau der Pilotmembrananlage für 2,6m² Filtrationsmodule



Module used



Results – model NaNO_3 solution

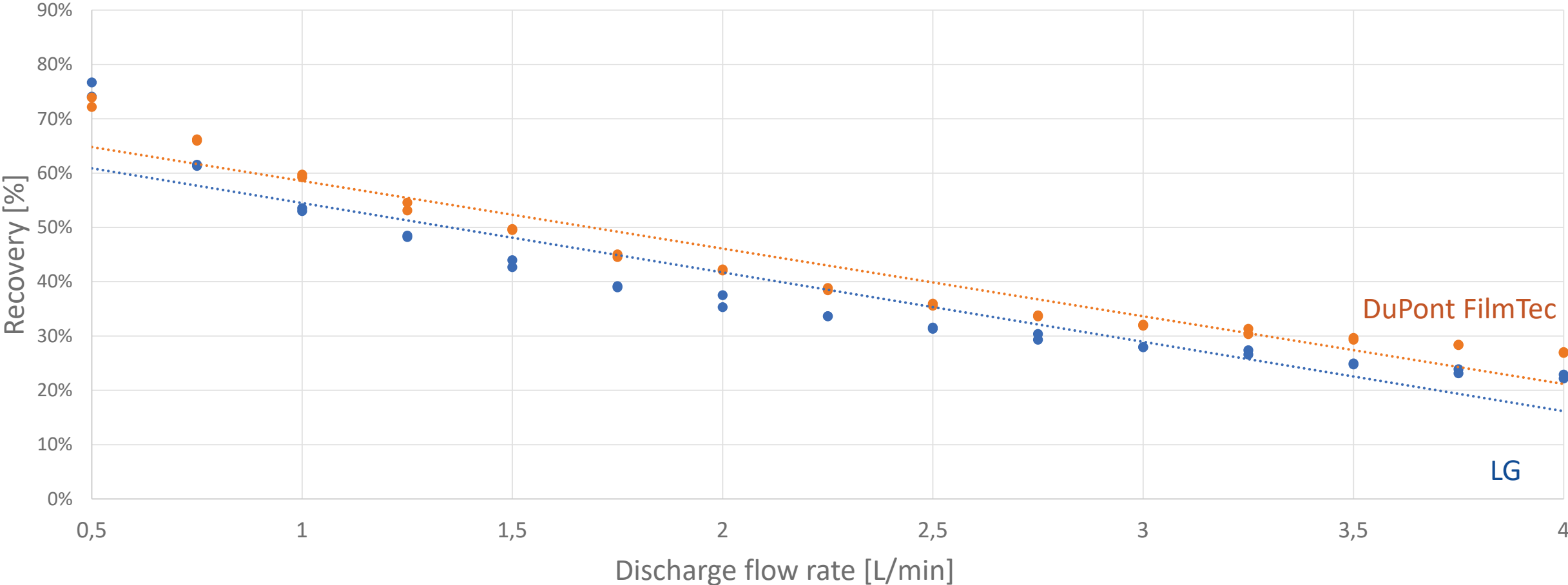


Temp. 17.6°C – 35.9°C ; P = 15 bar; C_{feed}: 0.9 g/L NaNO_3

Conclusion

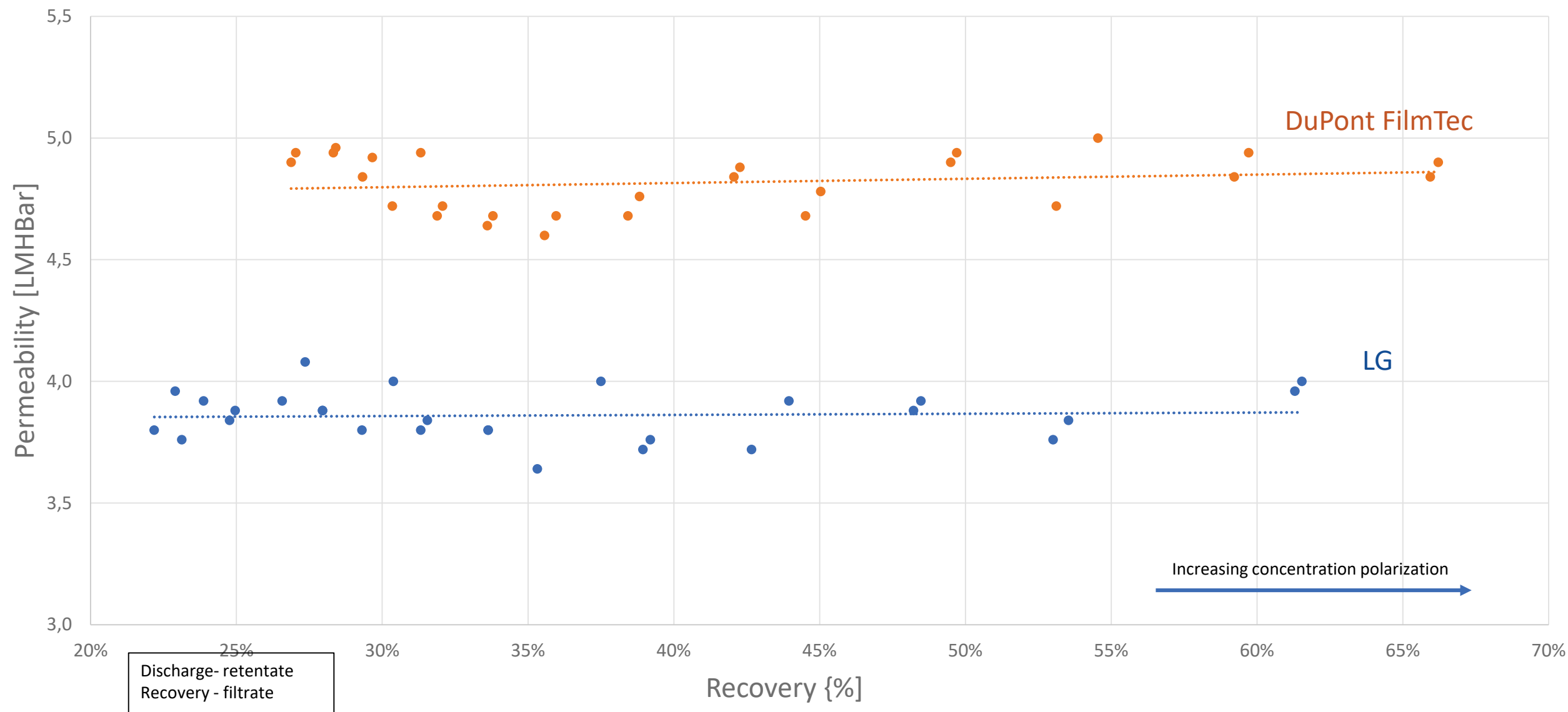
- Increasing recovery, rejection for both tested RO modules drops
- For all recoveries, **LG module BW-2540 UE shows better rejection and permeability** than Dupont FilmTec BW30-2540
- With increasing recovery, permeability for both modules drops.

Results – Raw water

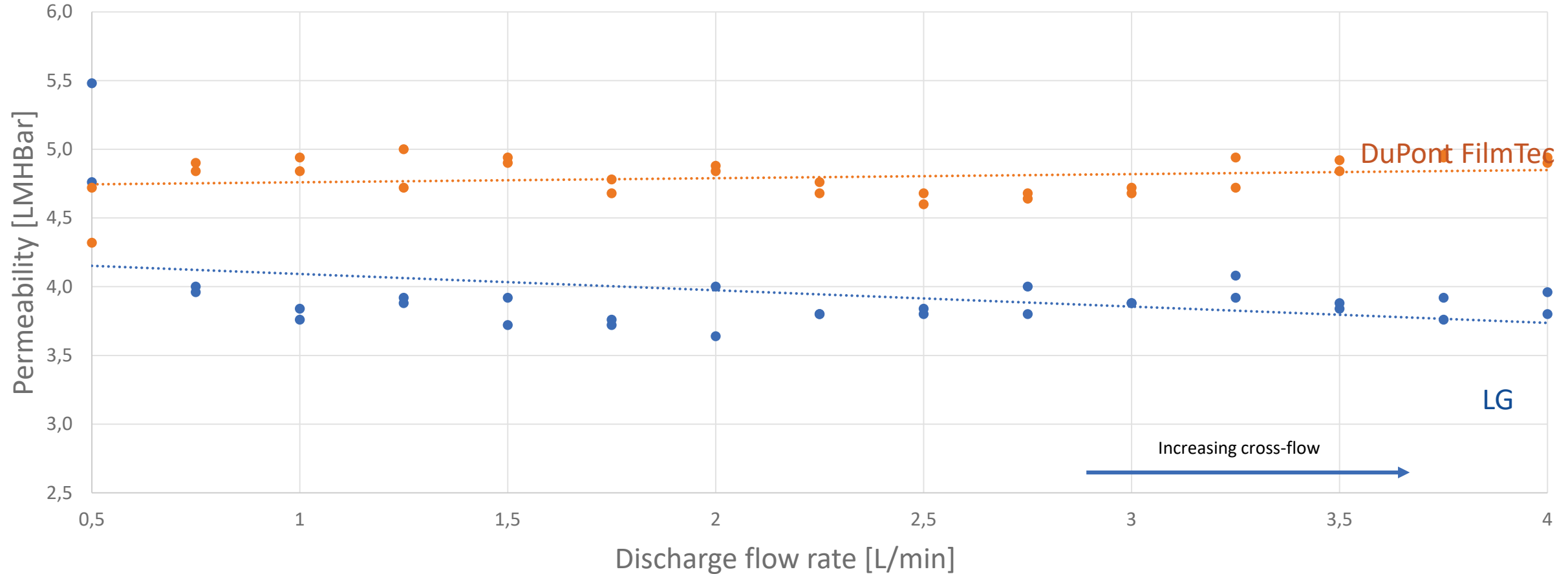


Discharge- retentate
Recovery - filtrate

Results – Permeability to Recovery



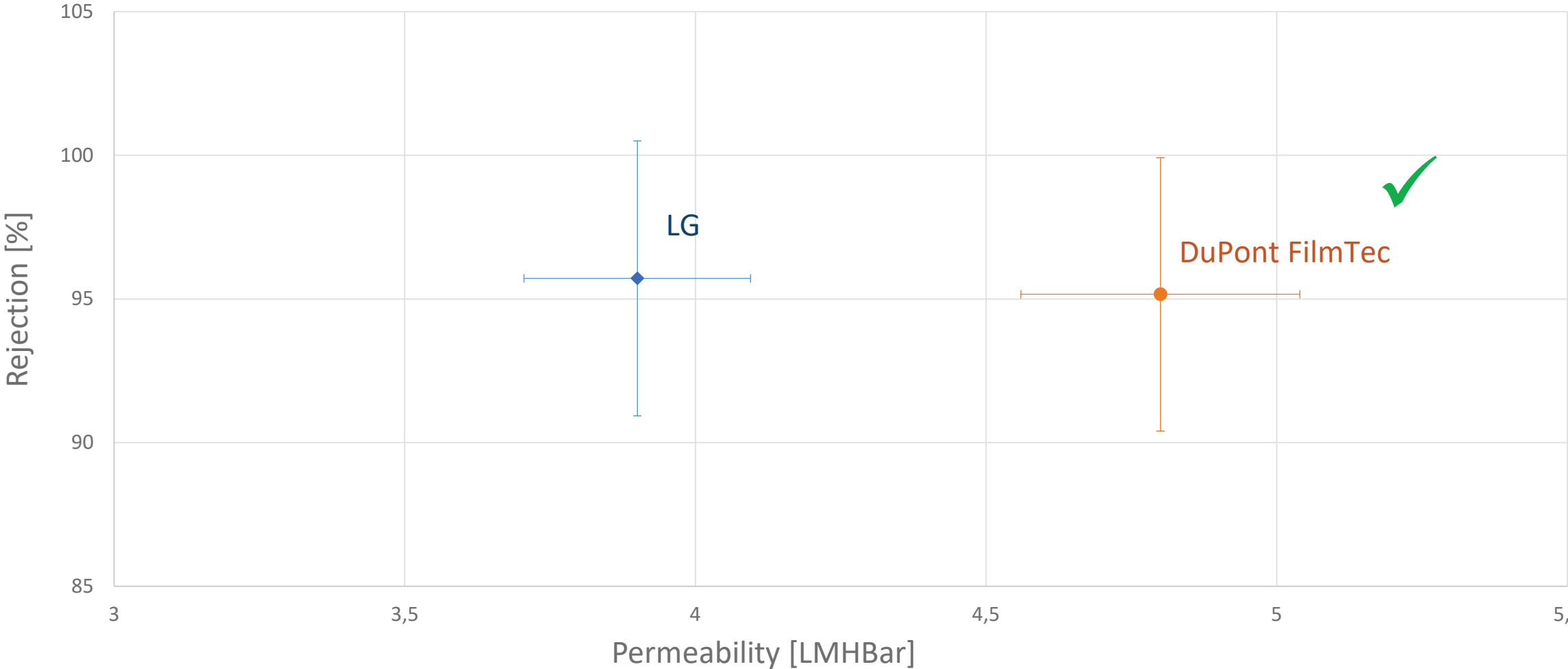
Results – permeability from discharge flow rate



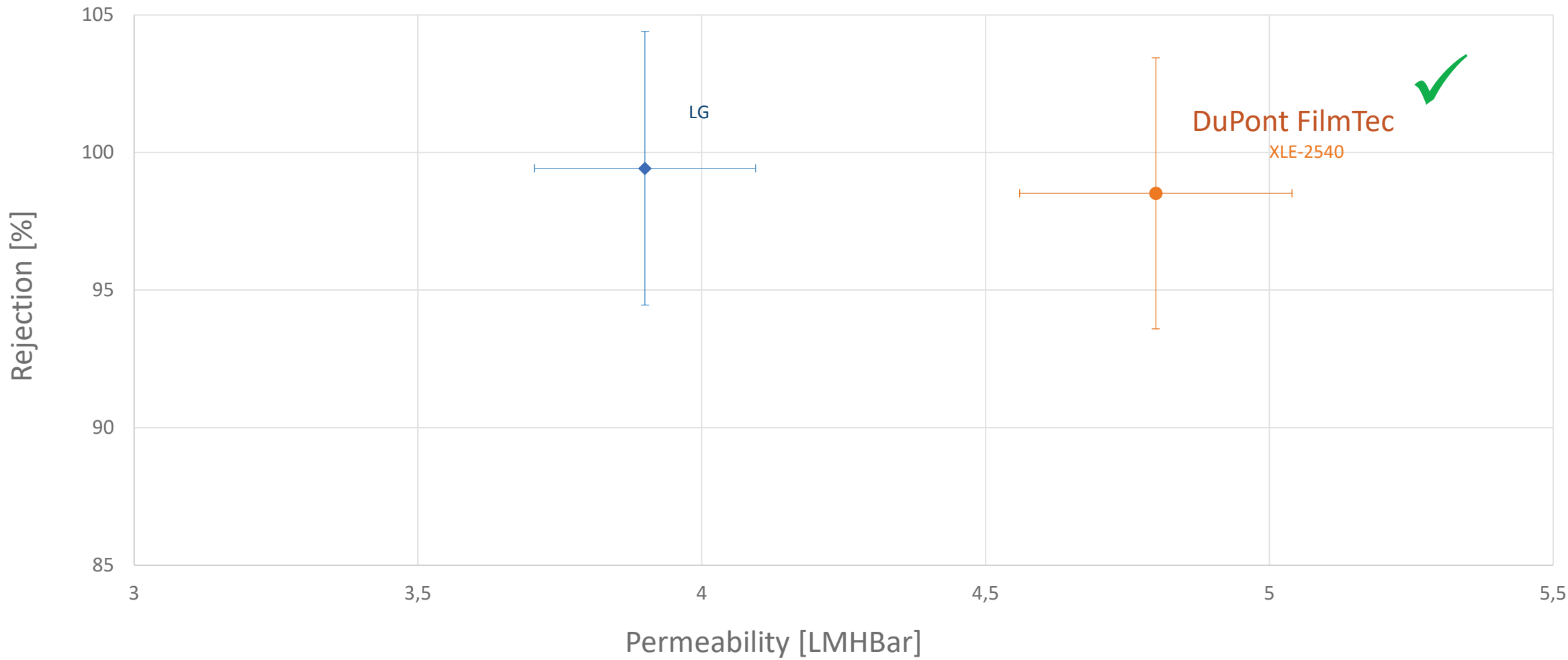
Discharge- retentate
Recovery - filtrate

Temp. 16.8°C – 21.1°C ; P = 15bar

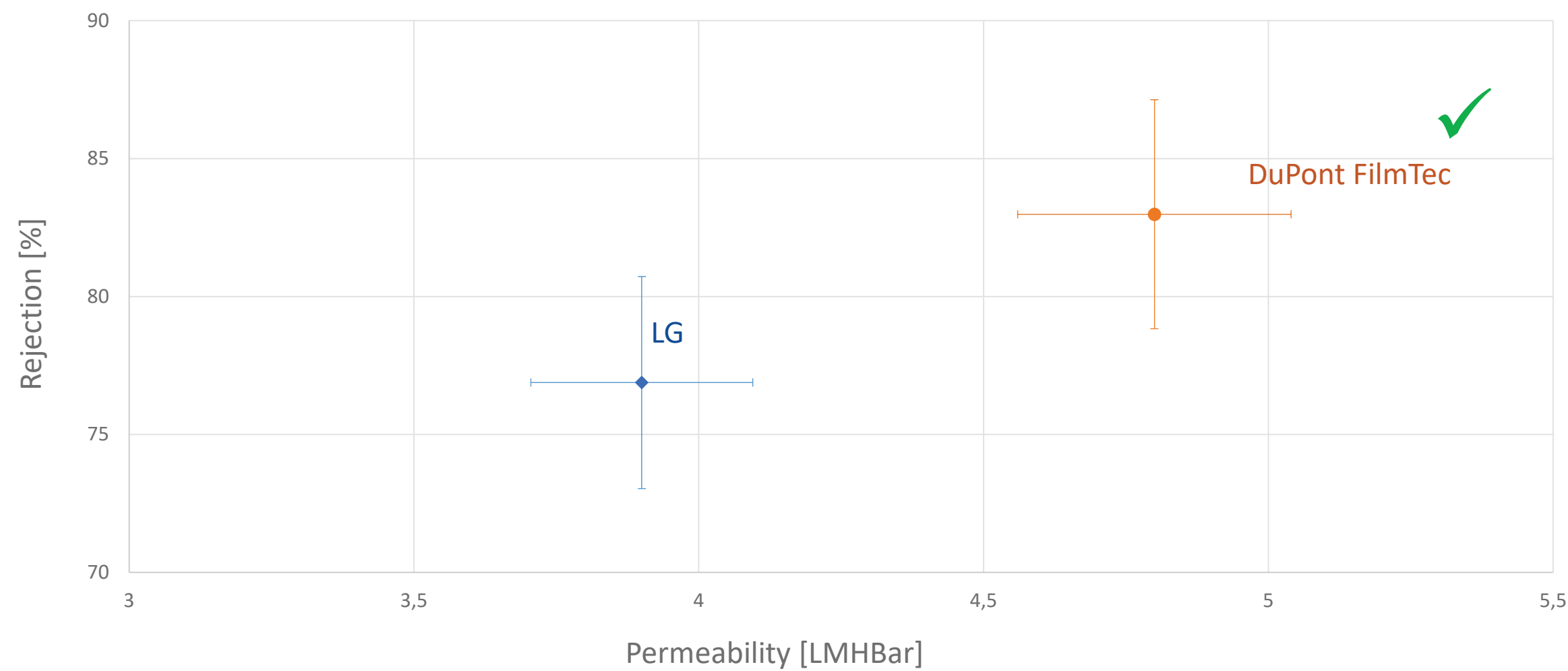
Results – NO3-N



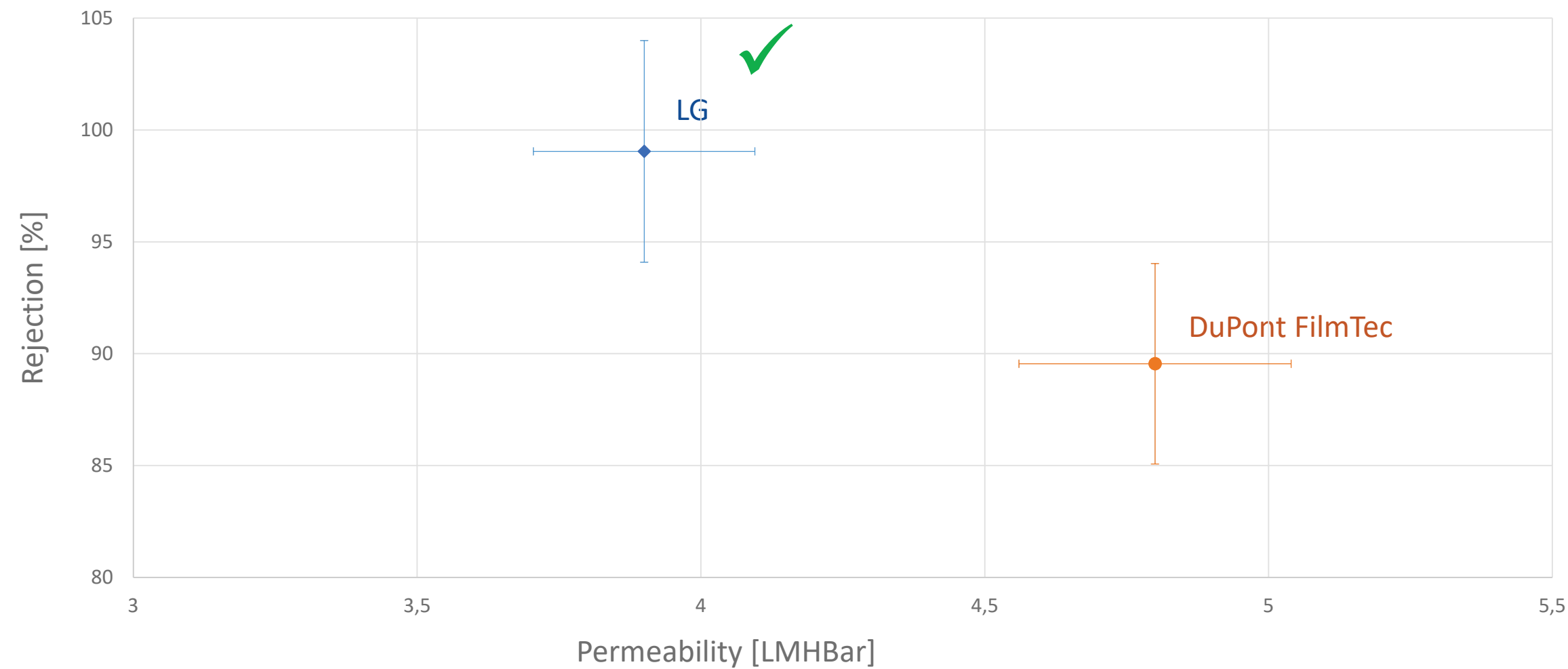
Results – NO2-N



Results – TOC



Results – TN



Conclusion

- Test with raw water show that higher permeability have Dupont Filmtec BW30-2540 module than LG BW-2540 UE for all tested recoveries.
- Rejection of the solutes based on conductivity was comparable for both tested modules
- Rejection of $\text{NO}_3\text{-N}$, $\text{NO}_2\text{-N}$ and TOC was comparable or higher for Dupont Filmtec BW30-2540 module but measured permeability was significantly higher.
- Only for TN, LG BW-2540 UE module shows higher rejection than Dupont Filmtec BW30-2540
- It is recommended to further design the system with Dupont Filmtec BW30 modules

Process water quality

Process simulation was run in DuPont Wave 1.82b since we decided to use Dupont Filmtec BW30 modules



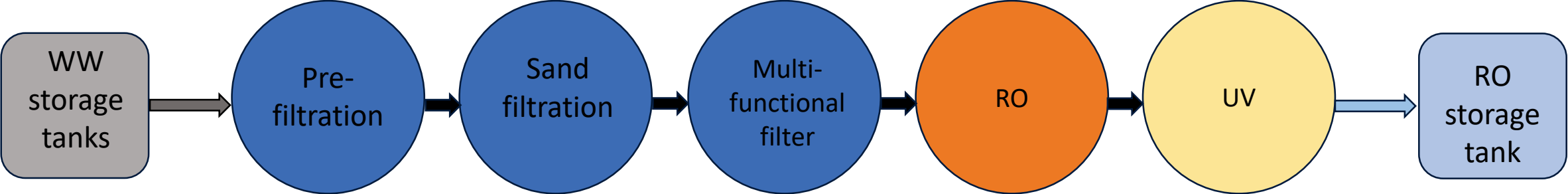
To run the process simulation balance of ions had to be completed. For the simulation we assumed following:

Parameter	Unit	Value
Conductivity at 25°C	[uS/cm]	<4.3
Aerobic microorganism	[KBE/mL]	< 0.1
Bacterial endotoxins	[EU/mL]	< 0.25
Hydrocarbons	[mg/L]	< 5
Heavy metals	[mg/L]	< 0.1
Aluminum	[ppb]	10
Nitrate	[mg/L]	< 0.2

➤ **The total amount of wastewater to be treated per day ca. 6.1 m³/d:**

- 5.1 m³/d sanitary wastewater
- 1.0 m³/d industrial wastewater

Block diagram



6.1 m³/day (250 L/h)

Parameter	Conc.
TOC [mg/L]*	16.7
DOC [mg/L]*	7.5
IC [mg/L]*	28.2
TN [mg/L]*	151
ON [mg/L]*	5.6
NH ₄ -N [mg/L]*	3
NO _x -N [mg/L]*	142.4
NO ₃ -N [mg/L]*	140
NO ₂ -N [mg/L]*	2.4
oPO ₄ -P [mg/L]*	6.8
TDS** [mg/L]	2315

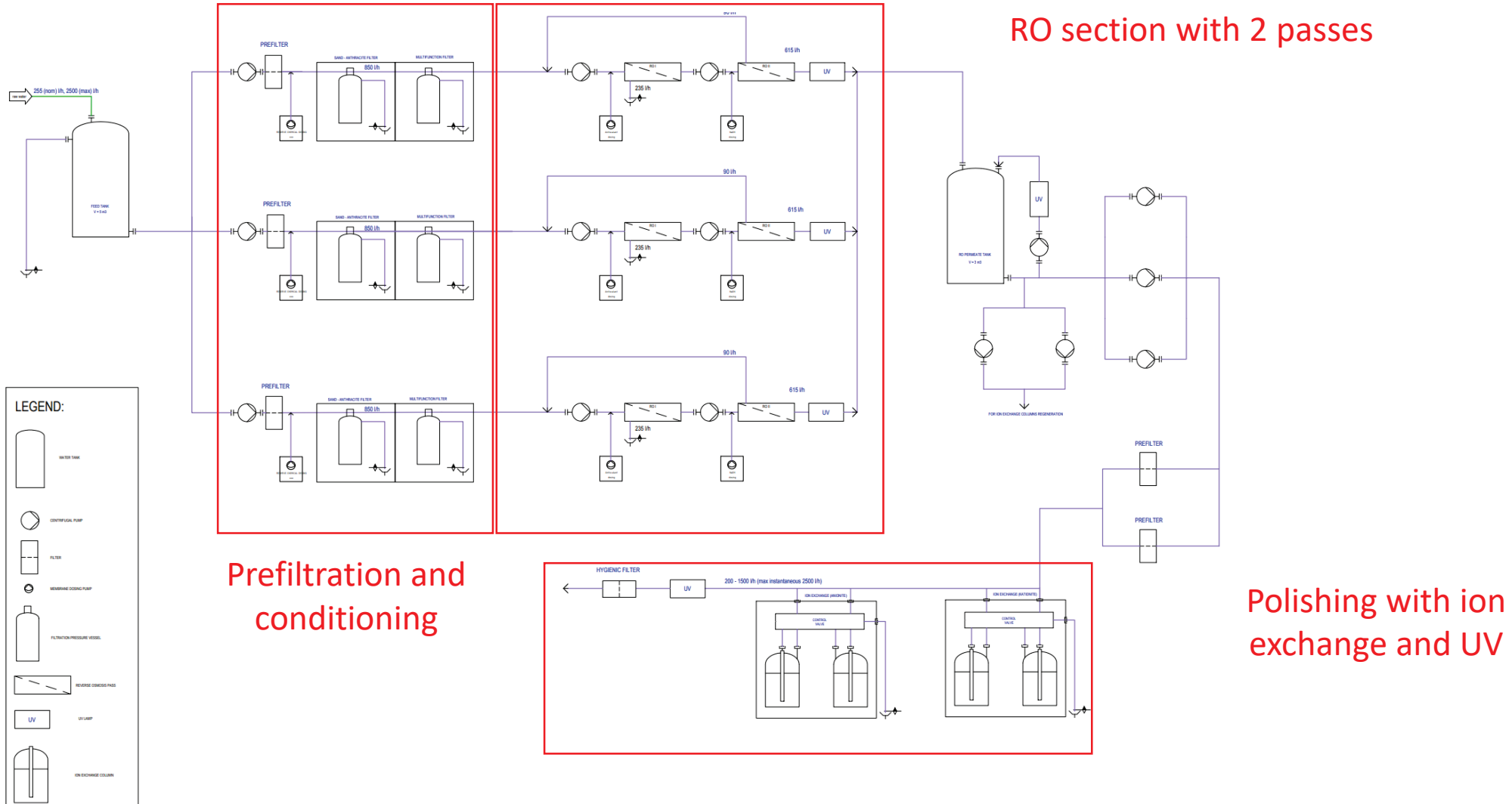
For simulation missing ions
were fileld with Na⁺ and Cl⁻



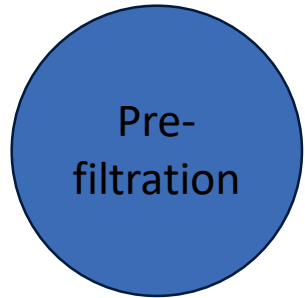
* measured by TU Dresden
** measured by Me-Sep
*** Assumed for simulation

Parameter	Conc.
TOC [mg/L]*	16.7
DOC [mg/L]*	7.5
IC [mg/L]*	28.2
TN [mg/L]*	151
ON [mg/L]*	5.6
NH ₄ -N [mg/L]*	3
NO _x -N [mg/L]*	142.4
NO ₃ -N [mg/L]*	140
NO ₂ -N [mg/L]*	2.4
oPO ₄ -P [mg/L]*	6.8
TDS [mg/L]**	2315
Na ⁺ [mg/L]***	503

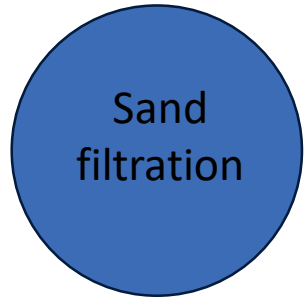
Block diagram



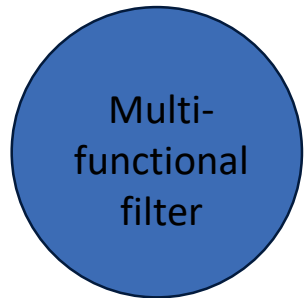
Prefiltration and conditioning



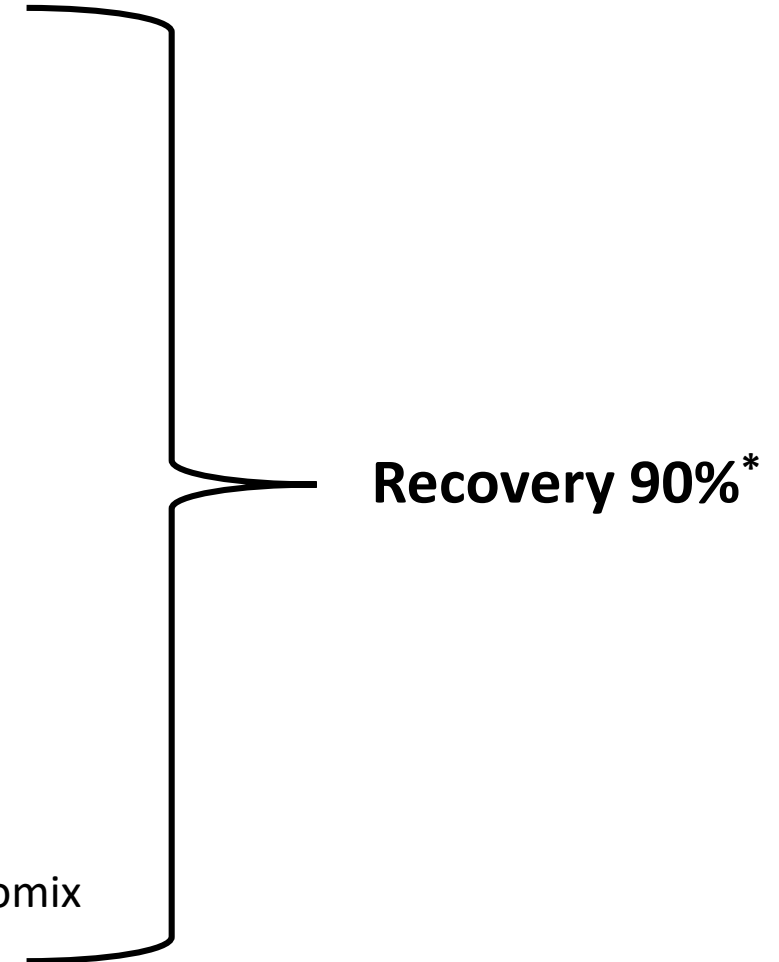
Removal of large particles and biology



Fine filtration and removal of organic compounds

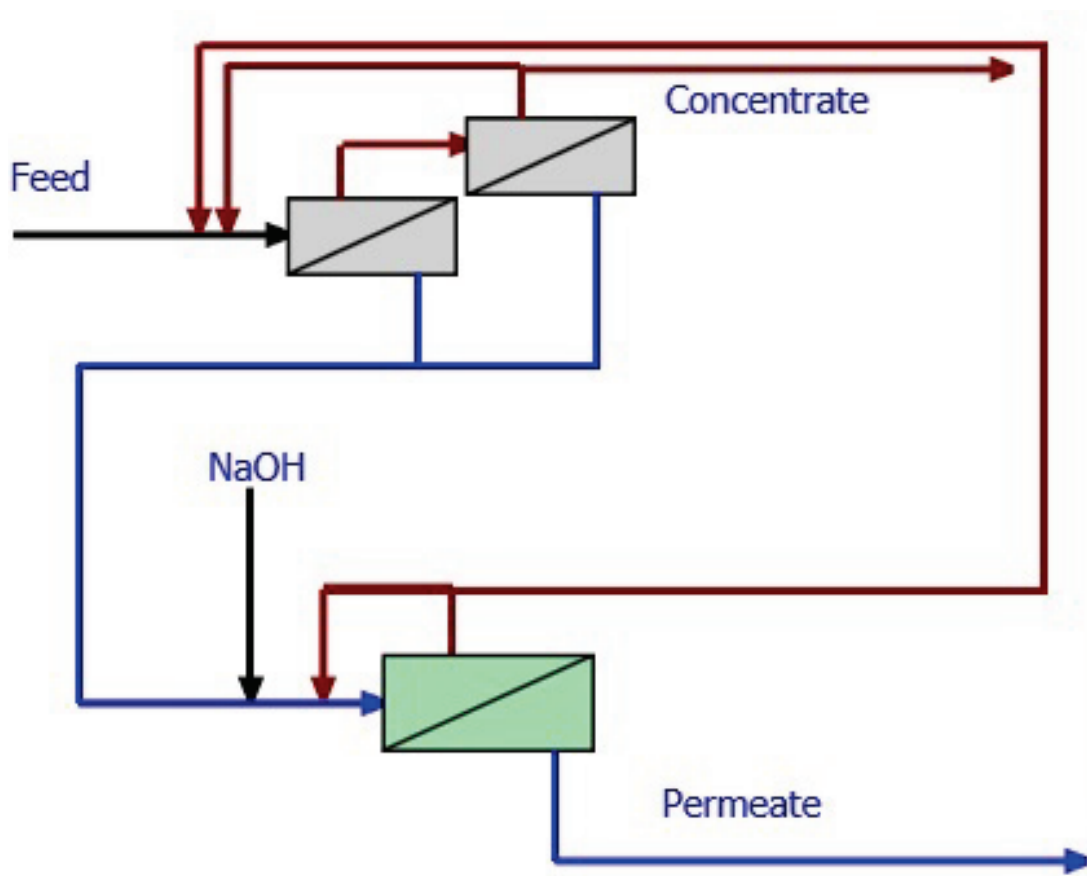


To remove suspensions, Fe, Mn, organics and hardness, resin Elcomix



*10% used for backwash

RO unit



Pass I: 2 stage RO unit with 6 BW30-4040 modules in 2 PV

Pass I: 2 stage RO unit with 3 SW30-4040 modules in 1 PV

Pass I: Recovery of the module: 6-8% - Pass recovery 36.6%

Pass II: Recovery of the module: 20 % - Pass recovery 55.4%

Total recovery 72.3%

Specific Energy: 1.69 kW/m³

RO unit – produced water quality

Parameter	Raw water	Pass I RO		Pass II RO	
		10°C	30°C	10°C	30°C
TOC [mg/L]	16.7	< 4	< 4	< 2	< 2
DOC [mg/L]	7.5	< 4	< 4	< 2	< 2
IC [mg/L]	28.2	5.9	7.3	0.3	0.7
TN [mg/L]	151	15.6	46.8	1	5.4
ON [mg/L]	5.6	< 1.5	< 1.5	< 0.5	0.5
NH ₄ -N [mg/L]	3	0.1	0.3	0.02	0.15
NO _x -N [mg/L]	142.4	14	45	0.52	4.7
NO ₃ -N [mg/L]	140	14	44	0.51	4.6
NO ₂ -N [mg/L]	2.4	0.2	0.7	0.01	0.1
oPO ₄ -P [mg/L]	6.8	0.4	1.6	< 0.05	0.05
Na ⁺ [mg/L]	503	32	109	1.2	10
Cond. [uS/cm]	3630	169	551	6	50
TDS [mg/L]	2350	113	374	4.5	35



Polishing is necessary !!!

Polishing with ion exchange



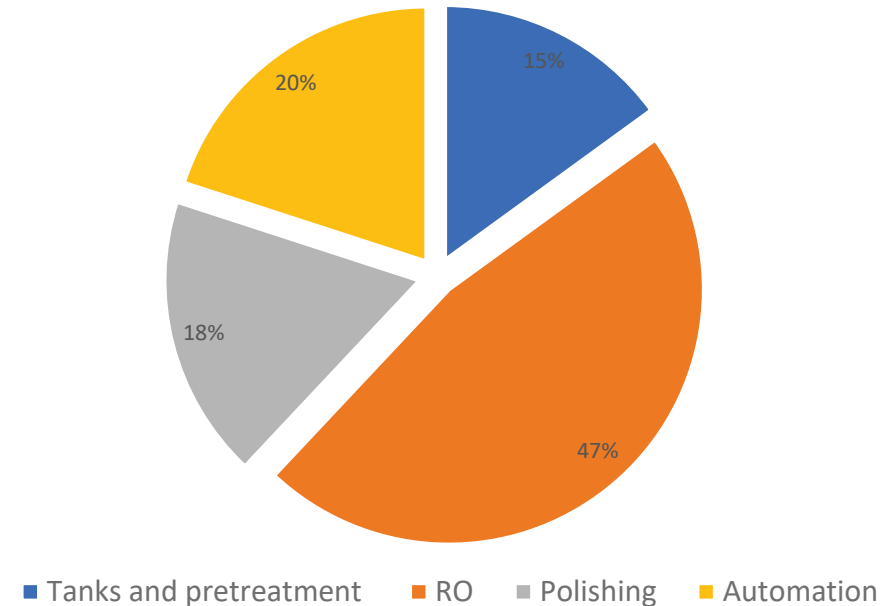
Kation exchange: AmberLite HPR1200 H
Anion Exchange: HPR4100 Cl

Parameter	Raw water	Polishing
TOC [mg/L]	16.7	< 1
DOC [mg/L]	7.5	< 1
IC [mg/L]	28.2	< 0.5
TN [mg/L]	151	< 0.15
ON [mg/L]	5.6	< 0.05
NH ₄ -N [mg/L]	3	< 0.1
NO _x -N [mg/L]	142.4	< 0.05
NO ₃ -N [mg/L]	140	< 0.05
NO ₂ -N [mg/L]	2.4	< 0.05
oPO ₄ -P [mg/L]	6.8	< 0.05
Na ⁺ [mg/L]	503	< 0.8
Cond. [uS/cm]	3630	< 4.3
TDS [mg/L]	2350	< 2.5

Summary

- 3 step process was proposed : Prefiltration, RO, Polishing and UV disinfection
- Total energy consumption of the system is estimated as 2.2-3.7 kW/m³
- Recovery of the system is about 80%
- Flexibility of the installation 0.2-2.5 m³/h
- System cost is about 300 - 500 kEuro
- Consumption of chemicals:
 - 33% HCl: 0.2 kg/m³
 - 30% NaOH: 0.3 kg/m³
 - Antiscaling agent : 6 g /m³

Cost structure of the installation



Conclusions

- Lab and pilot tests allowed empirical selection the best performing membrane for the purification of the water from the bioreactor to the proces water quality.
- The proces consists of 4 steps: Prefiltration, RO, Polishing and UV disinfection
- Full scale modular instalation for proces water recovery and recycling based on membrane technology was designed



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