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Executive Summary

Water is a vital resource for both human needs and the functioning of our ecosystem. Fresh water availability and water use have been a growing problem for centuries¹. Process industries are using huge amounts of water for cooling and/or production therefore sustainable water treatment solutions from a resource efficient and economical perspective have to be developed towards independency from freshwater resources and closed loop recycling and reuse of water. In addition, the importance of phosphorus, which is essential for plant growth and food production, increases correspondingly with population growth^{2,3}. Phosphorus and especially its acid are used in different industries such as the steel industry. To increase resource efficiency, modified membranes can be applied to purify and therefore to recycle spent acid. With the focus on finding efficient and economic solutions for water and resource recovery in process industries, various new technologies were tested on industrial pilot scale within the EU-funded project INSPIREWater.

At the ArcelorMittal case study, the magnetic separator and innovative reactor were tested to increase the cooling water treatment efficiency in steel industry regarding solid removal and lowering the corrosion potential. The acid resistant LbL membrane was applied in the NF process at the Sandvik case study for demonstrating its potential, improving spent acid recovery in steel industry. At the Clariant case study, the fouling resistant UF and RO membrane, the MOL[®]LIK-technology, the FO-HBRO[™] and FO-MD were demonstrated and evaluated for treating challenging wastewater of the chemical industry towards ZLD.

This report summarizes the innovation roadmap for each innovative technology increasing water and resource efficiency in process industries demonstrated in the INSPIREWater project. The innovation roadmaps contain a status analysis of the development, the market and economy of each technology as a basis to optimally understand and plan the actions needed for a successful upscaling, market entry and/or market expansion considering the fields of technology implementation, exploitation and dissemination.

¹ Lee (1951) Aristotle – Meteorologica – with English Translation

² Binder et al. (2009) Phosphorflüsse in der Schweiz. Stand, Risiken und Handlungsoptionen

³ Filippelli (2008) The global phosphorus cycle: Past, present, and future



1. Magnetic separator

1.1 Introduction

In iron and steel industry, 75% of the water is used for cooling processes as direct cooling of the hot steel e.g. in hot rolling or descaling processes and for indirect cooling applications as machine and furnace cooling with no contact to the hot steel. The remaining 25% of water are used for gas cleaning (blast furnace, converter) and material conditioning (slag granulation). The flow rates of the cooling circuits are between 100 m³/h and 10'000 m³/h.

The most common method for solid removal in e.g. hot rolling mills is the combination of a removal of coarse scale in scale pits, a further scale separation in settling tanks partly by use of polymer before filtration in discontinuous, pressure operated or continuous sand filters. During the filtration, high back flush water flows of about 3 - 5% (30 – 500 m³/h) of the treated flow with low solid contents of 1 - 4 g/L occur, requiring a post-treatment. This requires, depending on the selected technology combination the use of chemicals (polymer dosage) and/or energy (e.g. operation of a centrifuge). The treatment is carried out with a decanter or settling tank with a downstream filter press.

In the project INSPIREWater, one of the work packages focus on the investigations of the magnetic separation as a chemical free and energy saving technology for removing suspended solids in a hot rolling mill cooling water circuit, as mandatory pretreatment step before desalting and softening to recover the water, and decrease the blow down amount and the freshwater intake.

The magnetic separator using strong field permanent magnets is seen as an innovative technology allowing a chemical free and energy saving separation of particles, because of pressure less operation and use of permanent magnets. These are shifted arranged in several lines in a flowed tank leading to a direct hit of particles on the magnets or leading the particles in the range of the magnetic field. Both effects allow high removal rates and a removal of fine particles (< 20 µm). The removed particles will be collected of non-magnetic protection tube surrounding the magnets until cleaning.

Basis for the separation of particles on magnets is the property of magnetic fields to exert an attraction on particles (magnetic force). The magnetic force FM is influenced by the variable's particle volume, permeability constant of the vacuum, particle magnetization and the gradient of the magnetic field strength at the location of the particle.

The system-specific particle size magnetization is defined by the magnetic bulk susceptibility and the magnetic field strength.

The magnetic force on a particle is influenced by the particle volume, the permeability constant of the vacuum and the susceptibility of the particle. In terms of process technology, the gradient of the magnetic field at the location of the particle and the flux density can be influenced. Crucial for the efficiency of the particle separation are the distances between particle and magnet, the flow conditions and the spatial arrangement of the magnets. Further on, the flow velocity effects the particle separation and “collection” on the magnet. High flow velocities lead to a concentration of the particles at the impact point of the flow as well as on the back of the magnet by induced turbulence. In the case of a low flow velocity, the magnetic attraction receives greater influence. The separation of the particles takes place in this case over the entire surface of the magnet. Due to the lower volume flows, the system must be sized larger. One possibility for estimating the maximum flow velocity is the calculation of the particle velocity caused by the magnetic field, that of the variables permeability constant of the vacuum, difference of particle volumetric particle susceptibilities and fluid, saturation magnetization, background field strength generated by a magnet, particle radius, wire radius a and viscosity of the fluid depends.

1.2 Technology perspective

The mobile plant designed by BFI used for in the 6-month field trial at the hot rolling mill in Spain, has a flow rate of 50 m³/h, consisting of a tank with a weir and two movable nozzle bars made at the inlet and outlet side of the magnets. The cooling water was taken from a settling tank after a scale pit before it is pumped to the operational sand filters and further to the cooling tower before the getting back to the production (see Figure 1.1).

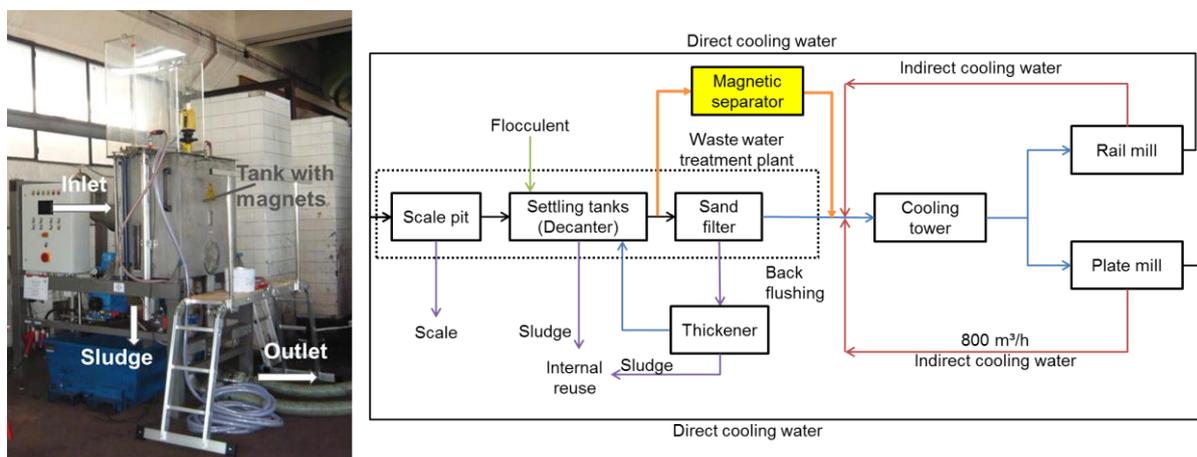


Figure 1.1 Magnetic separator installed at the hot rolling mill cooling water circuit



The mobile plant contains three lines of shifted arranged magnets, each line with 12 magnets. The magnets are installed in non-magnetic protection tubes with a theoretical maximum magnet load about 36 kg dried solid in total. Further components are the flow measurement device for regulating the wet pit pump flow rate and an overflow sensor inside the tank. The movement of the nozzle bars and the cleaning water flow can be activated or deactivated independent from each other for determining the minimum spraying time for tailored water saving magnet cleaning. In general, the cleaning process consists of the following steps:

1. Stopping feed flow
2. Emptying the tank
3. Moving out of the magnets by hydraulic system
4. Cleaning of protection tubes by spraying water from two movable nozzle bars
5. Moving nozzle bars in rest position above the water level without spraying water
6. Moving in of the magnets

The total cleaning time between stopping and restarting the feed flow is about 5 minutes.

The cleaning water pressure can be adjusted manually. The side window allows an optical process control. The automation of the magnetic separator allows a remote control and operation by e.g. variation of the parameters flow rate, spray time, time between two cleaning periods.

The applicability of the magnetic separator was demonstrated in a 6-month field trial from 09/2017 to 03/2018 with the treatment of 31'000 m³ of cooling water at a cooling water circuit of a hot rolling in parallel operation of conventional sand filters. During the field trials as part of the INSPIREWater project, the magnetic separator operated stable and reliable with constant outlet solid contents, even in the case of strongly varying inlet solid contents between 17 mg/L to 152 mg/L. The achieved solid content in the treated water close or below the 10 mg/l, which is the limit of detection for solid measurement with gravimetric methods. The determined cleaning water demand was about 40 l, leading to an occurring sludge amount between 51 l – 68 l with a solid content of 19 wt.-% to 31 wt.-%, depending on the magnet load before the cleaning. The ratio of occurring sludge and treated volume was between 0.002 and 0.010%.

1.2.1 Strengths and weaknesses of magnetic separator

The strengths and weaknesses of the magnetic separator are shown in Table 1.1 in the SWOT analysis. Strong field magnetic separation has a great economic potential as shown in terms of reduced energy for pumping, chemical dosage and especially decreasing the sludge amount about factor 15 up to 100 and more. Furthermore, the selective chemical free separation of iron particles opens the potential for a metallurgical reuse as iron ore or scrap substitute. High prices of strong field magnet materials as neodymium effecting the investment costs could



slow the application of the magnetic separator. Further on, only a low amount of sludge with a high solid content occur during magnet cleaning, decreasing the post treatment to a dewatering skip without energy or chemical usage.

Table 1.1 Overview of SWOT analysis of magnetic separator

INTERNAL	<ul style="list-style-type: none"> • Chemical free – metallurgical reuse of selective separated particles • Energy saving – pressure less operation • Low cleaning water demand • Low occurring sludge amount decreasing post treatment effort (only dewatering skip) – minimum factor 10 lower • Compact technology with no moving parts in direct contact with the solid containing cooling water avoiding wear and clogging 	<ul style="list-style-type: none"> • Temperature limitation to 60°C inlet water temperature • Insufficient cleaning in case of high viscose oil in cooling water which is only at temperatures > 40°C “water like”
	S	W
EXTERNAL	<ul style="list-style-type: none"> • Huge market potential in certain industries with different application fields e.g. treatment of different metal working fluids, gas washing waters and quenching liquids • Saving of operational costs (energy, chemicals) including low maintenance compared to conventional systems • Selective chemical free recovery of iron particles for metallurgical reuse 	<ul style="list-style-type: none"> • Increasing prices for permanent magnet raw materials • Sales price to high
	O	T

1.2.2 Key performance indicators of magnetic separator

During the field trials, it was shown that the magnetic separator is applicable to the hot rolling cooling water circuit for removing solids and to produce a low amount of high solid containing sludge. The solid removal efficiency was up to 93% leading to a constant outlet solid content close or below the 10 mg/l, even in the case of varying inlet solid contents about a factor of 3 (Table 1.2). About 40 l of cleaning water were required leading to a total sludge amount of 51 - 68l with a solid content of 19 - 31 wt.-%. The ratio of occurring sludge to treated volume is between 0.002 – 0.010% compared to 3 – 5% of sand filters.

Table 1.2 Overview of key performance indicators for demonstrating and monitoring the performance of the magnetic separator

KPI description	Unit	KPI
Solid removal rate	%	Up to 93
Outlet solid content	mg/L	10 - 15
Ratio sludge / treated volume	%	0.002 – 0.010
Solid content of sludge	wt.-%	19 - 31
Cleaning water demand	L	40

1.3 Market perspective

1.3.1 Market overview - Opportunities and threats of magnetic separator

Main field of application of the magnetic separator is the treatment of cooling water with product contact in the iron and steel industry, which was demonstrated for a cooling water circuit for a hot rolling mill over 6 months in the INSPIREWater project. Typical further cooling water applications are in continuous casting lines and descaling of steel with cooling water at high pressures up to 200 bar, both requiring a low solid content in the feed water. High solid contents cause wear of the nozzles leading to disturbed spraying patterns and further on to inhomogeneous material properties in the microstructure. In the case of descaling, the effects are insufficient cleaning and increased maintenance costs. Further applications in the iron and steel industry are the treatment of gas washing water, cold rolling emulsions and quenching bathes.

Furthermore, the technology can also be included in existing cooling water circuits as a replacement or addition to conventional solid removal treatment techniques for handling increasing solid contents because of increased productions capacities or new processed materials. A further shown application is the concentration of the solid content of sand filter



back flush waters to ensure a reliable sludge dewatering with constant low water contents in centrifuges. Additional applications beside the iron and steel industry are in the treatment of metal working emulsions and oils used by automotive manufacturers and suppliers, plant engineering and construction. A further one is the removal of scale from oil, polymer or salt quenching bathes in the area of surface and heat treatment. A possible threat would be increasing prices for permanent magnet raw materials leading to high sales prices far above the prices of sand filters. The opportunities and threats of the magnetic separator are summarized in the SWOT analysis in Table 1.1.

In EU24, over 500 steel producing sites producing on the average 170 million tons of steel per year exists¹, which are potential places of the application of the technology. Currently, the application of the magnetic separator focuses on the German market, with the aim to extend the applications on the European market. Focus are countries with iron and steel and / or automotive industry with related suppliers as e.g. Spain, Sweden, Italy, The Netherlands, Czech Republic, Austria and Poland.

It is estimated that each sold magnetic separator will have a capacity installed of 50 m³/h, being operated 8'000 h/a, leading to a capacity of 400'000 m³/a per plant. In dependency of the specific local site situation and requirements, it can be assumed e.g. that for a site stream filtration of a 2'000 m³/h water circuit, 4 magnetic separators with a yearly capacity of 1'600'000 m³ are needed. Further, it is assumed that, each Basic Oxygen Furnace or Electric Arc Furnace is connected to a casting line and a hot rolling mill.

In Germany exist about 21 Basic Oxygen Furnace and 42 Electric Arc Furnaces². Considering a cooling water circuit of 1'000 m³/h for casting and hot rolling, the estimated installed capacity in Germany will be about 101 million m³ per year. In EU27 101 Basic Oxygen Furnaces and 231 Electric Arc Furnaces exist meaning the same amount of casting lines and hot rolling mills¹. Using the same assumptions as for Germany, the estimated installed capacity will be about 664'000 m³/h or 5'312 million m³ per year in EU27, shown in Table 1.3.

¹ Eurofer (2018) Steel industry in figures

² Best Available Techniques (BAT) - Reference Document for Iron and Steel Production Industrial Emissions Directive 2010/75/EU (Integrated Pollution Prevention and Control)

Table 1.3 Number of application fields identified in European countries and estimated overall operational capacity per year and country

Country	Number of application fields	Estimated overall operational capacity (m ³ /a)
Germany	63	101'000'000
EU27	332	5'312'000'000

1.3.2 Economical evaluation

As an example, the saving potential for one site with a cooling water circuit of 1'000 m³/h is explained in the following including a comparison of the magnetic separator with continuous and discontinuous sand filters shown in Table 1.4.

Table 1.4 Comparison of the magnetic separator with continuous and discontinuous sand filters

Flow rate [m ³ /h]	1'000		
Separator	Magnetic separator	Sand filter, continuous	Sand filter, discontinuous
Sludge occurring [m ³ /h]	0.6	9 - 100	30 - 50
Operating pressure [bar]	Pressure less	Pressure less	3 - 6
Energy demand [kW/h]	-	-	167
Compressed air [Nm ³ /h]	-	130 – 140 (4 bar)	25 (4 bar)
Maintenance	-	Sieve and pump cleaning, change of sand	Change of sand

Further economic advantages of the magnetic separation, beside potential savings in the field of energy, operational costs for sludge treatment and investment costs in the case of a new installation or replacement are:

- low amount of backwashing water: Magnetic separator 0.6% - Sand filter: 3 - 10% of treated volume flow
- constant outlet solid contents even in the case of factor 3 and more varying inlet solid contents
- low space demand with compact technology
- selective removal of iron for a metallurgical reuse.

For the case of a cooling water circuit of 1'000 m³/h, following savings depending of the specific site situation are possible:



- energy saving by pressure less operation without compressed air requirement of about 20 - 120 k€ /a
- operating cost reduction for sludge dewatering about 50 - 60 k€ (abstinence decanter centrifuge due to minor sludge occurrence) because of lower sludge amounts with higher solid contents in the range of 19 - 31 wt.-% instead of 1 – 4 g/L
- reduced investment costs by abstinence of decanter centrifuge (300 k€ for treatment of a flow rate of 50 m³/h)

Additional savings can be achieved from the improved separation efficiency and the associated increase in the lifetime of nozzles of descaling units or in continuous casting lines, less scaling in pipe-works and reduced maintenance costs.

1.4 Roadmap

1.4.1 Current technology readiness level

BFI started in the INSPIREWater project with the magnetic separator at a TRL of 5, building prototypes with different capacities. BFI's focus in the INSPIREWater project is to get the long-term testing experience with a prototype with a flow rate of 50 m³/h.

Finally, a technology readiness level of 7 – 8 could be reached, because the capacity of the prototype is sufficient for a direct use in smaller cooling water circuits.

1.4.2 Barriers and challenges for implementation

The magnetic separation using strong field permanent magnets for the treatment of cooling water is a new technology in this area. The level of awareness has to be increased on the German and especially in the European market by using different channels as e.g. presentations on conferences, publications in journals or the internet.

Further on, there is a need for the installation of a first magnetic separator in an industrial full-scale application, as reference plant showing its applicability. Because of the novelty of the technology, a tendency to select a “well-known technology” could be possible.

A challenge for the application of the magnetic separator in other branches could be permanent water or better media temperatures above 60°C, which occur in a few special processes as hot degreasing. New magnet materials allowing higher permanent water temperatures are in development but are accompanied by lower magnetic force.



1.4.3 Actions for implementation

Regarding the technical development, the integration of the BFI patented online solid measurement for a tailored magnet cleaning, leading to a longer operational time and saving water for magnet cleaning, is needed. The principle function was demonstrated during the field trials at the cooling water circuit in Spain.

The development of a magnetic separator with a capacity of 200 – 300 m³/h would be a next step for scaled up applications in cooling water treatment.

For further applications in different media as metal working emulsions/oil and degreasing processes, an adaption of the cleaning procedure of the magnets could be necessary for removal of oil and grease.

Regarding the exploitation of the magnetic separator, the technology will be operated for trials at potential customers and especially in the case for potential new applications. In Table 1.5 the business CANVAS, the major elements of the business plan of the magnetic separator are summarized as an additional step to increase exploitation.

The graphical illustration of the roadmap Figure 1.2 gives an overview of the actions for the future implementation of the magnetic separator.

1.5 Conclusion

The magnetic separator was demonstrated as part of the INSPIREWATER project in an ArcelorMittal site in Spain, achieving a technical readiness level of 7.

It allows a chemical free and energy saving solid removal in combination with a minimum of 10 times lower sludge amount connected to lower sludge treatment effort.

The technology can be applied in further applications in the iron and steel industry (treatment of gas washing water, cold rolling emulsions) as in further industries as automotive, plant engineering or surface treatment.

Table 1.5 Business model CANVAS – Magnetic separator

Key partners	Key activities	Value proposition	Customer relationship	Customer segments
<ul style="list-style-type: none"> • Magnet supplier • Equipment supplier • Construction companies • Licensee selling and building of the magnetic separator including after sales services 	<ul style="list-style-type: none"> • Further development <p>Licensee:</p> <ul style="list-style-type: none"> • Commercial activities • Marketing activities • After-sales and customer services • Equipment production • Engineering design <p>Key resources</p> <ul style="list-style-type: none"> • Know-how of magnetic separation/magnet technology 	<ul style="list-style-type: none"> • Energy saving • Chemical free process • Simplifying subsequent processes (e.g. post treatment of sludge) • Selective removal of Fe for metallurgical reuse • Compact and easy useable, full automated technology 	<ul style="list-style-type: none"> • Testing of technology under local specific site conditions • Consultant work for technology comparison and evaluation, realization of application • Research and development for new applications <p>Channels</p> <ul style="list-style-type: none"> • Conferences, fairs, workshops • Internet, YouTube • Being part of water/process water related comities 	<ul style="list-style-type: none"> • Iron and steel industry • Automotive • Plant engineering • Surface treatment
<p>Cost structure (licensee)</p> <ul style="list-style-type: none"> • Equipment purchases • Construction 		<p>Revenue streams</p> <ul style="list-style-type: none"> • Selling the plants (licensee) • Licensing the technology 		

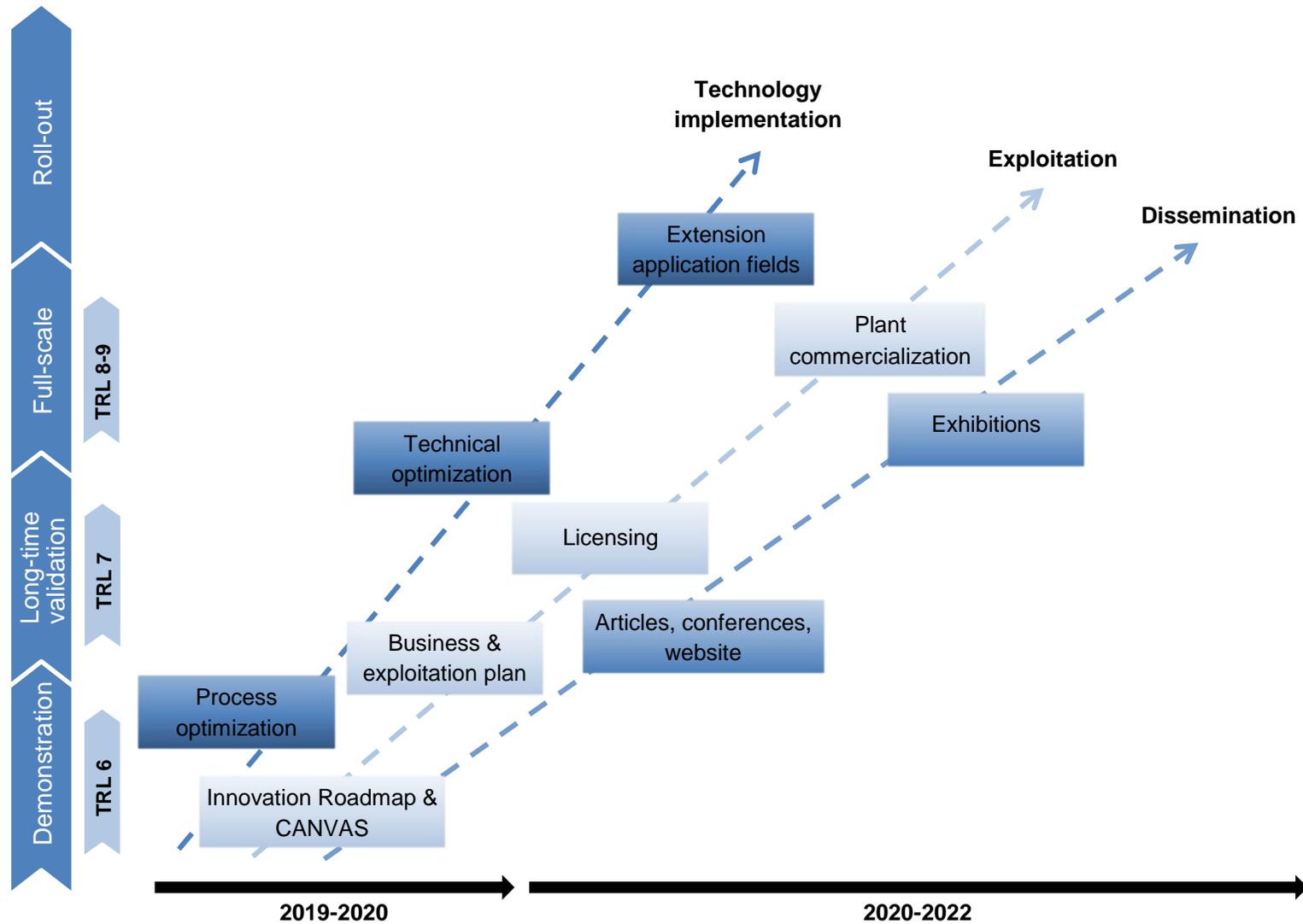


Figure 1.2 Innovation Roadmap of magnetic separator

2. Innovative reactor

2.1 Introduction

For direct and indirect cooling processes, the highest amount of water (75%) is used in the iron and steel industry. Whereby, the solids are mainly removed physically in a wastewater treatment plant. For desalting and softening of the water, many chemicals (antiscalant, corrosion inhibitor and biocide) must be added to the cooling water circuit to reduce the amount of dissolved salts and microorganisms, which have got into the water circuit by passing the cooling tower. Since, dissolved salts cause scaling and corrosion in the cooling water system, leading to higher maintenance effort. ArcelorMittal Rail Mill Gijón currently faces up these kinds of problems, although a conventional chemical treatment is being applied in the circuits to keep their impacts under control. Therefore, a new innovative reactor has been proposed as an alternative solution to decrease the scaling and corrosion potential. Beside the main effects of the innovative reactor TPL-160 (Figure 2.1a) by TRIENXIS performance, a Spanish supplier, against microorganisms and scaling, it is supposed that it promotes precipitation (carbonates and metals precipitates), decreasing water turbidity and collecting more sludge in wastewater treatment plant decanters. The reactor TPL-160 consists of two different parts: electromagnetic coil and electrical panel (230 V and 50 Hz), shown in Figure 2.1b.

a)



b)

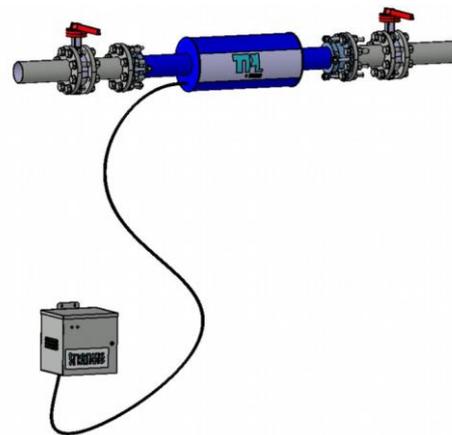


Figure 2.1 Reactor TPL-160 as a) picture and b) scheme (electromagnetic coil in blue and electrical panel in grey)

The operational conditions requested by the supplier are:

Table 2.1 Reactor TPL-160 required operational conditions

Minimum water flow rate, m³/h	800
Maximum water flow rate, m³/h	1'100
Room temperature, °C	-15 to 50.5 °C
Max. fluid temperature, °C	121
Max. fluid pressure, bar	15 (at 22°C, PVC)

The TPL-160 transmits two high-frequency electrical pulses in a medium liquid. The characteristic shape of frequency waves are shown in the following Figure 2.2 representing oscilloscopic measurement of different electrical shocks generated per second.

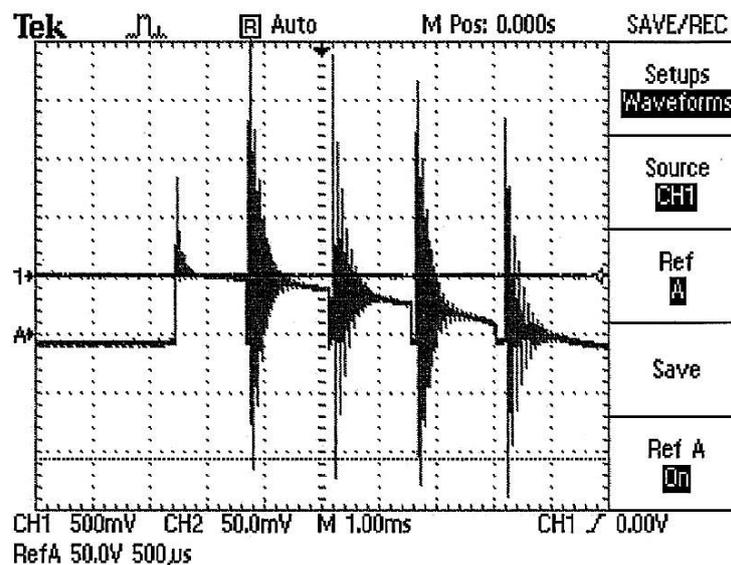


Figure 2.2 Frequency waves diagram

According to the information of the technology supplier, following factors will be affected by treating the water with the TPL-160. The sharp oscillations, 660 pulses per second, generating an average greatly variable, that does not allow the adaptation of living forms (biocide action) and creates in the environment sudden changes in the balance of forces of repulsion. On the other hand, the TPL-160 modifies the form of precipitation of solids passing from scale to a safe, easily filterable powder. The equipment achieves this by "activating" naturally small-suspended particles found in the water. These suspended particles act as nuclei for the

precipitation of dissolved minerals. Furthermore, TPL-160 activates the suspended particles, eliminating the electric charge static surface. If this charge is not eliminated, suspended particles do not precipitate. The powder produced by the TPL-160 (precipitated salts) does not stick and adheres to the equipment surfaces, but it can be easily removed from the tank with filtration or centrifugal separation. Regarding its bactericide effect, the high frequency pulse damages the bacteria membranes, creating small pores. This will break the wall and prevent bacterial reproduction stabilizing development microbial. This bacterial lysis is accomplished by exposure to a field enormously variable that does not allow a biological adaptation of life. The variation of the field electric, 660 times per second, creates enormous environmental stress subjecting chemical compounds that comprise the permeable walls of the bacteria to repeated and increasing alteration. This variability of fields gets affected, as because of its intensity, virtually all types of organic compounds. The breakdown of these compounds achieves lysis of the bacterial walls and high microbial mortality in general.

2.2 Technology perspective

In the INSPIREWater project, an industrial trial has been carried out in Rail Mill Gijón cooling water circuit, in order to test the innovative reactor (TPL-160 by TRIENXIS performance) for the first time at any ArcelorMittal site. The equipment has been implemented in the reheating furnace outlet in the cooling water circuit (Figure 2.3). The objective of this trial was to study the removal /reduction of dissolved salts e.g. sulphates, iron and hardness by electro precipitation, using this new reactor, compared to reverse osmosis, and improve water corrosion rate.

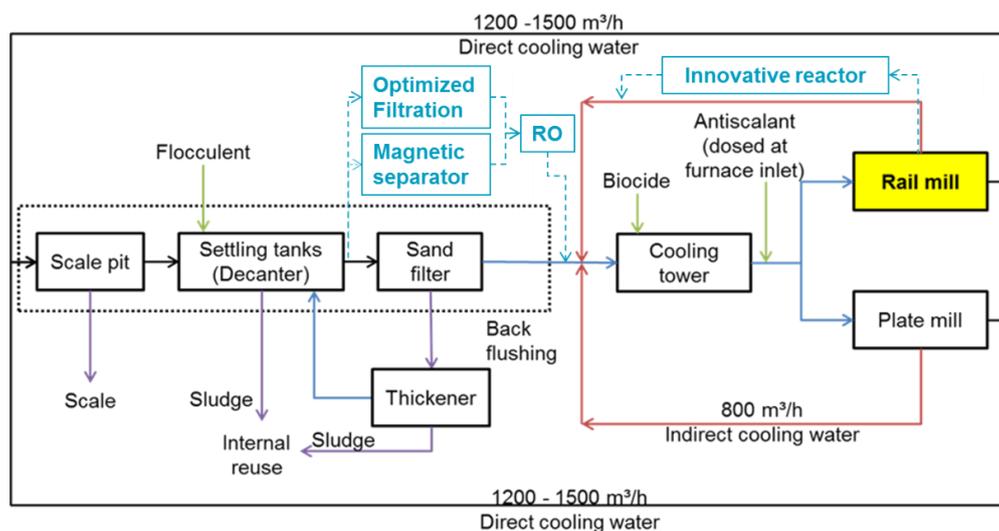


Figure 2.3 Situation of the innovative reactor at plant scale

2.2.1 Strengths and weaknesses of innovative reactor

This innovative technology, that is currently patented, is presented as an alternative to control corrosion and scaling issues in cooling water circuits and it is mainly applied in automotive sector. The devices work within electric pulses (low energy consumption) and they are easy to install in line in the circuits (see further strengths in SWOT analysis Table 2.2).

However, this equipment claims strict operational requirements to assure an optimal performance. Furthermore, as a general decrease of chemical additives is supposed, it can require significant changes in the current chemical treatment, with great reluctance from the sites.

Table 2.2 Overview of SWOT analysis of innovative reactor

INTERNAL	<ul style="list-style-type: none"> • Salts precipitation boosted by the equipment is a non-sticking “dust” • Easy implementation into the existing cooling system & maintenance • Lower chemical additives consumption and biocide • Low energy consumption • Biocide effect • Low risk of technology copy • Distribution company experienced on international market • License agreement with distribution company • Low CAPEX • Patented technology • CE certificate 	<ul style="list-style-type: none"> • Strict operational requirements to assure an optimal performance • It can need significant changes in the current chemical treatment (decrease chemical additives doses with the risk that implies to the installation) • Development based on high experiences in water treatment of the automotive industry
	S	W
EXTERNAL	<ul style="list-style-type: none"> • Water scarcity, increasing water reuse • Huge-market potential within certain industries • Significant reduction biocide consumption, avoiding side-products from oxidant biocides (chloride) • Replacement of conventional oxidants biocides 	<ul style="list-style-type: none"> • Chemical treatment is very extended as the main treatment for the cooling circuits • Reluctance of removing chemicals from cooling water circuits (standard treatment) • Reluctance of process industry to new technologies • Reluctance of the Purchasing Departments to make a contract with small companies (no multinational)
	O	T

2.2.2 Key performance indicators of innovative reactor

Two different trials were carried out with the innovative reactor, each for a duration of three months:

- Blank test, with TPL-160 switched off
- Test, with TPL-160 switched on

During the trials, some measurements were taken (see equipment in Figure 2.4):

- **Water analysis** (on-line): pH and conductivity + once a week: total hardness, phosphates, conductivity, turbidity, calcium hardness, suspended solids, dissolved iron, calcium, chlorides and sulphates). Two points of measurement: inlet and outlet of the reheating furnace.
- **On-line corrosion measurements** (using LPR probe) and **corrosion rack measurement** (two points of measurement with two corrosion coupons each: inlet and outlet of the reheating furnace).
- **Chemical reagents consumptions** (antiscaling, corrosion inhibitor, biocide etc.). Only the chemical additives with direct effect on water quality will be considered.

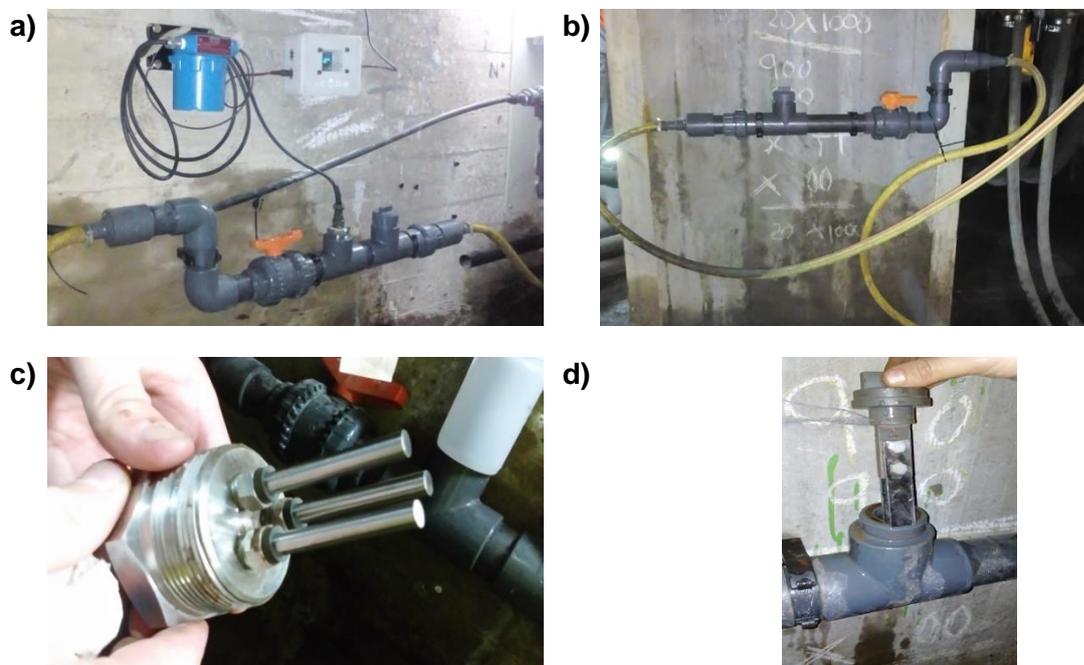


Figure 2.4 Corrosion measurements. a) Reheating furnace inlet: LPR probe and corrosion coupons, b) Reheating furnace outlet. Corrosion coupons, c) LPR probe and d) corrosion coupons

Although the performance of the technology was widely followed-up and monitored during the trial, some water analytical parameters involved in corrosion/scaling issues were chosen as KPIs (Table 2.3):

Table 2.3 Overview of key performance indicators for demonstrating and monitoring the performance of the innovative reactor

KPI description	Unit	KPI
Orthophosphate consumption (corrosion inhibitor)	kg/year	Reduction
Conductivity	μS/cm	20% decrease
Turbidity	NTU	Reduction
Cooling water total hardness	mg/L	20% decrease
Cooling water calcium hardness	mg/L	20% decrease
Cooling water sulfate concentration	mg/L	Reduction
Cooling water chloride concentration	mg/L	Reduction

2.3 Market perspective

2.3.1 Market overview - Opportunities and threats of innovative reactor

This technology is hardly implemented in steel industry, being the main target market the automotive industry, especially as biocide in cooling towers of the cooling circuits. There is no equipment installed at any ArcelorMittal site.

Equipment's supplier comes from Spain, although its reactor is installed in several countries, mainly in automotive industry.

Table 2.4 Operation sites using TPL technology

PSA Group	CNH Group	GESTAMP Group	RENAULT Group	MERCEDES Group
Vigo (SPA)	Madrid (SPA)	Palencia (SPA)	Ayrton Sena (BRA)	Vitoria (SPA)
Poissy (FRA)	Valladolid (SPA)	Vigo (SPA)		
Rennes (FRA)	Suzzara (ITA)			
Kaluga (RUS)				
Shenzeng (CHI)				
Mangualde (POR)				
Aulnay (FRA)				

However, conventional chemical treatments for cooling water systems pose the main competitors to this innovative technology (see SWOT analyses Table 2.2). Furthermore, there



is an important reluctance from process industry to new technologies, even, removing standard chemical treatments because of the risk of corrosion/scaling issues and the resulting plant/production problems.

2.3.2 Economical evaluation

One of the main advantages of this technology is that the equipment is offered by the supplier in a rental system, avoiding a huge initial investment by the sites (no CAPEX required). The rental price will depend mainly on the water flow rate (and the equipment model required to treat this water flow rate) to be treated and other installation details, and for this reason, every case must be studied. On the other hand, the associated cost savings due to the decrease in chemical reagents consumption cannot be predicted in advance and it depends on each case specifically, e.g. the water flow rate to be treated and current water chemical treatment program, etc.

2.4 Roadmap

2.4.1 Current technology readiness level

TPL technology is currently patented by:

- U.S. Patent 6,063,267 and 6,641,739
- Canadian Patent 2335496
- Japan patent 3595505

According to EU definitions, TPL technology readiness level is TRL 9: actual system proven in operational environment.

2.4.2 Barriers and challenges for implementation

The main challenge that TPL equipment can find in industrial environment is that is an innovative technology, implemented mainly in automotive sector and less known in other types of industries. For this reason, some reluctance from the companies must be initially overcome, considering furthermore, that the implementation of this technology translates into a significant change in the current conventional water treatments (using chemical additives).

When a TPL equipment is implemented in a cooling water circuit, some requirements must be considered:

- Coil-pipe assembly: on the same pipes of the cooling water circuit (flanges/supports to maintain the additional weight). Revision of the pumping system (adequate flow/volume)



- Electric Panel: on a flat and dry surface and close to the TPL at a suitable height for handling workers
- TPL pipe must always be completely filled with the fluid to be treated
- TPL must precede any filtration system or element that may influence the water inlet flow to the TPL (i.e. pumps, elbows, etc.): water flow into the equipment must be linear
- Take care of the water flow direction, considering the arrows symbols on the equipment
- The equipment must be switched off if the pipe is empty of water

2.4.3 Actions for implementation

Although TRL 9 is already achieved, only automotive industry is using this technology. For this reason, more commercial effort and marketing strategies should be put in dissemination of the results to make this technology more known in other industrial sectors. To increase the company sales force, e-mailing campaigns, attending industrial exhibitions and fairs, advertisements and information dissemination in specific magazines and websites etc. will be further extended and updated by new marketing approaches.

As a first step, following tools were developed to better understand the business strategy behind the technology and the needed actions for a successful market expansion:

- Business model CANVAS (Table 2.5)
- Roadmap presentation of actions in the form of graphical illustration, taking into account the current state of the technology: TRL 9 (Figure 2.5)

2.5 Conclusion

TPL equipment is based on an innovative technology for cooling water treatment that is able to achieve three main effects:

- Stabilization of microbiological growth
- Biofilm removal
- Improving and maintaining physic-chemical quality of the water

This technology is already completely developed, even patented, and, it is, nowadays, well-known and implemented mainly in automotive companies as biocide (main effect). However, other industries show reluctance to change their conventional water treatments, based on chemical reagents addition, to this innovative treatment, based on electrochemical issues. For this reason, more technical information e.g. commercial activities about this technology and successful industrial cases should be disclosure and spread in different industrial fields with the aim of becoming a more known technology that can be an alternative to the conventional cooling water treatments.

Table 2.5 Business model CANVAS–Innovative reactor

Key partners	Key activities	Value proposition	Customer relationship	Customer segments
<ul style="list-style-type: none"> • Chemical products manufacturers or suppliers 	<ul style="list-style-type: none"> • Commercial activities • Marketing activities • After-sales and Customer services • Engineering design • Equipment production 	<ul style="list-style-type: none"> • New water treatment technology with two main effects decreasing corrosion: <ul style="list-style-type: none"> - Biocide reduction(decrease in chemical biocide reagents consumption) - Salts precipitation reduction 	<ul style="list-style-type: none"> • Technical commercial visits • After-sales technical support 	<ul style="list-style-type: none"> • Industrial sites with cooling water circuits that include cooling towers
	<p>Key resources</p> <ul style="list-style-type: none"> • Set of pieces to build up the equipment • Technical staff • Operating license • Insurances • Suppliers 	<ul style="list-style-type: none"> • Equipment easy to implement in the current facilities • Easy maintenance • Low energy consumption 	<p>Channels</p> <ul style="list-style-type: none"> • Phone calls • E-mailing • Professional fairs (water treatment sector) 	
Cost structure		Revenue Streams		
<ul style="list-style-type: none"> • Infrastructures cost • Personnel cost • Spare parts to build up equipment 		<ul style="list-style-type: none"> • Equipment rental and technical support • After-sales service and maintenance service • Sale of chemical products 		

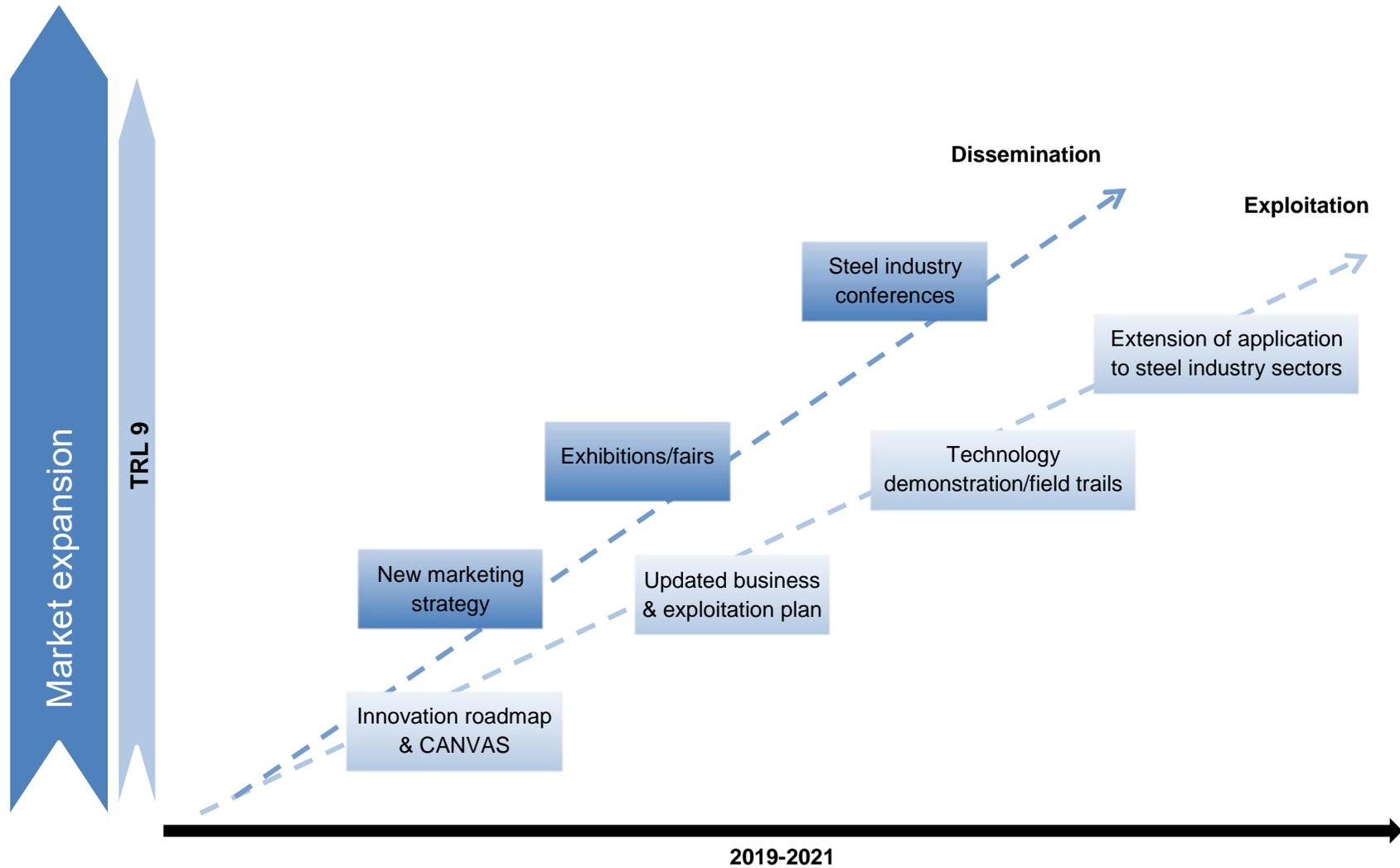


Figure 2.5 Innovation Roadmap of innovative reactor

3. Layer-by-Layer modified acid resistant nanofiltration membranes

3.1 Introduction

The recovery of resources from waste streams is getting hugely important striving for circular economies. Several industry branches are even aiming increasingly at zero-liquid-discharge. Nanofiltration (NF) is a well-known technique for separation and has recently become attractive for resource recovery applications. For instance, studies have shown that NF is a suitable technology for phosphorus (P) recovery from acidic disintegrated sewage sludge. P is a limited resource of global importance with natural deposits that are restricted to a few countries. NF can enable P recovery from waste streams by allowing the P to permeate through the membrane and retaining multivalent positively charged impurities^{1 2 3}. The general process scheme is shown in Figure 3.1.

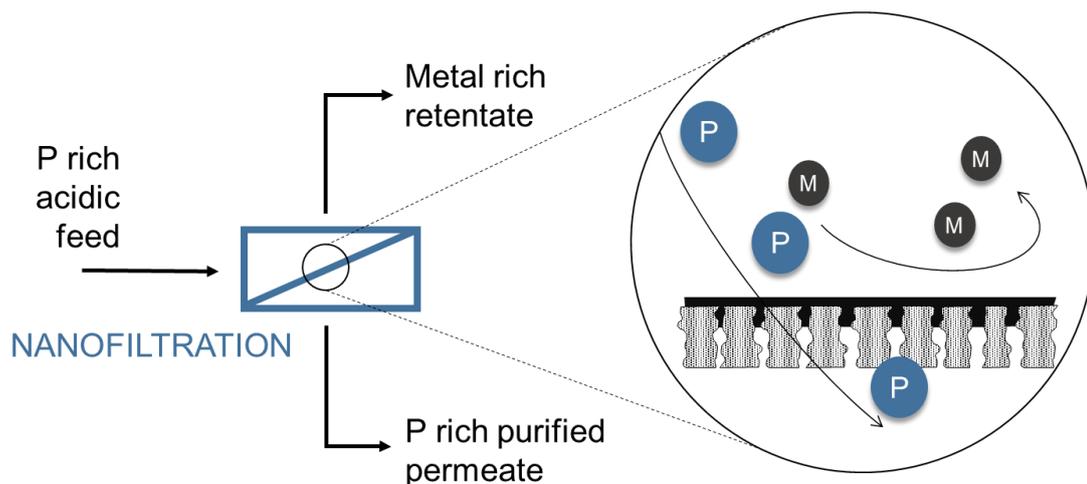


Figure 3.1 General process scheme of P recovery from any acidic waste stream applying NF

In the INSPIREWater project, the application of NF is expanded to the recovery of spent pickling phosphoric acid, which is characterized by economic importance. Spent pickling acid is a highly challenging waste stream in the process industry. Currently, most of the spent acid is neutralized without any recovery step. By applying NF, the acid will directly be reused in the pickling process. During piloting of the Sandvik case study, the conventional NF membranes are successfully applied to recover spent acid, however increasing the efficiency of the process

¹ Niewersch (2014) Nanofiltration for Phosphorus Recycling from Sewage Sludge

² Schütte et al. (2015) Phosphorus Recovery from Sewage Sludge by Nanofiltration in Diafiltration Mode

³ Niewersch et al. (2014) Nanofiltration for the Recovery of Phosphorus - Development of a Mass Transport Model

in terms of P-yield and flux is still a crucial aspect. In previous studies, it was demonstrated that the application of layer-by-layer (LbL) modified membranes could enhance the process performance of P recovery from sewage sludge ash in terms of P yield and energy consumption⁴⁵.

LbL membranes are fabricated by depositing oppositely charged polyelectrolytes (PE) on a charged support material, in general a porous UF membrane (Figure 3.2). Sodium chloride acts as a counter ion for the PE and influences the properties of the membrane. The LbL technique has the potential to be a suitable solution for overcoming the above-mentioned drawbacks (low flux, low P-yield) of commercially available membranes^{6 7}. In addition, LbL assembly gives the membrane tailored properties such as charge, hydrophilicity, chemical resistance, high flux, resistance to fouling and therefore an overall improvement of the separation process. LbL is a simple method to prepare polyelectrolyte multilayers in different variations. Oppositely charged polyelectrolytes can be used to form multilayers on any charged substrate including charged porous ultrafiltration membranes⁸.

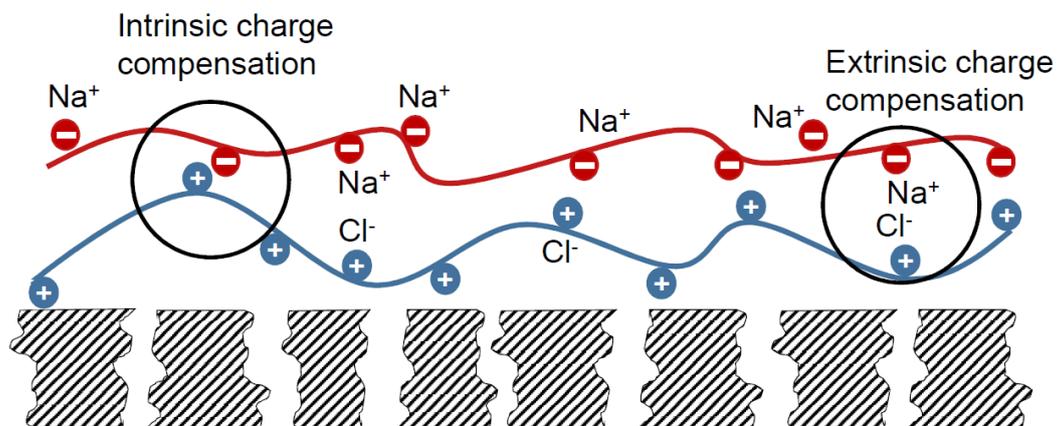


Figure 3.2 Deposition of a polycation and polyanion on a porous ultrafiltration membrane, indicating intrinsic and extrinsic charge compensation

⁴ Remmen et al. (2019) Phosphorus Recovery in an Acidic Environment Using Layer-by-Layer Modified Membranes

⁵ Paltrinieri et al. (2019) Improved Phosphoric Acid Recovery from Sewage Sludge Ash Using Layer-by-Layer Modified Membranes

⁶ Laakso, Pihlajamäki, and Mänttari (2018) Effect of Polycation Structure on the Fabrication of Polyelectrolyte Multilayer Hollow Fiber Membranes for Loose Nanofiltration Applications

⁷ Joseph et al. (2014) Layer-by-Layer Preparation of Polyelectrolyte Multilayer Membranes for Separation

⁸ Decher (1997) Fuzzy Nanoassemblies: Toward Layered Polymeric Multicomposites

3.2 Technology perspective

In the INSPIREWater case study, we demonstrated that LbL membranes used for nanofiltration could be applied to enhance the P recovery process from spent pickling acid in Sandviken. Therefore, two types of LbL membranes were prepared and compared to the conventional NF membranes tested in the case study. The coating conditions can be found in Table 3.1. The UF hollow fiber membrane modification with PE was realized through the dynamic coating method⁹. A dead-end filtration set-up was used at a constant pressure of 3 bar. The PE were deposited inside the lumen of the membrane and followed by a rinsing step with demineralized water (DMW) at pH 5 - 6. UF membranes of PES are negatively charged. For that reason, the multilayer deposition was always achieved by starting with the positively charged PE, followed by consecutively alternating adsorption of polyanions and polycations, until a certain amount of bi-layers were reached. Each coating cycle comprises the retention in the lumen and thus on the coating of the membrane inner surface by a certain amount of PE, considering that the pore size of the support membrane used was smaller than the hydrodynamic radius and molecular weight (MW) of the applied PE. After each cycle (i.e., the deposition of one PE), the membrane was flushed with deionized water until a conductivity of less than 3 $\mu\text{S}/\text{cm}$ was reached to ensure that the excess of PE solution was completely washed out.

For the LbL membrane coating, the positively charged poly(diallyldimethylammonium chloride) (PDADMAC, MW = 400 - 500kDa, 20 wt% in water) and poly(allylamine hydrochloride) (PAH, MW = 50 kDa, 10 wt% in water) and the negatively charged poly(sodiumstyrenesulfonate) (PSS, MW = 1'000 kDa, 25 wt% in water) were used.

Table 3.1 Coating parameters for the tested LbL membranes

Membrane	Support layer	Membrane area [m^2]	Positive PE	Negative PE	C_{PE} [g/L]	C_{NaCl} [g/L]	Bi-layers
(PAH/PSS) ₃	PES	0.18	PAH	PSS	0.1	0.5	3
(PDADMAC/PSS) ₆	PES	0.18	PDADMAC	PSS	1	0.5	6

The LbL membrane was included in the pilot plant as indicated in Figure 3.3. The NF short-term operation, using the microfiltration permeate as a feed, was conducted with a pressure of 7 bar, temperature at 30 °C and without permeate recovery steps. The flowrate was adjusted

⁹ Remmen et al. (2019) Phosphorus Recovery in an Acidic Environment Using Layer-by-Layer Modified Membranes

to achieve a Reynolds number above 2'300, hence operating in a turbulent mode. The composition of the feed can be found in Table 3.2.

Table 3.2 Average composition of the NF feed solution

Cr	Fe	Ni	S	P
[g/L]	[g/L]	[g/L]	[g/L]	[g/L]
8.61±0.69	23.98±1.67	10.16±0.76	44.63±3.66	97.24±7.41

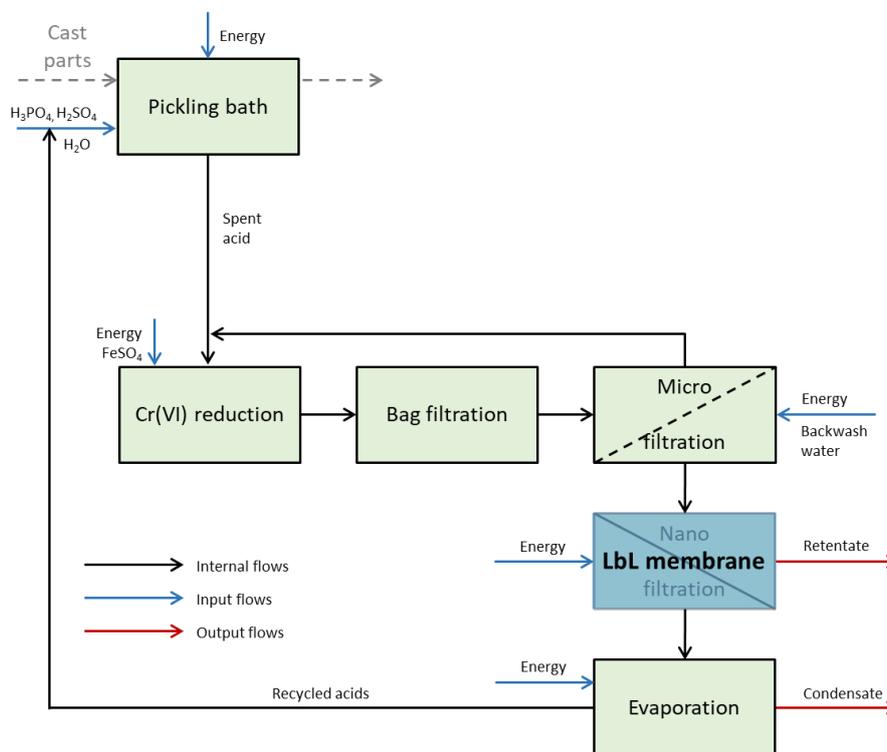


Figure 3.3 Proposed flowchart using LbL membrane during the nanofiltration step

In addition, the LbL membranes are tested as an alternative to the microfiltration (MF) as a pre-treatment for particle removal before the NF. A feasibility study is conducted to identify the potential of combining the particle removal step and a first ion separation step by applying LbL membranes.

3.2.1 Strengths and weaknesses of acid resistant LbL membranes

The unique selling proposition of LbL membranes for acidic filtration, compared to conventional NF membranes, is the high flux, which leads to a higher flexibility of the design of any filtration process (Table 3.3). In addition, operation cost can be saved due to applying a lower pressure and therefore lower energy demand, as well as the investment cost due to smaller units needed in the process configuration. Another strength focusing on P recovery are the lower P

retentions, which could result in higher P yields depending on the process configuration by using LbL membranes.

The major problem for establishing LbL membranes on the market is the high sensitivity towards the osmotic strength of the solution, which leads to changes of the physical properties of the PE layers and thereby also to lower metal retentions negatively affecting product quality. To provide better predictions of the LBL membrane performance more effort has to be put into the research of the transport mechanism through the modified membrane. Moreover, the current labor-intensive coating procedure has to be improved towards an economic viable membrane production.

Table 3.3 Overview of SWOT analysis of acid resistant LBL membrane

INTERNAL	<ul style="list-style-type: none"> • High fluxes • High process flexibility • Lower operation & investment cost • Higher product yield 	<ul style="list-style-type: none"> • Sensitive to high osmotic load in the solution • Lower retention values for multivalent ions • Insufficient knowledge of membrane transport mechanism • Labor-intensive coating method
	S	W
EXTERNAL	<ul style="list-style-type: none"> • First tailor-made membrane on the market for acidic application • Great application potential for NF for separation tasks with currently low fluxes • Increase of process sustainability 	<ul style="list-style-type: none"> • Difficult to patent because of various membrane modification options • Risk of technology copy • Many competitors on the membrane filtration market
	O	T

3.2.2 Key performance indicators of acid resistant LbL membranes

LbL membranes clearly outperform commercially available membranes due to much higher fluxes, allowing higher concentration rates, leading to less concentrate and lower operation cost (Table 3.4). Additionally, in the tests for the Sandvik case study it could be shown that the LBL membrane has an acid resistance for a pH below 1. Whereby, the most commercially available membrane are limited to pH 2. Moreover, the better P permeability of the membranes could lead to a higher P recovery and further cost savings. So, the innovation potential of LbL

membranes, leading to an innovative ability, which is necessary to successfully establish LbL membranes on the market in long term, is already proven.

Table 3.4 Overview of key performance indicators for demonstrating and monitoring the performance of LBL membranes compared to commercially available and applied NF membranes at the Sandvik case study

KPI description	Unit	LBL	Duracid
Permeability	LMH/bar	2	0.1
Pressure	bar	10	40
Energy consumption	kWh/m ³ feed	1.7	2.4

3.3 Market perspective

3.3.1 Market overview - Opportunities and threats

In the INSPIREWater project, the market for spent acid recycling, directly at industrial site (closed loop), was in focus. For this market, industries using acid as process material, mainly for surface modification, are particularly relevant. A summary of this market can be found in Table 3.5.

Table 3.5 Number of application fields identified in European countries and estimated overall operational capacity per year and country similar to the Sandvik case study, using phosphoric acid and/or sulfuric acid¹⁰

Country	Number of application fields	Estimated overall operational capacity (m ³ /a)
Europe	15-30	4'500 - 9'000
World	50-100	15'000 -30'000
Total	65-130	19'500-39'000

Within the EU, Germany produces the highest amount of spent acid. The share is almost one third of the total amount of spent acid. Therefore, the German spent acid market is targeted first.

The largest part of the spent acid discharged in Germany is originating from the inorganic chemical industry. In 2017, 239'300 t of spent acids were discharged in German disposal facilities. With 214'100 t, sulfuric acid plays the major part. This is 89.5% of all spent acids originating from the inorganic chemical industry. The second highest amount of spent acid in

¹⁰ INSPIREWater Project Proposal (2017)



Germany is produced during metal surface treatment. 186'200 t of acid were disposed in 2017 by this industry¹¹. Most of these acids are pickling solutions. The composition of the mixture depends on the surface treated.

The main driver of all industries, which can be used to successfully launch a product on the market, is the decrease of production cost. Table 3.6 summarizes the drivers of the relevant industries and describes the unique selling proposition of acid resistant LbL membranes for closed loop acid recycling at industrial sites. A major opportunity of the tailor-made LbL membranes emerging on the market is the possibility of enabling many different separation tasks under acidic conditions, which require a high flux for creating a more sustainable process in the industries. However, the high competition on the membrane filtration market as well as the difficulty in patent registration of LbL membranes due to the various membrane modification options makes market uptake still challenging. Nevertheless, the successful implementation of the closed loop process for spent acid recovery will open the market for similar applications in the process industry and other similar processes using high concentrated acids (mainly steel industry and manufacturing industry, but also in chemical industry). Therefore, a full-scale realization could constitute a breakthrough for spent acid recovery in large amounts.

Table 3.6 Drivers for the relevant industries and unique selling proposition of acid resistant LbL membranes for closed loop acid recycling at industrial sites

Driver	Unique selling proposition of LbL membranes
Saving of raw material (financial)	Economical recycling of acids (higher flux, higher yields)
Decrease disposal (financial)	Economical reduction of acid waste (higher concentration rates)
Less impact on the environment (social)	Lower impact on the environment during production and during recovery process

3.3.2 Economical evaluation

An approximate economical evaluation was carried out by Remmen¹² to identify the impact of LbL membranes on operational and capital cost. Investment cost were calculated according to Samhaber¹³. The author has used cost data of implemented full-scale units to create an approximate model for investment cost depending on membrane area. In Table 3.7, all assumptions made for the cost analyses are given for a LbL membrane and commercially available membrane obtained from the pilot site in Sandviken.

¹¹ Statistisches Bundesamt Deutschland (2019) Umwelt Abfallentsorgung

¹² Remmen (2019) Layer-by-layer modification of membranes for resource recovery (in preparation)

¹³ Samhaber & Kepler (2005) Erfahrungen und Anwendungspotential der Nanofiltration

Table 3.7 Assumptions to assess the life cycle costing for P recovery from spent pickling acid

	Duracid	PES(PDADMAC/PSS) ₆
Permeability [LMH/bar]	0.2	2
Module size [m ²]	25	60
Permeate recovery [%]	70	80
Price [Euro/m ²]	250	100
Electricity price [Euro/kWh]	0.15	0.15
Plant depreciation [a]	10	10
Membrane replacement [a]	2	2

Next to CAPEX for the unit, membrane replacement and energy cost were calculated. Despite the influence on the viability of a P recovery process, assumptions about revenue are not taken into account^{14 15}. In this scenario both membranes achieve 50% P recovery, hence the savings due to less H₃PO₄ consumption is equal for both membranes. Due to the lower metal retention, the feed volume for LbL membrane is higher compared to the Duracid membrane. Resulting in higher energy consumption for the pre- and post-treatment for the NF process step, which is by an approximate factor of two higher for the LbL membrane. However, the focus in this roadmap is on the NF process, hence in Figure 3.4 the costs per m³ treated feed for the LbL membrane and the commercial membrane as a function of TMP are summarized only for the NF step. The TMP ranges were chosen to show the best possible performance of the membrane as well as taking pressure limits of the membrane modules into account. The overall cost, when aiming at 50% P recovery, is two to almost three times lower for the LbL membrane compared to the conventional spiral-wound module. With increasing pressure, the overall process cost decreases for both membrane types as the unit size decreases with increasing pressure. The main cost for both membranes occur from investment cost for the membrane unit. These are even higher per m³ treated feed, as the Duracid membranes are treating less feed, which results in higher unit costs per m² membrane area according to the equation proposed by Samhaber¹⁶. Energy cost are considerably lower for the LbL membrane due to lower applied pressure. Another important factor is the high packing density of hollow-fiber membranes, leading to a reduced amount of modules¹⁷. For this specific case, it can be

¹⁴ Nättorp, Remmen, and Remy (2018) Cost Assessment of Different Routes for Phosphorus Recovery from Wastewater Using Data from Pilot and Production Plants

¹⁵ Hukari, Hermann, and Nättorp (2016) From Wastewater to Fertilisers - Technical Overview and Critical Review of European Legislation Governing Phosphorus Recycling

¹⁶ Samhaber & Kepler (2005) Erfahrungen und Anwendungspotential der Nanofiltration

¹⁷ Frank et al. (2001) Capillary Hollow Fiber Nanofiltration Membranes

concluded that PES(PDADAMAC/PSS)₆ can decrease the annual cost for OPEX and CAPEX to over 50% compared to a Duracid membrane.

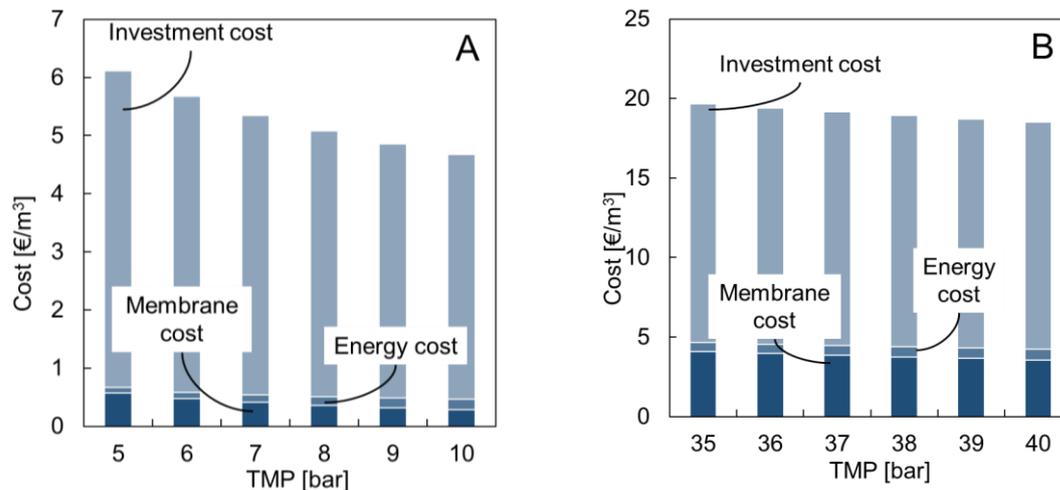


Figure 3.4 Costs per treated feed flow in €/m³ as a function of TMP for A) a LbL membrane and B) a commercial membrane¹⁸

The capacity of the unit calculated for Sandviken is small (<10'000 m³) and therefore might not be representative for many NF applications in an acidic environment. To give an outlook on the economic viability of other unit sizes, the costs per treated feed flow as a function of TMP is depicted in Figure 3.5. Three membrane unit sizes are chosen to describe a broad range. This approach will help to find an economic optimum for process employing LbL membrane depending on TMP. In this case, the assumption was made that LbL membranes can be operated up to 40 bar as well as that the same amount of feed per year is treated for both, the LbL and conventional TFC membrane. If the assumptions, made for the LbL membrane, are technically feasible still needs to be proven. The membrane replacement cost are below 10% of the total cost per treated feed volume. Therefore, the influence on the lifetime is minor compared to e.g. the investment cost. For larger membrane units the most economic operation point can be identified. For a unit with a feed flow of 1'000'000 m³ per year, an optimum can be found at a TMP of 14 bar. Here the estimated cost for treatment of 1 m³ feed solution summarizes to 0.55 Euro per m³. When operating a membrane plant with a capacity of 100'000 m³/a the lowest cost per m³ can be achieved at 17 bar. The increase in energy costs as a result of the higher TMP is leading to the higher overall cost, as the investment and membrane cost decrease due to the smaller membrane unit.

¹⁸ Remmen (2019) Layer-by-layer modification of membranes for resource recovery (in preparation)

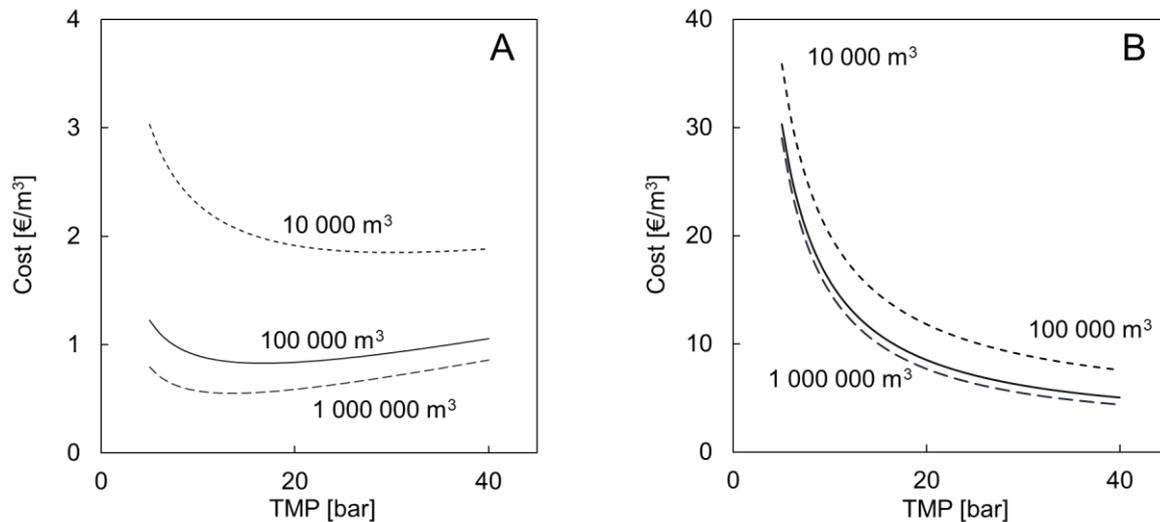


Figure 3.5 Costs per treated feed flow in €/m³ as a function of TMP for A) a LbL membrane and B) a Duracid membrane depending on the membrane unit capacity per year (Remmen 2019)

To conclude, LbL modules, which can withstand pressures up to 20 bar, might be the most economical solution for larger membrane units. Hence, the next technical development should aim at this pressure range. The total cost for the Duracid membrane decreases with increasing pressure. A minimum is not reached within the studied range for all three unit sizes. However, the lowest value is 4.41 € per m³ for 1'000'000 m³ at 40 bar.

3.4 Roadmap

3.4.1 Current technology readiness level

The concept and lab-scale tests have been carried out at the University of Applied Science of Northwestern Switzerland (FHNW) supported by internal funding of FHNW and Pentair. Market analysis and piloting for the case study of P recovery has been conducted during the H2020 project INSPIREWater and a Bridge Proof-of-Concept project funded by the Swiss National Science Foundation. These projects enabled to achieve the first steps towards TRL 6 for acid resistant LbL membranes for spent pickling acid recovery.

3.4.2 Barriers and challenges for implementation

The main challenge for the implementation of LbL membranes on the market will be identifying potential partners, which are able and willing to include LbL membranes in their product portfolio. In this case, two major collaborators will play a role, on the one hand an Original Equipment Manufacturer (OEM) and on the other hand a membrane manufacturer. Gaining the acceptance on a traditional market, such as the process and steel industry, can only be



achieved if well established companies introduce the product (LbL membranes) on the market. In addition, existing infrastructure might be adjusted to the newly designed hollow fiber membrane modules. This hurdle as well can be taken by addressing the whole value chain from membrane producer to the end user during the design of the first full scale application. Furthermore, additional research needs to be carried out to provide more facts for the aimed application. In most cases, a successful pilot study will convince user to invest in the new technology.

In summary, the most important part is to address the whole value chain in cooperation with motivated and innovative companies.

3.4.3 Actions for implementation

For implementation of acid resistant LBL membranes on the market, three major steps need to be carried out (see overview of all actions needed in Figure 3.6):

1. Product development (module & plant design)
2. Clear market positioning
3. Full-scale testing

During earlier stages of the development process, requirements which acid resistant LbL membrane modules have to meet were identified, now during the product development the “what” requirement will be turned into “how” the requirements can be specifically be met. The final product will be designed. The R&D departments of all involved partners (research institute, membrane manufacturer, Original Equipment Manufacturer (OEM)) will develop and test one or more physical versions of the product concept.

Positioning of the product is a very important step during the product development process. The focus of this action step is on understanding the targeted end user and how to communicate the benefits of acid resistant LBL membrane modules in a compelling way.

The following criteria need to be evaluated to allow a successful positioning:

- **Drivers** – Industrial driven or change in legislation
- **Size** - Large enough segment to justify the development and marketing cost.
- **Homogeneous** - Similar end user
- **Heterogeneous** - Differences of the end user to other segments
- **Substantial** - The projected profits must exceed the costs for development
- **Accessible** - The segment must be accessible to marketing and distribution



The final step before the launch into the market is the full-scale testing of the acid resistant membrane modules on site of an end user industry. After the successful demonstration, the launch of the first acid resistant LbL membrane module will be executed. As part of the business plan, a business model CANVAS was developed, presented in Table 3.8 as a next step for the technology exploitation.

Currently, a group of researchers at the RWTH Aachen University is working on applying artificial neural networks to predict LbL membrane performance for model solutions in neutral pH¹⁹. Transferring this knowledge to acidic environments could enable individually fine-tuning of many novel NF applications as LbL membranes for various applications.

3.5 Conclusion

In the INSPIREWater project, the short-term demonstration of acid resistant LbL membranes for spent acid recovery was conducted on the SANDVIK site in Sweden, realizing the first steps towards TRL 6. The higher fluxes of the LbL membranes clearly outperform commercially available NF membranes with allowing higher concentration rates, which will lead to lower disposal and operation cost if the assumptions made for long time operations are correct. In addition, the implementation of LbL membranes in NF processes enables a reduction of investment costs due to lower membrane production cost and lower number of modules. For a successful market uptake of acid resistant LbL membranes, the product development, clear market positioning and long-term full-scale testing have to be implemented as the next major action steps.

¹⁹ Rall et al. (2018) Rational Design of Ion Separation Membranes

Table 3.8 Business model CANVAS – Acid resistant LbL membrane

<p>Key partners</p> <ul style="list-style-type: none"> • Membrane manufactures • Original Equipment Manufacturer (OEM) 	<p>Key activities</p> <ul style="list-style-type: none"> • Engineering processes • Long-term pilot studies • Process optimization • Identification of new markets • Membrane optimization 	<p>Value proposition</p> <ul style="list-style-type: none"> • Acid resistant nanofiltration membrane (LbL) • Customized solutions • Know-How • First on the market 	<p>Customer relationship</p> <p>Customer expect</p> <ul style="list-style-type: none"> • Membrane optimization • Competent consulting • Reliability 	<p>Customer segments</p> <ul style="list-style-type: none"> • Process owner • Metal industry • Recycling industry • Mining industry • Waste water treatment plant operator
<p>Key resources</p> <ul style="list-style-type: none"> • Personal • Membranes and polyelectrolytes 			<p>Channels</p> <ul style="list-style-type: none"> • Conferences and exhibition • FHNW network • EU projects 	
<p>Cost structure</p> <ul style="list-style-type: none"> • Personal costs • Material costs • Energy cost • Infrastructure of production 		<p>Revenue Streams</p> <ul style="list-style-type: none"> • Consulting in optimization and expertise • Membrane optimization • Pilot tests • Selling of membrane 		

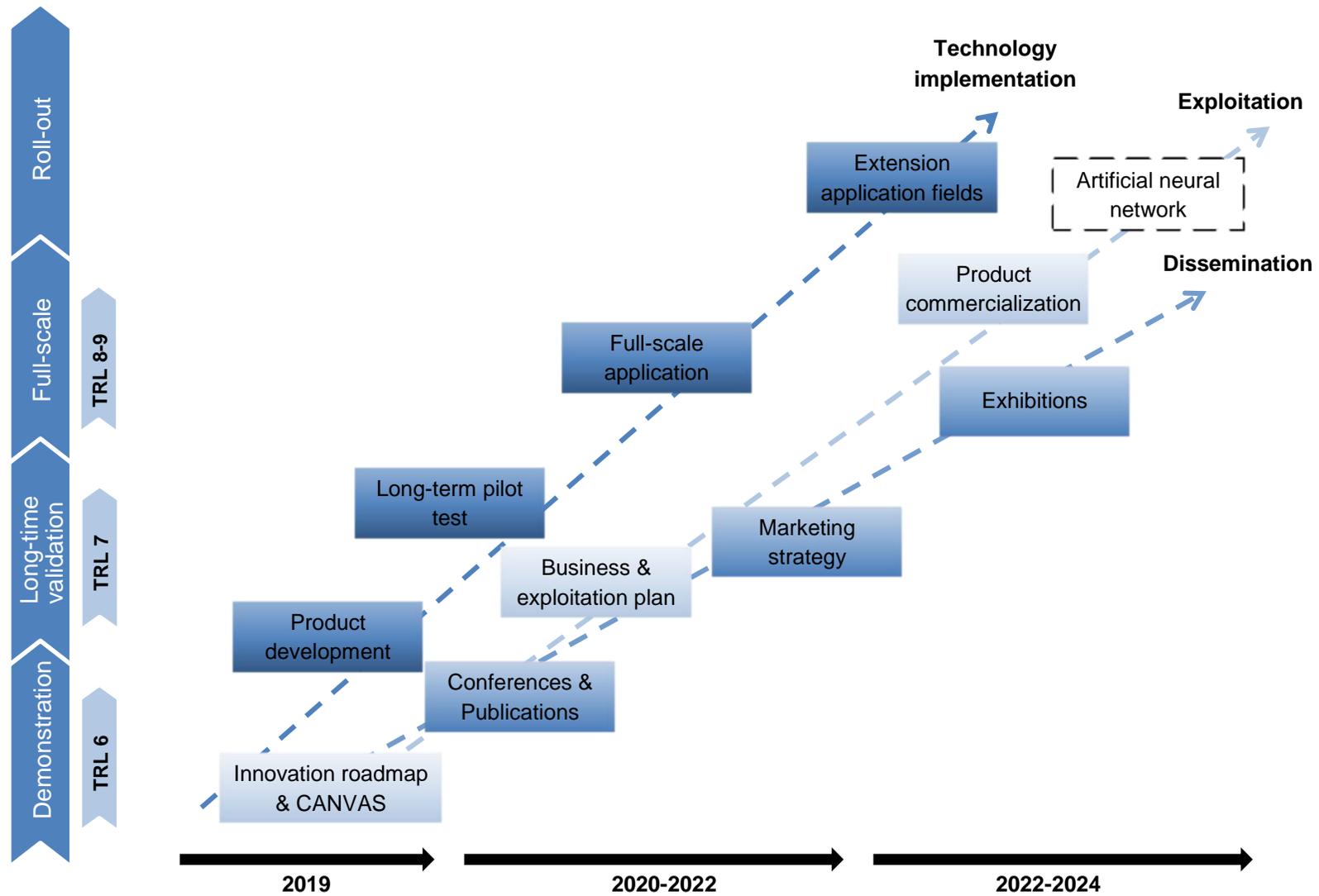


Figure 3.6 Innovation Roadmap of acid resistant LbL membrane



4. Catalytic Water Treatment – MOL[®]LIK Technology

4.1 Introduction

Since the first description of technical membrane processes, about 2'000 years ago, it has been taken a long time until membrane technologies were growing from smaller applications to large industrial processes^{1 2}. Today, membrane systems are used at various places for saving water as well as providing high quality water in challenging areas. Since suitable water quality is required nearly everywhere, the economic importance of membrane systems is growing from year to year^{3 4}.

The present membrane plants – can provide high-quality water in an economical manner – with a significantly reduced energy consumption compared to the classic evaporation. On the other hand, at membranes, pressures up to 80 bar affect water structures and lead to the formation of deposits, which highly increase the operation and maintenance cost⁵. The use of suitable heterogeneous catalysts can suppress negative effects on water structures and extend cleaning intervals of the membranes at the same time. As a result, the emitted chemicals concentration on the concentrate is reduced in comparison to conventional operation while minimizing undesirable side effects on animal and plant life.

MOL[®]LIK – a technical proven catalytic water treatment technology⁶ – is a biocide-free technology that reduces the risk of fouling, scaling and corrosion on surfaces, even at long distances. With this technology, there are entire cooling systems of power plants operated completely free of any biocide – with less than 2 kg of the special catalyst⁷. These catalysts are speeding up solubility by faster supply of molecular water. H₂O_{molecular} is required for preparing the hydration shells. As faster, these shells are prepared as better flux on filter units and as better solubility of substances. After showing good results on areas with quite stable water quality, like cooling and process water treatment, catalytic water treatment was tested in challenging areas of industrial wastewater. Within the INSPIREWATER project, the scope was

¹ Lee (1951) Aristotle – Meteorologica – with English Translation

² Chauhan et al. (2015) Purification of Drinking Water with Application of Natural Extracts

³ Bates et al. (2008) Climate Change and Water. Technical Paper of the Intergovernmental Panel on Climate Change

⁴ Global Water Intelligence IDA (2017) IDA Desalination Yearbook - Water Desalination Report

⁵ Koppe (2018) Limitations in water supply – strategies for solving a global issue, International MerWaterDays

⁶ Koppe & Battagello (2017) Water structures & membrane systems – rising performance with catalytic water treatment; Merseburg University of Applied Sciences

⁷ Koppe et al. (2016) Maximale Performance durch Wasser ohne Chemie – Energieeffiziente Wasserbehandlung

investigating the potential for improving membrane performance with catalytic water treatment in the field of effluent water reuse.

The core element of the MOL[®]LIK-technology is a proprietary ultra-thin metall catalyst foil, made of nickel, chromium and iron. By the special activation process, the metal foil is prepared as follows: An approximately 50 nanometer thick mineral layer is formed – with a clear demarcation to the metal structure. As a result, areas with excess electron and electron deficiency are formed within the mineral structure (see Figure 4.1).

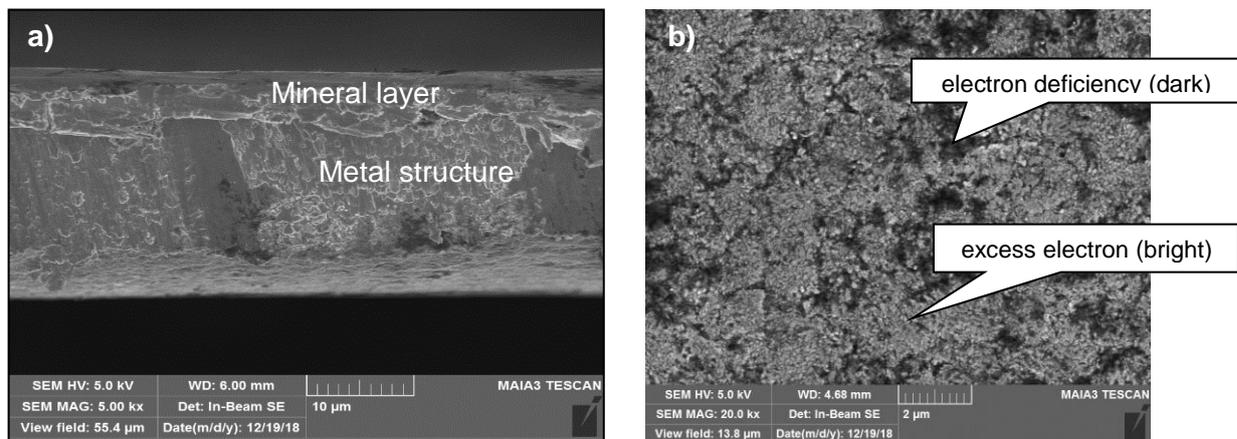


Figure 4.1 Structure of MOL[®]LIK catalyst with SEM analysis (scanning electron microscope)⁸
a) Texture of MOL[®]LIK catalyst with mineral layer and metal structure and b) Surface of the mineral layer with electron-deficient and electron-rich areas

A matter of special importance is the fact that the iron ions in the mineral layer are mainly bound in tetrahedral gaps of a spinel structure⁹. On these lattice sites they cannot be further oxidized under normal conditions. They have an unpaired electron that is capable of interactions in the sense of a LEWIS base. Hydrolysis reactions can reduce COD (chemical oxygen demand) and TOC (total organic carbon), especially by degrading free ATP (adenosine triphosphate). Moreover, the catalytic supply of molecular water by the MOL[®]LIK-technology ensures a fine dispersion of lime particles resulting in lower turbidity and smaller concentrations of filterable substances¹⁰. It should be noted that all reactions take only place until the temperature- and pressure-dependent thermodynamic equilibrium position between the individual water structures has re-established in the water again¹¹. In addition, the function of

⁸ Benshueng et.al (2018) SEM analysis of MOL-catalyst, Beijing University of Chemical Technology

⁹ Koppe J R & Koppe J (2008) Draht als flexibler Metall-Mineral-Verbund, Symposium „Draht verbindet“

¹⁰ Koppe (2017) Die Dissoziation des Wassers, Merseburg University of Applied Sciences, ISBN 978-3-942703-81-9

¹¹ Koppe (2017) Das Wasser, Merseburg University of Applied Sciences, ISBN 978-3-942703-68-0

the catalyst lasts for many years¹². The minimum life time is 3 years if properly handled, with 5 to 10 years possible in technical applications.

Examples of installations of catalyst modules are shown in the following pictures¹³. Figure 4.2 presents the installation of a MOL[®]LIK catalyst module, at a pressure less storage tank for a tap water supplying network, with a flow rate of 100 m³/h. The installation of a MOL[®]LIK catalyst module at the make-up water supply of a power plant is shown in Figure 4.3.



Figure 4.2 MOL[®]LIK catalyst module
(Capacity: up to 100 m³/h water)



Figure 4.3 MOL[®]LIK catalyst module
(Capacity: up to 2000 m³/h water)

4.2 Technology perspective

With the scope investigating the potential for improving membrane performance with catalytic water treatment in the field of effluent water reuse, MOL[®]LIK was tested at the pilot plant shown in Figure 4.4 within the INSPIREWater project. The following parts were partly published by Wünsch et al.¹⁴ as a conference paper.

A secondary effluent from a production site of CLARIANT was used as the influent of the pilot plant. The flow rate entering the pilot plant was approximately 5 m³/h and split into two identical lines. One line in operation on conventional mode – the other line was a combination of conventional mode and MOL[®]LIK catalyst (MC, MOL Katalysatortechnik).

The secondary effluent is recovered in the pilot plant by end-of-pipe technologies, namely by ultrafiltration (UF, IntegraFlux[®] SFP-2880XP, DowDuPont) and reverse osmosis (RO,

¹² Koppe et al. (2016) Maximale Performance durch Wasser ohne Chemie – Energieeffiziente Wasserbehandlung

¹³ Koppe et al. (2015) Conservation of pipe works and membranes with energy efficient water treatment

¹⁴ Wünsch et al. (2019) High quality reuse of chemical industry wastewater by energy efficient membrane processes towards zero liquid discharge (ZLD), Aachener Tagung Wassertechnologie (in press)

FILMTEC® FORTILIFE® CR100, DowDuPont). The option to reduce the organic load of the secondary effluent by granular activated carbon (GAC, Organosorb® 20, Desotec) filters before UF-RO was temporarily tested as well.

Table 4.1 summarizes important parameters characterizing the secondary effluent from the existing wastewater treatment plant (WWTP), which serves as influent to the pilot plant. The existing WWTP fulfills all requirements for water discharge. The high organic load, expressed in the COD, is challenging for the industrial water treatment application, regardless of the technology used.

The RO reject stream is further concentrated via forward osmosis (FO). The diluted draw solution is recovered by high brine reverse osmosis (HBRO). A second FO stage coupled with membrane distillation (MD) is tested in batch tests to further process the concentrate from the FO-HBRO step. The minimized volume of concentrate can be treated by thermal evaporation and incineration, which is not tested within the demonstration. Residues (mainly salts) would be sent to the final disposal. Depending on the desired reuse quality, the water recovered by RO and FO might need an additional purification step (e.g. advanced oxidation) in case that the reused water shall come in contact with products.

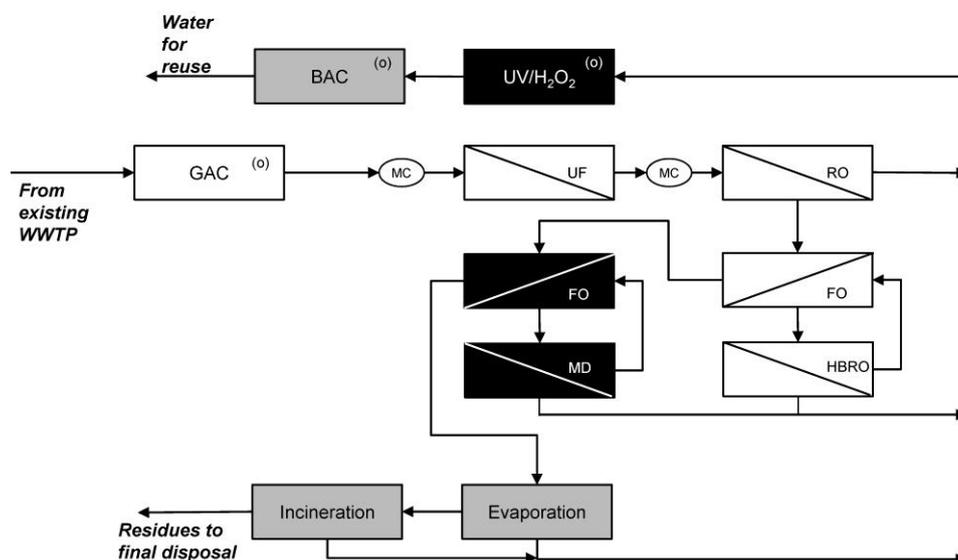


Figure 4.4 Scheme of the investigated process train. White filling: investigated in continuous pilot operation. Black filling: investigated in batch operation. Grey filling: not investigated within this study. “(o)”: Treatment option to achieve higher water quality.

Table 4.1 Average values ± standard deviations of important pilot plant influent characteristics. Data from continuous secondary effluent monitoring and laboratory analysis (October 2017 – April 2019).

Parameter	Unit	Limits	Value
COD	mg/L	< 700	416 ± 133
BOD	mg/L	< 300	27.4 ± 30.4
pH	-	6 – 10	7.8 ± 0.2
TSS Total suspended solids	mg/L	< 250	34.3 ± 29.1
Conductivity	mS/cm	-	9.4 ± 1.7
Hardness	°dH	-	41.8 ± 7.2
1,4-dioxane	ppm	-	19.2 ± 12.9

In the INSPIREWater project, it was not possible so far to operate the pilot plant set-up by using the challenging industrial secondary wastewater effluent as influent in a way that an external verification body (ETV – environmental technology verification) would allow the measured data for verification of the effects of the MOL[®]LIK catalyst. The reason was that no stable operational time of more than 5 days could be achieved.

However, effects of catalytic acceleration of the formation of molecular H₂O were found on RO membranes on a pilot installation in the field of wastewater reuse within INSPIREWater as well as the Belgian CARVE project (Chemicaliënrijke Afvalwater Recuperatie in der VoEdingsindustrie). Within the CARVE project, approaching zero liquid discharge, a stable operational time of the pilot plant of more than 14 days could be achieved. Moreover, it was possible to verify clearly positive effects of catalytic water treatment on membranes in the field of wastewater reuse in food and beverage industry. The treated wastewater was fed to RO (reverse osmosis) via an MBR (membrane bioreactor).

At CARVE the MOL[®]LIK catalyst was installed at the downstream of an MBR (membrane bioreactor) in the RO feed water. The scope of the pilot plant was checking out possibilities in minimizing the tap water demand for the food production process. Cause of the fact that the MBR was operating in the field of food and beverage, the wastewater stream was more stable – in direct comparison with the wastewater stream at the UR-RO-pilot plant setup in the INSPIREWater project.

Following cycles were operated and evaluated during the CARVE project:

Table 4.2 CARVE-Project – Description of operational mode

Cycle	Description of operational mode
A	Conventional operational mode
B	Conventional operational mode with catalyst (MOL [®] LIK) installed (Installation point: piping between the MBR and RO)

In Figure 4.5 is shown, that just a few days after the start of the experiment, noticeable differences between catalysed (MOL[®]LIK-treated) and conventionally treated water are evident. The catalysed water stabilizes the flow rate at a higher level than conventional treatment. At the same time, fewer cleaning cycles were necessary in the catalysed experimental cycle¹⁵. Parallel there were found improvements on pressure drop in the area of up to 30 percent – in direct comparison with conventional operational mode. This observation fits the water model described before.

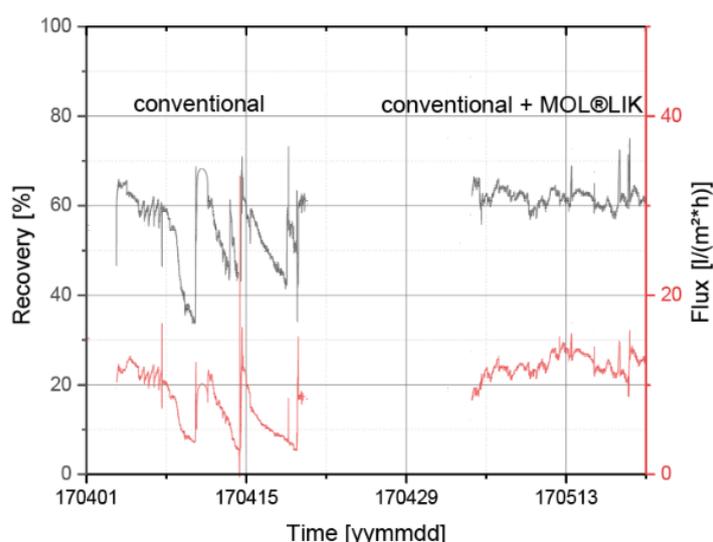


Figure 4.5 Overview of MOL[®]LIK at membranes in the field of effluent water treatment in food and beverage industry (based on ¹⁶)

4.2.1 Strengths and weaknesses of catalytic water treatment (MOL[®]LIK)

In a world where water scarcity is a growing issue, catalytic water treatment is a simple plug and play improvement solution for membranes. The MOL[®]LIK technology can be also used for improving conventional filter applications as well as many other areas where deposits formation in water should be avoided. The hugest weakness is that it is a largely unknown

¹⁵ Reyniers et al. (2017), passim, CARVE Project

¹⁶ Reyniers et al. (2017) Chemicaliënvrije Afvalwater Recuperatie in de VoEdingsindustrie – CARVE Project Meeting



novel technology, which is hard to explain how it works (overview of all strength and weaknesses in Table 4.3).

Table 4.3 Overview of SWOT analysis of MOL[®]LIK technology

INTERNAL	<ul style="list-style-type: none"> • Simple, plug and play • Flexible system, usage in different areas (membranes, filter systems, cooling tower, tap water supply) • Low CapEx & OpEx (capital & operational expenditure) • Very short payback time (ROI) • Suppress preventive formation of biofilms • Reduction of fouling, scaling and corrosion • Minimizing chemicals demand • Credible references 	<ul style="list-style-type: none"> • Largely unknown • Hard to explain what is the mechanism behind • No direct analytical technique to qualify concentration of “molecular water” - only indirect measurements to evaluate effectiveness and performance • Scientific proof is missing • No worldwide service options
	S	W
EXTERNAL	<ul style="list-style-type: none"> • More areas where scaling and fouling are an issue • Targets of the World Water Development Report • Stricter legislations • European approval for drinking water • Define easily monitored performance parameters • High transferability to sectors with water streams • Public growth of environmental awareness 	<ul style="list-style-type: none"> • Single source supplier • Immediate competition with established technologies • No comparable technology on the market • Commercial regulations at customers stop testing and eventually adoption • Low awareness of economic and ecological value of water treatment by end users
	O	T

Credible references – especially in the field of cooling water treatment – in combination with a very short payback time (ROI - return on investment) are helpful for waking up customer interest. However, without a scientific proof and no worldwide service options, commercial regulations at customers stop testing and eventually implementation. On the other hand, the public growth of environmental awareness with the possibility in defining easily monitored performance parameters are supporting the development of catalytic water treatment. That means novel technologies can lurch the market in combination with a proper after-sales

support. Therefore, projects like INSPIREWater in combination with technology verification tools are helpful in supporting to overcome customer's inhibitions.

4.2.2 Key performance indicators of MOL[®]LIK

At industrial projects - where the catalyst is involved in - the focus is mostly on the development of defining performance parameters, which can be easily monitored and used for demonstrating the efficiency of catalytic water treatment. In Table 4.4, examples of performance parameters are shown.

Table 4.4 Overview of key performance indicators for demonstrating and monitoring the performance of MOL[®]LIK technology

Process	MOL [®] LIK effect	Measurand	Measured effect	References
Membranes	Reducing difference in pressure (at constant flow rate)	Δp (p= pressure)	Pressure drop improved up to 30 %	17 18
Filtration	Improving filters cleaning interval	$\Delta p/\Delta t$ (t= time)	Cleaning interval improved from every 3 days to >> 30 days	19
Dissolving	Speeding up dissolving processes	$\Delta m/\Delta t$ (m= dissolved substance)	Dissolving process up to 10 times faster	20 21
Evaporation	Reducing scaling	Turbidity	Turbidity of cooling circuits less than 5 NTU	22 23
Heat exchange	Increasing heat transfer	k-value ΔT (T= temperature)	ΔT improved up to 2 K on industrial cooling circuits	24
Chlorination	Acceleration of chlorine disproportionation	Δ redox potential Organochlorides (AOX) chloramine	Improved performance on public swimming pools achieved	25 26

¹⁷ Koppe et al. (2011) RO-Anlagen - Chemiefreie Antifouling Strategie, VGB München.

¹⁸ Reyniers et al. (2017) Chemicalienvrije Afvalwater Recuperatie in de VoEdingsindustrie – CARVE Project Meeting

¹⁹ Koppe & Battagello (2017) Water structures & membrane systems – rising performance with catalytic water treatment; Merseburg University of Applied Sciences

²⁰ Koerner et al. (2015) Biozidfreie Wasserbehandlung von Kühlkreisläufen

²¹ Klimkewitz et al. (2014) Erstmalige Anwendung der biozidfreien Wasserbehandlung in Lackierstraßen bei AUDI, 13. VDMA-Wasser- und Abwassertagung

²² Ernhofer (2016) 10 Jahre Kühlwasserbehandlung mit dem MOL-Verfahren bei BAYERNOIL

²³ Schoenfelder et al. (2016) Behandlung von Ostseewasser für das Steinkohlekraftwerk Rostock, MerWaterDays

²⁴ Hagen (2015) Antifouling- und Antilegionella-Konzepte; VDI-Wissensforum „Kühlkreisläufe“

²⁵ Maurer et al. (2013) Biofilme abbauen und Legionellen vorbeugen

²⁶ Sinner et al. (2016) Fun splash without any risks in swimming pools, MerWaterDays

4.3 Market perspective

4.3.1 Market overview - Opportunities and threats of catalytic water treatment (MOL[®]LIK)

Rough estimates the global water usage is divided into three broad categories:

- 70% agriculture
- 22% industry
- 10% domestic²⁷.

The global view hides a much more divers pattern of end use by deeper look on figures of water distribution and availability. For example, in The Netherlands about 61 percent of water budget is used by industry and only 34 percent is spent on agricultural applications²⁸.

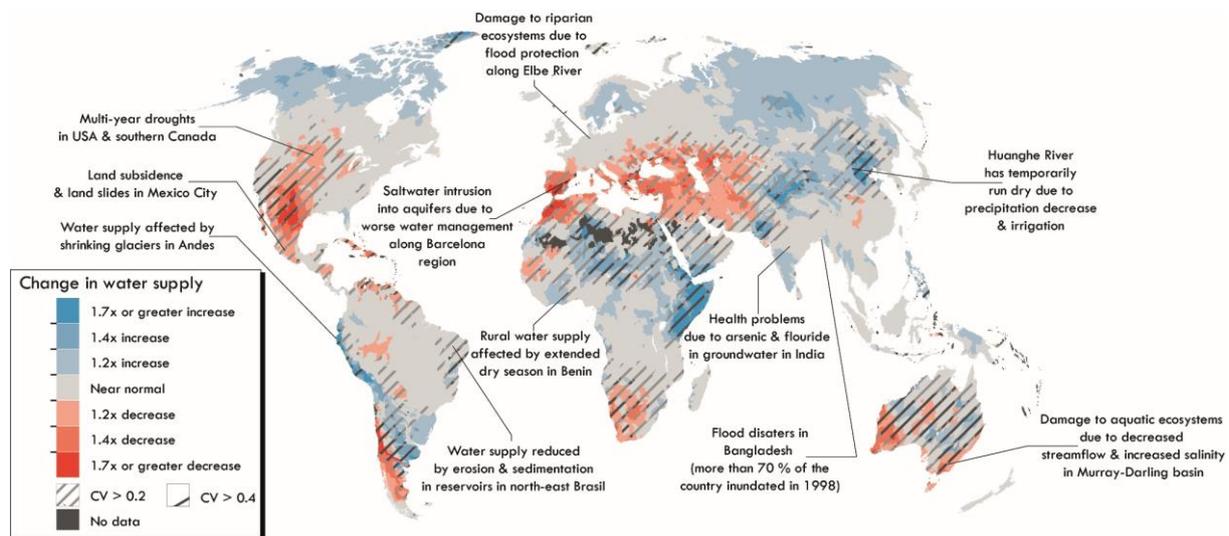


Figure 4.4 Weighted mean projected change in water supply from baseline (1950–2010) to future period (2030–50) under business-as-usual scenario with examples of current vulnerabilities of freshwater resources and their management (based on ²⁹ ³⁰)

With reference to Figure 4.4, we are living in a world where water scarcity is a growing issue. Due to this fact, water has to be used more intensively³¹. As water is used more intensively, more areas where scaling, fouling and biofouling are an issue will be there. Efficient and smart solutions, which are satisfying the public growth of environmental awareness, will be the market players with the hugest growing potential.

²⁷ Postel & Vickers (2004) Boosting Water Productivity, in State of the World

²⁸ Zeman et al. (2006) World water resources: Trends, challenges, and solutions

²⁹ Luck et al. (2015) Aqueduct Water Stress Projections: Decadal Projections of Water Supply and Demand Using

³⁰ Bates et al. (2008) Climate Change and Water. Technical Paper of the Intergovernmental Panel on Climate Change

³¹ Singh et.al (2016) Water Resources Management - Select Proceedings of ICWEES-2016

As more of these new technologies enter the market, it will be harder for the conventional technologies to compete. As wider range of possible applications, the harder novel technologies will be competed. That means for technologies like MOL[®]LIK it will be a huger challenge being accept on the market. It will be a combination of legislations and end users, which will support the market to launch efficient, smart solutions. With Figure 4.5 is shown in which areas smart solutions can enter more easily and in which areas more efforts will be required.

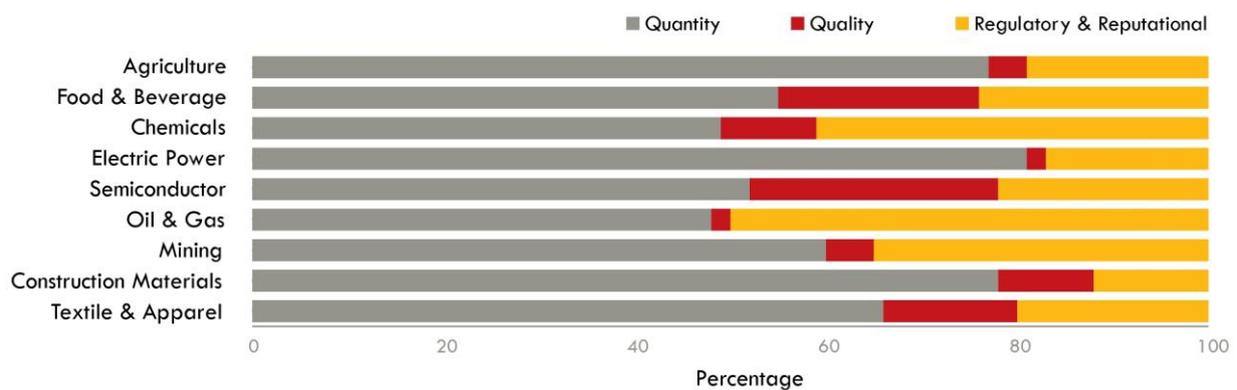


Figure 4.5 Pre-set weighting schemes for water usage on core industry sectors (based on³²)

The water market itself has the hugest growing potential of the future – because without water life as we know it wouldn't be possible. Here water reuse – as it is tested within INSPIREWater – plays a chief part. Projects like INSPIREWater will support smart solutions by entering the worldwide water market.

4.3.2 Economical evaluation

Effects of catalytic acceleration of the formation of molecular H₂O in the field of wastewater reuse where found on RO membranes on a pilot installation within the INSPIREWater as well as the Belgian CARVE project. With the following table, the effect of catalytically accelerated formation of molecular H₂O on 1 µm pre-filter units in the area of surface water treatment is shown:

³² Gassert et al. (2014) Aqueduct global maps 2.1 constructing decision-relevant global water risk indicators, World water resources

Table 4.5 Performance development on 1 µm pre-filters by the usage of catalyst in the area of surface water³³

Operational mode	Conventional	Catalytic (MOL [®] LIK)
Flow Rate [m ³ /h]	40 - 90	65 - 100
Differential pressure [bar]	0.1 - 0.3	0.05 - 0.1
Cleaning Interval [days]	2 - 3	30

In addition to the description of the development of the filter units, the following effects have been observed on the downstream RO membranes:

Table 4.6 Performance development on membranes by the usage of catalyst in the area of surface water (Catalytic retrofit was in 2016)³⁴

Design capacity	Conventional	Catalytic (MOL [®] LIK)
Permeate flow rate 120 m ³ /h (stable)	Decreases to 80 m ³ /h (in short times)	120 m ³ /h (stable)
CIP-Cleanings 4 CIP per year	22 till 28 CIP per year	10 CIP per year
Membranes lifetime 5 years	2 years	> 3 years

That means:

- ✓ Improved efficiency of chemicals used
- ✓ Permanent securing of the flow rate
- ✓ Less fouling issues on the membrane surface
- ✓ Minimize cleaning efforts by more than 50 %

The effect of catalytic water treatment on membrane applications is a much more stable operational mode. At CIP-intervals of industrial membrane systems there were enlargements of more than factor 2 observed – with improving the cleaning efficiency at the same time. In Figure 4.6, a brief conclusion of the effects after retrofitting industrial facilities with MOL[®]LIK is shown.

³³ Koppe (2018) Limitations in water supply – strategies for solving a global issue, International MerWaterDays

³⁴ Koppe (2018) Limitations in water supply – strategies for solving a global issue, International MerWaterDays

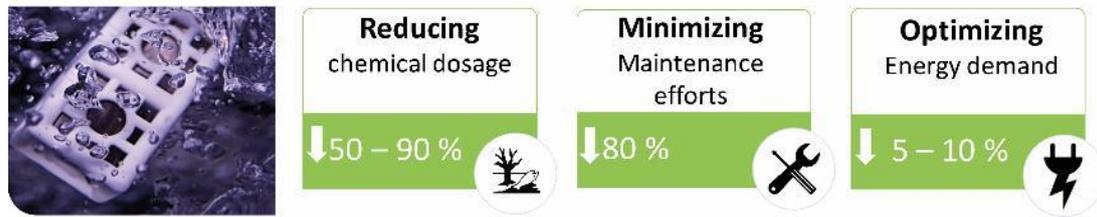


Figure 4.6 Overview of possible savings after MOL®LIK implementation on industrial facilities

4.4 Roadmap

4.4.1 Current technology readiness level

In the field of wastewater treatment, the TRL of MOL®LIK was improved from TRL 5 to 6 with the pilot plant within the INSPIREWater project in combination with the Belgian funded CARVE project. In INSPIREWater, the data analysis of the membrane performance and the impact of the catalyst is still ongoing. In direct comparison with CARVE, the data evaluation in INSPIREWater is more challenging due to no stable pilot plant operation of more than 5 days. With the scheduled ETV-verification at an industrial site with a more stable water quality, but comparable to the case study site in Tarragona, the possibility for further TRL improvements of MOL®LIK within INSPIREWater is given.

In areas of quite stable water quality MOL®LIK has already a TRL of 9 and is used for treating the make-up water supply as well as cooling circuits of entire power plants.

4.4.2 Barriers and challenges for implementation

Water treatment with suitable catalysts is a new way for solving troubles on water treatment. That means that with biocide-free MOL®LIK, a completely novel technology is lurching the market. According due the conventional strategies of market-dominating companies in solving troubles by increasing own turn over though higher chemicals dosage and maintenance efforts, the tendency in selecting “well-known technologies” instead of switching to a novel solution is common.

One challenge for MOL®LIK is showing the market that there is a potential in saving end-users’ money – without any risk on production capacity. The growing awareness of the global climate change in combination with stricter governmental regulations will support the development of smart solutions like MOL®LIK.

The ETV-verification on membranes on industrial scale within INSPIREWater will support the overcome customer’s inhibitions for retrofitting existing membranes plants with the catalyst. However, before catalytic water treatment is implemented as a standard solution on novel plants there will be further dissemination actions required.



4.4.3 Actions for implementation

Even with the TRL of 9 and the long-term applications in the field of power plants and refineries - the MOL[®]LIK technology is still largely unknown, which means that huger implementation and dissemination efforts are required. The level of awareness regarding the catalytic water treatment has to be increased on the European market by using different channels like presence with lectures & posters at conferences, fairs and workshops for sharing the results of pilot projects and references. The combination with activities at water boards, consultants and social media will give extra value on improving the awareness of catalytic water treatment.

Further actions for implementation are:

- ETV-verification on membranes on industrial scale within INSPIREWater
- Placing further pilot projects at industrial sites with well-known customers for creating references
- Preparation of Innovation Roadmap and Business model CANVAS for exploitation (see Table 4.4 & Figure 4.7)
- Common seminars, lecture series and papers with universities and customers for sharing knowledge on water chemistry and catalysis
- Further investigations of the processes in water and its structural formation changes

4.5 Conclusion

Water stress is a topic that our ecosystem has mastered since its creation. The combination of filtration and membrane technologies has been proven to separate high quality water and salts for millions of years. Humans just started using membrane technologies to provide clean water just a few thousand years ago. The driving force for this were economic aspects. Only for 60 years membrane technologies are available, which can provide high-quality water on a technically relevant scale. Economic aspects from the market are currently the driving force to make these systems more efficient. For these efforts to be targeted, knowledge about the physic-chemical processes on membranes is essential.

By using the catalytically accelerated water treatment, the system performance and thus the profitability of membranes systems is increased. At the same time, this reduces the negative impact on the ecosystem by reducing the associated use of chemicals and energy.

In a world where water scarcity is a growing issue, there will be more areas where scaling, fouling and biofouling of water treatment technologies e.g. membrane filtration, will arise. Efficient and smart solutions, which are satisfying the public growth of environmental



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awareness will be the market players with the hugest growing potential. Here catalytic water treatment – so called MOL[®]LIK technology – as a simple plug and play solution for improving efficiency in various areas could play an important role.

As more smart technologies enter the market, the harder it will be for the conventional technologies to compete by minimizing the effect on existing business models. It will be a combination of legislations and end users, which will support the market launch of resource efficient smart solutions. Stricter the legislations on chemicals usage, water reuse and energy efficiency will support the launching of smart and new technologies.

Here it was helpful that MOL[®]LIK technology was demonstrated on wastewater reuse at CLARIANT site in Tarragona within INSPIREWater as well as the Belgian funded project CARVE by improving its technology readiness level on wastewater applications. Further activities at market launching will be supported by the scheduled ETV-verification on an industrial scale membrane system within the INSPIREWater project together with further dissemination and implementation activities.



Table 4.4 Business model CANVAS – MOL®LIK on membranes at wastewater reuse

Key partners	Key activities	Value proposition	Customer relationship	Customer segments
<ul style="list-style-type: none"> Worldwide acting end users Equipment suppliers as potential resellers Consultants Planning offices Suppliers who are interested in improving their plant performance 	<ul style="list-style-type: none"> Commercial & marketing activities Acquisition of new customers & projects Sharing knowhow on water structures and catalysis Technology verification on MOL®LIK Adjusting equipment production 	<p>Improved end-users' facilities performance through:</p> <ul style="list-style-type: none"> Reduction of scaling, fouling, corrosion Suppress preventive formation of biofilms Reduction of difference in pressure on membranes Improved filters and membranes lifetime Increased heat transfer Reduced chemicals & energy demand Minimized maintenance efforts Easy to implement & maintain on current facilities 	<ul style="list-style-type: none"> Support on chemical & water questions Commission support Platform with possibility for exchange between customers After-sales support Preparing common papers 	<p>All kind of customers with higher demand of water and heat transfer. Especially:</p> <ul style="list-style-type: none"> Power plants Chemical industry Steel industry Oil and Gas Food & beverage Pharma industry Public swimming pools Tap water supplying networks
<p>Cost structure</p> <ul style="list-style-type: none"> Equipment purchases Storage costs Dissemination & implementation activities 		<p>Revenue Streams</p> <ul style="list-style-type: none"> Selling the catalyst for various selling options Equipment rental and technical support After-sales services 		

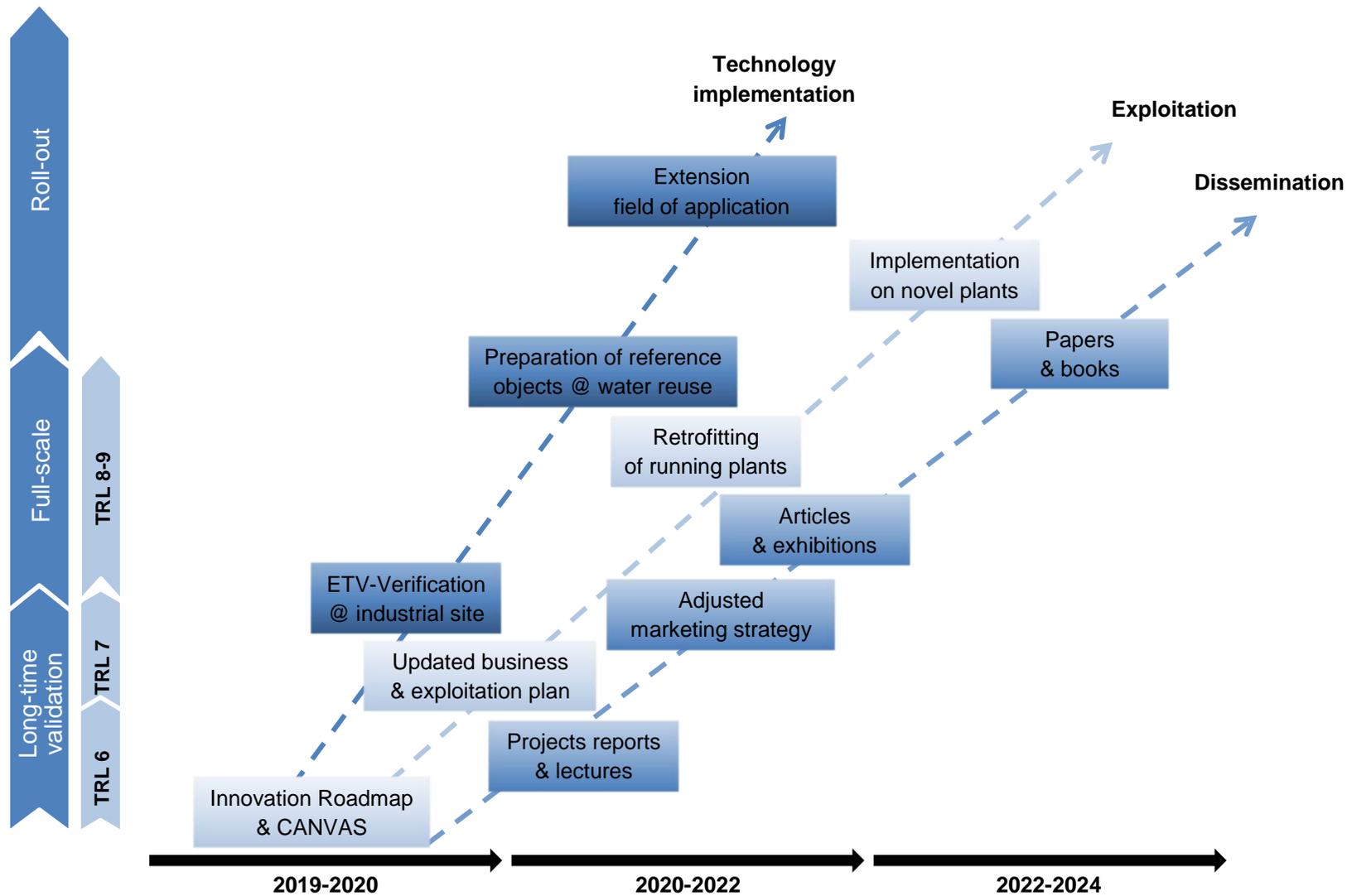


Figure 4.7 Innovation Roadmap of catalytic water treatment (MOL®LIK) on membranes at water reuse

5. Forward osmosis coupled with high brine reverse osmosis™ and membrane distillation

5.1 Introduction

Zero liquid discharge (ZLD) is a wastewater treatment concept whose aim is to separate the wastewater into clean water and a solid stream of pollutants. The existing ZLD approaches use multi-effect evaporation and crystallization units to recover the water resulting from a reverse osmosis unit. From an energy consumption point of view, the bottleneck of the current approach is the highly energy intensive evaporation step, which typically requires 30 – 120 kWh/m³¹.

Forward osmosis (FO) coupled with high brine reverse osmosis (HBRO™) and membrane distillation (MD) is an innovative technology developed by BLUE-tec, which has the potential to compete with evaporators in wastewater treatment aiming zero-liquid discharge. In forward osmosis, the wastewater and a high concentration salt solution are brought into contact with a semipermeable membrane. Due to differences in osmotic pressure, clean water naturally passes through the membrane from the wastewater stream towards the salt solution, resulting in a concentration of the wastewater. Unlike evaporation, which is a very energy intensive process, forward osmosis takes place without any phase change. The working principle is illustrated in Figure 5.1.

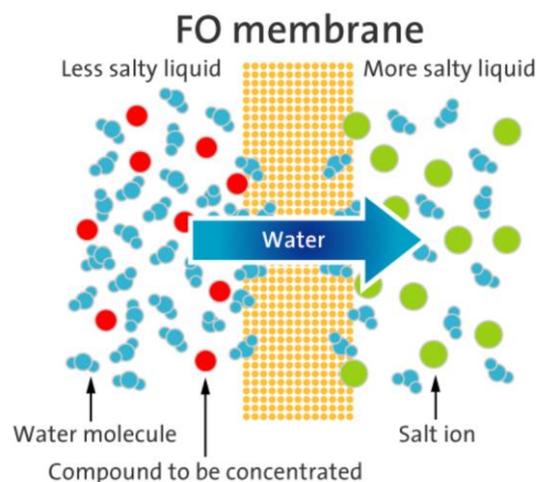


Figure 5.1 Schematic representation of the forward osmosis technology

¹ Semiat (2008) Energy Issues in Desalination Processes

The draw solution concentration is recovered through the HBRO™ technology, which uses specific high pressure membranes resulting in both a high concentrated draw solution and clean water. The difference between reverse osmosis and HBRO™ is that the HBRO™ membranes are partially permeable to salt; therefore, the difference in osmotic pressure on the two sides of the membrane is decreased. This allows higher osmotic pressure draw solutions to be obtained using the same mechanical pressure as applied for conventional RO. A comparison between the working principle of the FO, RO and HBRO™ is shown in Figure 5.2.

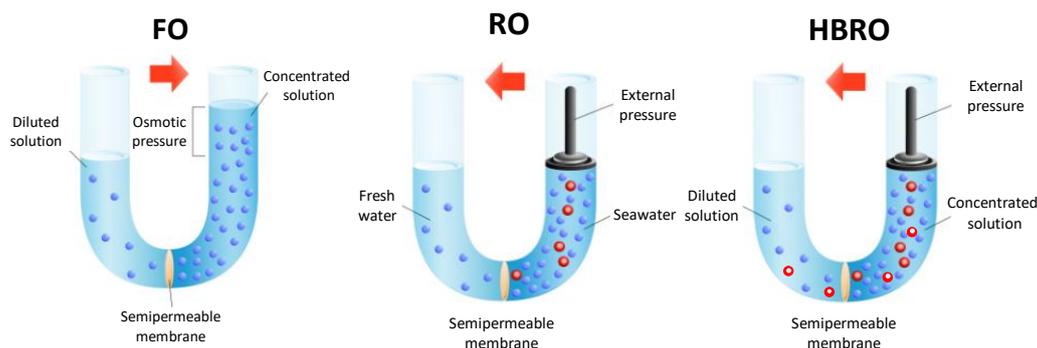


Figure 5.2 Schematic representation of FO, RO and HBRO™ technologies

The working concept of the membrane distillation technology is presented in Figure 5.3. The feed solution is heated and the vapors are transferred on the other side of a hydrophobic membrane, where they are condensed. The advantage of using membrane distillation compared to evaporation is that in membrane distillation it is not required to heat the feed solution until the boiling point, making possible the use of waste heat (82°C) which is available in high quantities in the industry. However, the amount of energy required, is similar to the one used in evaporation. Therefore, membrane distillation is usually applied to recover high concentration draw solutions.

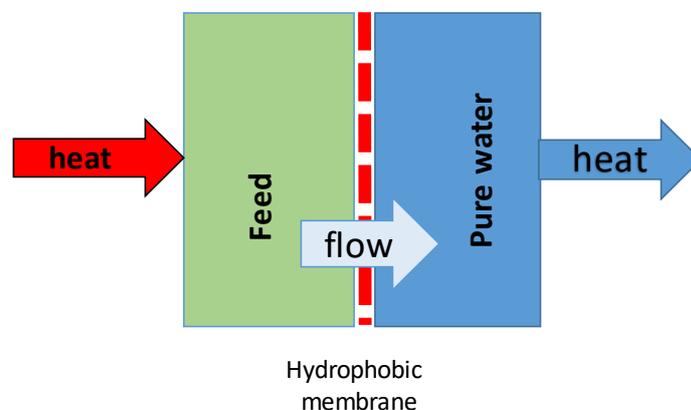


Figure 5.3 Schematic representation of membrane distillation technology

5.2 Technology perspective

The pilot plant constructed by BLUE-tec and operated in Tarragona had a feed capacity of 12 m³/d. NaCl was used as draw solution in the HBRO™ section of the pilot. The block scheme of the pilot is presented in Figure 5.4. The concentrated wastewater (1), treated before with an ultrafiltration and reverse osmosis, together with a concentrated draw solution (6) are fed to the FO section of the pilot. The wastewater concentrate stream (2) leaves the system after clean water passed through the FO membrane. The diluted draw solution (3) is fed to the draw solution tank (DS), from which it is fed to the HBRO™ section of the pilot (4). The HBRO™ section produces clean permeate (5) and concentrated draw solution (6) which is fed to the FO section again. Photos of the FO and HBRO™ sections of the existing unit are shown in Figure 5.5.

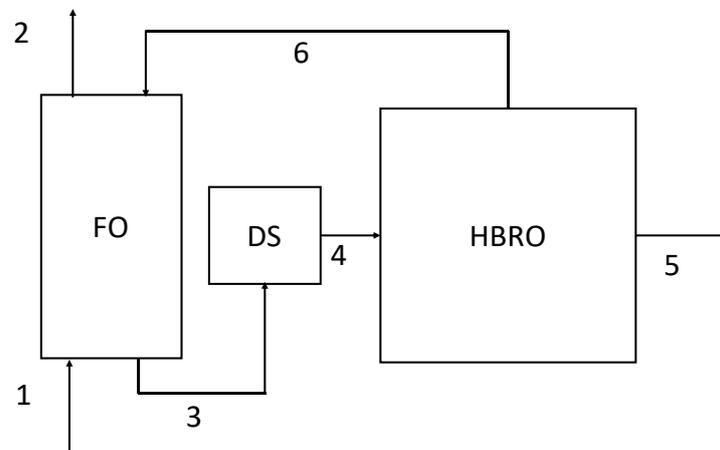


Figure 5.4 Block scheme of the FO-HBRO™ pilot



Figure 5.5 FO (left) and HBRO™ (right) sections of the BLUE-tec pilot operated in Tarragona

A batch of FO-HBRO™ concentrate was sent to Renkum, The Netherlands and further concentrated using an FO-MD bench scale unit. The scheme of the FO-MD set-up is shown in Figure 5.6. The set-up uses an air-gap membrane distillation module (AGMD), which uses the

part of heat required to condense and cool down the vapor for pre-heating the feed, reducing thus the energy requirements. A photo of the set-up is shown in Figure 5.7.

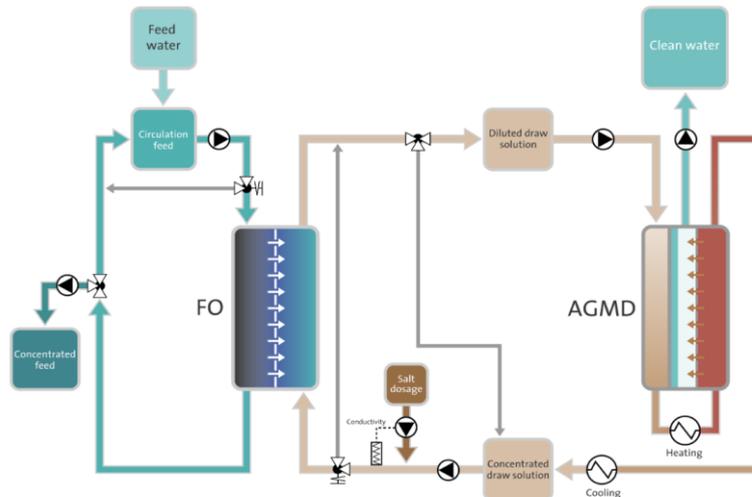


Figure 5.6 Scheme of the FO-MD pilot



Figure 5.7 FO-MD pilot used for tests in Renkum

5.2.1 Strengths and weaknesses of forward osmosis

The technology strengths and weaknesses are the direct result of the working principle and the stage of development of the technologies. As shown in the previous section, by using FO membranes, the wastewater concentration takes place naturally, without phase transition and without the necessity to use high temperature or high pressure. However, the membranes need to be further developed to reduce the reverse salt flux and the possibility of various components to pass from the wastewater inside the draw solution. Because of the absence of phase transition, the energy requirements inside the HBRO™ section are lower compared to the evaporators, but high pressure is necessary, which imposes some safety measures for the pilot operation. When membrane distillation is used, water needs to be evaporated, which

means that the heat of vaporization needs to be provided to the system. However, because lower temperatures are required compared to conventional evaporators, the energy can be provided as low quality heat, and the units are built to efficiently use the heat provided to the system, so that the major part of the heat of evaporation is recovered within the MD-unit. The strengths and weaknesses are summarized in Table 5.1.

Table 5.1 Overview of SWOT analysis of forward osmosis coupled with high brine reverse osmosis and membrane distillation

INTERNAL	<ul style="list-style-type: none"> • Ambient temperature and pressure required for wastewater concentration • No phase transition required for wastewater concentration • Low energy consumption of the HBRO™ compared to evaporators • Low quality heat can be used in the MD 	<ul style="list-style-type: none"> • Frequent membrane cleaning might be necessary , definition of adequate cleaning procedures required • FO are still under development • High reverse salt flux, subject of advanced membrane research • High pressure is required in the draw solution recovery section • Insufficient FO membrane selectivity considering exchange of components in feed and draw solution • New MD technology is still improvable, but high potential available
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EXTERNAL	<ul style="list-style-type: none"> • Huge market potential in industry for concentration of different type of streams not just wastewater but also product streams with high added value 	<ul style="list-style-type: none"> • Better FO membranes are not developed fast enough • A lot of research is currently done for improving ZLD wastewater treatment, so there is the possibility that new technologies which are competitive with FO-HBRO™ and FO-MD will be developed
	<div style="display: flex; justify-content: space-around; font-size: 2em; font-weight: bold;"> O T </div>	

5.2.2 Key performance indicators of forward osmosis

During the tests, it was shown that in principle forward osmosis coupled with high brine reverse osmosis can be used for wastewater concentration. Stable and consistent operation was achieved during the tests and fluxes between 5 and 7 L/m²/h and volumetric concentration factors between 2 and 3 were obtained in continuous operation. A maximum volumetric concentration factor 9 was obtained in closed loop operation. Frequent cleanings were

necessary for both the FO and HBRO membranes, leading to the consumption of 1.5 L of acid detergent, 1.2 L base detergent and 0.02 L of HCl for each m³ of permeate produced by the pilot. The energy consumption was 36 kWh/m³ of permeate. The results are summarized in Table 5.2. With FO-MD, additional volumetric factors between 3 and 4.5 were obtained using FO-HBRO™ concentrate obtained in continuous operation leading to a total concentration factor between 6 and 13.5. The distillate quality was much higher compared with FO-HBRO, 0.08 - 1 g_{NaCl}/L. The gained output ratio (GOR) of the unit was 1.6 at 1.9M draw solution concentration, which is an energy consumption of 430 kWh/m³ distillate in form of low-grade waste heat of 80°C. It is expected that this energy consumption can be reduced to 250 – 300 kWh/m³ by optimizing the specific process conditions in the MD module (feed flow, temperature difference and limitation of heat losses) (Table 5.3).

In consideration of the total INSPIREWater treatment train for water reuse of the chemical industry, the total concentration factor of the RO-brine with FO technologies is between 6 and 13.5 and for the direct treatment of the UF permeate 12 to 27. This corresponds with a total water recovery over the RO, FO-HBRO™ and FO-MD system of 83 to 96%.

Table 5.2 Overview of key performance indicators for demonstrating and monitoring the performance of the FO-HBRO™

KPI description	Unit	KPI
FO flux	L/m ² /h	5 - 7
Volumetric concentration factor	–	2 - 3/9**
Permeate quality	g _{NaCl} /L	0.1 - 2.7*
NaCl consumption	kg _{NaCl} /h	1 - 2.7*
Energy demand	kWh/m ³ _{permeate}	20***
Cleaning detergent acid	L/m ³ _{permeate}	1.5
Cleaning detergent base	L/m ³ _{permeate}	1.2
Cleaning detergent HCl	L/m ³ _{permeate}	0.02

* The higher value was obtained towards the end of the testing period, due to membrane wear

** Volumetric concentration of 9 was reached in a batch test

*** In the pilot an energy consumption of 36 kWh/m³ was found, with the use of pressure recovery systems an energy use of 20 kWh/m³ is expected.

Table 5.3 Overview of key performance indicators for demonstrating and monitoring the performance of the FO-MD

KPI description	Unit	KPI
FO flux	L/m ² /h	4 - 6
Volumetric concentration factor	–	3 - 4.5
Distillate quality	g _{NaCl} /L	0.08 - 0.1
Energy demand	kWh/m ³ _{distillate}	300*

* In the test set up, an energy consumption of 430 kWh/m³ was measured. It is expected, that by optimizing process conditions an energy consumption of 250 – 300 kWh/m³ could be achieved

5.3 Market perspective

5.3.1 Market overview - Opportunities and threats of forward osmosis

The main opportunities for applying the FO-HBRO™ lay in industries in which concentration of products is required and evaporation costs need to be reduced or where thermal degradation of products needs to be avoided. Industrial and domestic wastewater treatment towards zero liquid discharge is a valid candidate, provided, that the wastewater does not contain components, which damage the membrane. With some adjustments, the technology can also be applied for food and pharma industries, given that the membranes will be further developed towards reducing the component permeability and that draw solutions which do not contaminate the product are successfully investigated. Slow membrane development can delay the technology application in some industries. In addition, the concentration of products in the industries mentioned above is an intensively researched topic, which poses the threat that other competitive technologies will emerge. The opportunities and threats of the technology are summarized in the SWOT analysis in Table 5.1.

In the European market, it is estimated that for the following five years the technology will be mainly applied in The Netherlands and surrounding countries in the wastewater, food and pharma industries. It is estimated that each unit will have a capacity of 4'000 - 40'000 m³_{wastewater}/a, assuming 8000 h/year of operation. The estimation is presented in Table 5.4.

Table 5.4 Number of wastewater application fields in European countries and estimated operational capacity per year and country

Country	Number of application fields <i>(sorted max to min)</i>	Estimated overall operational capacity (m³/a)
The Netherlands	3	12'000 - 120'000
Germany	1	4'000 - 40'000
Belgium	1	4'000 - 40'000
Total	5	20'000 - 200'000

On the long run, it is expected to construct 20 additional pilots in Europe, as predicted in the project proposal, assuming that the FO membranes and the draw solution will be further developed. Outside Europe, the technology has a big potential to be applied in India, due to strict regulations regarding ZLD². It was estimated that currently there are 150 units in Tirupur

² Muhammad & Lee (2019) Zero-liquid discharge (ZLD) technology for resource recovery from wastewater: A review

region, 450-500 in India and 10-15 in the regions close to India (for example Bangladesh). It is estimated that by 2025 40 new units will be constructed in India³.

5.3.2 Economical evaluation

The FO-MD technology is used only to further concentrate the FO-HBRO™ concentrate, which means that the amounts of wastewater treated through FO-MD are low. Moreover, due to high-energy consumption compared to evaporation, the use of the technology is feasible only in cases where low-grade heat is already available on the site. Therefore, the economic calculations will focus on the FO-HBRO™ technology.

As shown in Table 5.4, for the following five years, it is estimated that five plants will be constructed and operated for wastewater concentration in The Netherlands and surrounding countries. For a concentration factor 3, it is estimated that the wastewater production will be reduced by 13'000 – 130'000 m³/a. Assuming a fresh water cost of 0.9 €/m³⁴, 11'700 – 117'000 €/a of direct cost savings is expected because of the clean permeate production. For an average total cost of 4 €/m³ wastewater treatment, the technology will bring cost savings in the order of 1 €/m³ of avoided wastewater, resulting in 13'000 – 130'000 €/a due to less wastewater treatment.

For treatment of (concentrated) wastewater, cost savings come from the reduced energy consumption compared to evaporators. The evaporators require 30-120 kWh/m³_{distillate}⁵ depending on the type, capacity and efficiency. During the tests it was calculated an energy consumption for the FO-HBRO™ technology of 36 kWh/m³_{permeate}. Assuming an average consumption of 75 kWh/m³_{distillate} for the evaporators and an energy cost of 0.1 €/kWh⁶, the savings in energy brought by the FO-HBRO™ range between 50'700 - 507'000 €/a. On full-scale applications, pressure (energy) recovery systems could be implemented, which would reduce the energy consumption to approximately 10 - 15 kWh/m³_{distillate}, resulting in further energy savings.

5.4 Roadmap

5.4.1 Current technology readiness level

As part of the INSPIREWater project, the FO-HBRO™ pilot plant was operated on the Clariant site in Tarragona, Spain. The pilot was on site between July 2018 and May 2019. Between

³ Meyyapaa Kannan (2019) Chairman Nuva Machine Works India Pvt Ltd (evaporator supplier) Tirupur, India, personal communication

⁴ INSPIREWater Project Proposal (2017)

⁵ Semiat (2008) Energy Issues in Desalination Processes

⁶ INSPIREWater Project Proposal (2017)

August and mid-October, the pilot behavior with different draw solution concentrations was tested, followed by the implementation of GAC filters on the site between mid-October and mid-November. Afterwards, tests with two types of wastewater were performed. On total, 21 pilot runs were performed with the wastewater, leading to a total operation time of 125 h, the maximum running time being 14 h. The operation was interrupted by cleanings, optimization and maintenance in the FO-HBRO™ and other units on site. The results from different runs are consistent with each other and the pilot was stable during the above-mentioned operation duration. This leads to the conclusion that the first steps towards technology readiness level 6 were achieved.

Four concentration tests have been performed using the FO-MD pilot in Renkum, the Netherlands, using FO concentrate from the FO-HBRO™ pilot, leading to results, which were consistent with each other. This makes the FO-MD applied for the current application a technology readiness level 5 technology.

5.4.2 Barriers and challenges for implementation

The technology novelty constitutes one difficulty for the implementation of the technology on a big scale. The technology is still not well known, which means that efforts need to be put in making the technology known.

A technical challenge is posed by the current development of the FO membranes themselves. To be able to apply the technology in fields like food and pharma, the FO membranes need to be developed towards reducing their reverse salt flux, to avoid product contamination with draw solution.

A challenge for wastewater treatment can be encountered if it contains components, which permeate towards the FO membrane and have the potential to foul/scale the HBRO™ membranes. In this case, frequent cleaning is necessary, which requires the consumption of cleaning water. In case of severe scaling, the membranes need to be replaced frequently.

5.4.3 Actions for implementation

The actions for future implementation of the technology are summarized in Figure 5.8.

From a technical point of view, a first step is represented by the process optimization. In this stage, various draw solutions and other FO membranes need to be tested. Minor changes in the process can already result in a successful improvement of the technology. Afterwards, the pilot plant can be further tested for wastewater applications in research projects and once the process is optimized, its application can be extended to other industries.

Regarding the technology exploitation, at first the technology will be rented to potential customers, followed by full-scale commercialization and licensing. The business model

CANVAS for the technologies as part of the business plan, being developed in the project, is presented in Table 5.5.

The novelty of the technology makes it unknown for potential customers, which makes dissemination as a major tool for a successful implementation. At first, the results obtained with the technology in various research project will be presented in papers, patents and conferences. When the technology becomes more mature, the technology can be presented through open days, exhibitions and guided visits.

5.5 Conclusion

As part of the INSPIREWater project, the FO-HBRO™ technology was demonstrated for short operation periods on the Clariant site in Tarragona, achieving thus the first steps towards technology readiness level 6. The FO-MD tests confirm the technology readiness level 5 of the technology.

The technology can be applied in domestic and industrial wastewater treatment, as well as for concentrating products in food and pharma industries. Further development regarding the draw solution and membrane properties need to be done to achieve full scale application. Being a novel technology, effort needs to be put in results dissemination.

Although, the relatively high specific energy consumption of the FO-MD unit, it could be an attractive solution for concentration FO-HBRO™ brines, due to the use of low grade energy (80°C), the compact construction and use of plastic materials.

Table 5.5 Business model CANVAS – FO-HBRO™ and FO-MD

Key partners	Key activities	Value proposition	Customer relationship	Customer segments
<ul style="list-style-type: none"> • Membrane suppliers • Equipment suppliers • Construction companies 	<ul style="list-style-type: none"> • Development of membrane technology • Evaluation in long term tests 	<ul style="list-style-type: none"> • Concentrate and recycle 	<ul style="list-style-type: none"> • Training and support on how to operate the installation 	<ul style="list-style-type: none"> • Water boards • Food industry • Pharma industry
	<p>Key resources</p> <ul style="list-style-type: none"> • Know-how on membrane technology • FO-HBRO™ and FO-MD installations 		<p>Channels</p> <ul style="list-style-type: none"> • Presence in fairs, conferences, open days • Being part of water boards 	
<p>Cost structure</p>		<p>Revenue Streams</p>		
<ul style="list-style-type: none"> • Equipment purchase • Construction 		<ul style="list-style-type: none"> • Selling the plants • Licensing the technology 		

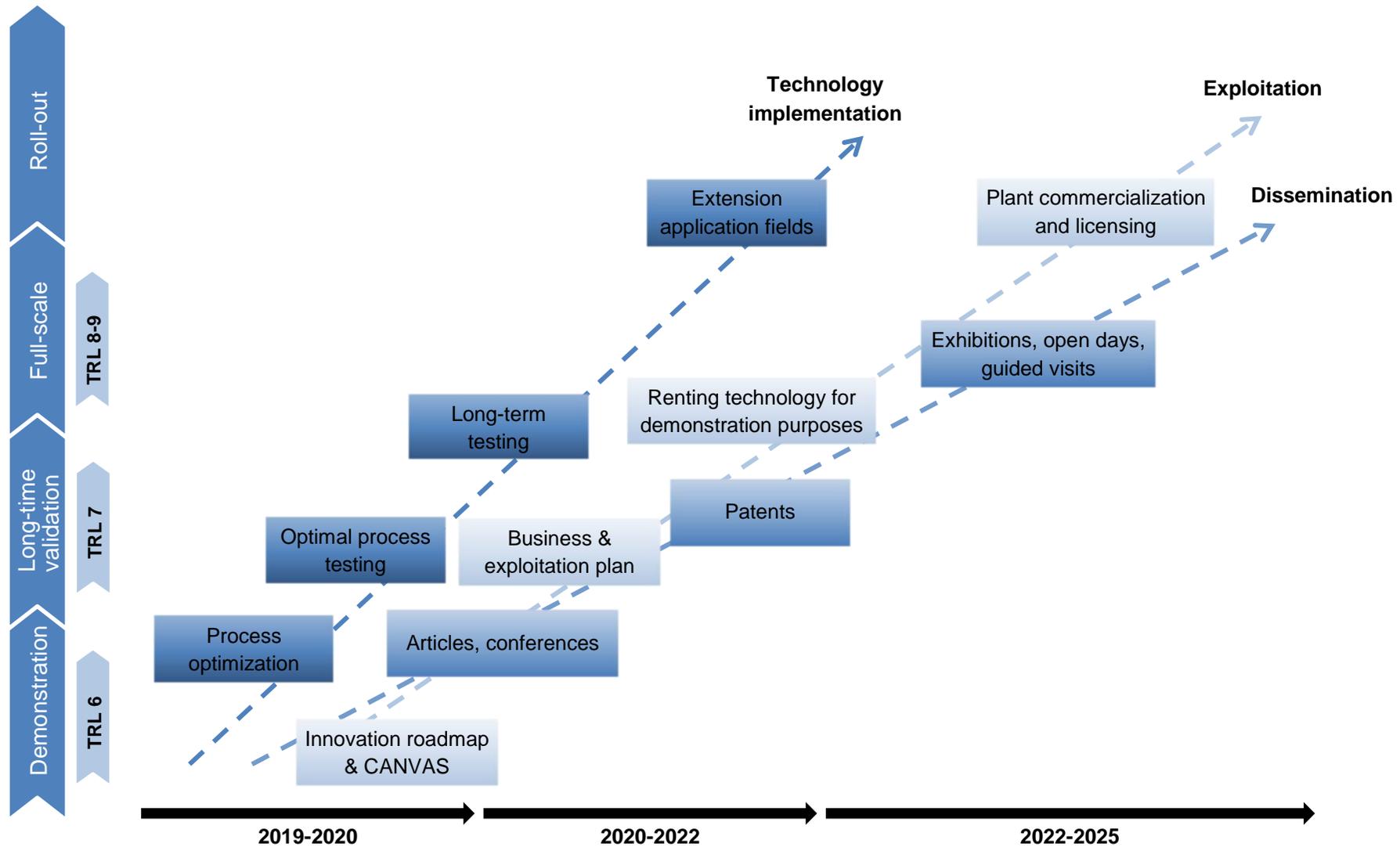


Figure 5.8 Innovation Roadmap of FO-HBRO™ and FO-MD technology



6. Fouling resistant reverse osmosis elements

6.1 Introduction

Biological and organic fouling is one of the more challenging types of fouling to control in reverse osmosis (RO) systems. It is defined as the growth and accumulation of micro-organisms and their respective extracellular polymeric materials (EPS) inside the feed channel of the spiral wound RO element. The feed channel becomes blocked and causes the feed-concentrate pressure drop (dP) across the RO pressure vessel to increase and the water permeability through the membrane to decrease. In addition, irreversible mechanical damages to the elements may occur if the pressure vessel dP exceeds 3.5 bar, which would further reduce the system efficiency. Clean-in-place (CIP) protocols employing aggressive chemicals are used to manage the system when biofouling occurs, but these may be necessary as frequently as every 1-2 weeks. In total, biofouling and frequent cleanings will affect energy and chemical consumption, element lifetime, water productivity and cost of water produced.

The DuPont FILMTEC™ FORTILIFE™ CR100 resistant RO elements offer relief to the end user who performs frequent cleanings due to fouling. The 8-inch elements offer 44 m³/d (11,500 gallons of water per day) with 99.7% stabilized salt rejection (99.4% minimum rejection) and a pressure drop of 0.1 bar at standard test conditions of 2,000 ppm NaCl, 225 psi (15.5 bar), 77 °F (25 °C), pH 8 and 15% recovery. These elements are built on DuPont FILMTEC RO characteristic performance standards, and offer greater fouling-resistance fueled by advances in membrane chemistry and module design for today's fouling prone water types. Fouling-resistant membrane chemistry carefully controls charge, roughness and hydrophilicity. Coupling a membrane with a mechanically engineered module designed to control shear profiles and element efficiency provides a complete package. A water treatment plant experiencing frequent cleanings due to rapid increase of differential pressure, a symptom of fouling, can expect the following relative to leading fouling resistant products currently available in the market:

- Up to 50% reduction in the number of cleanings in comparison with the conventional product. The commercial product it is equipped with an organic fouling-resistant membrane chemistry that reduces blocking by organic biofilm attachment and natural organic matter. Furthermore, after installation, the product reduces cleaning frequency of an RO system, which, in turn, helps improve up time and reduce maintenance costs when the results are compared to the conventional product BW30XFR-400/34 (see Figure 6.1).

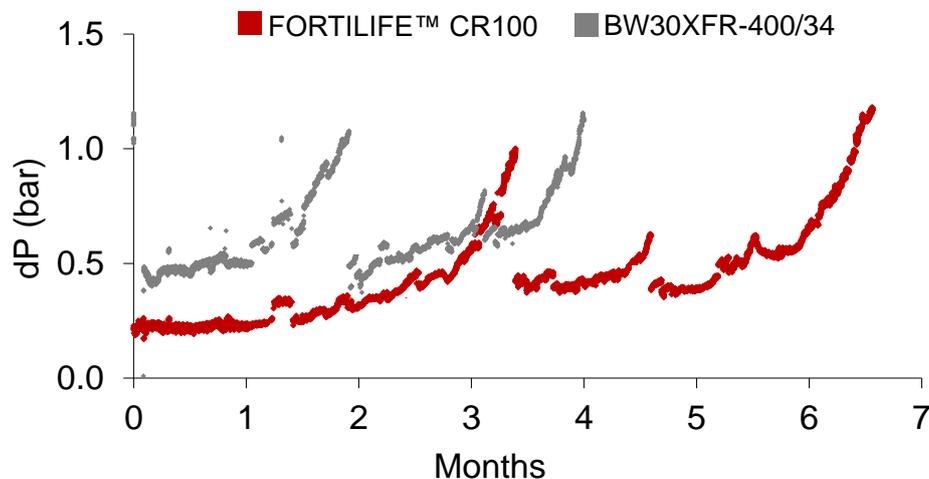


Figure 6.1 Pressure drop (dp) progress over time on FILMTEC™ BW30XFR and FILMTEC™ FORTILIFE™ CR100

- Effective and efficient cleaning of biofilm, organic compounds and scale, achieved through the ability to use a wide pH range during cleaning (pH 1–13)
- Up to 10% energy savings at the same water productivity

6.2 Technology perspective

In the case study in Clariant, Tarragona, in total 18 DuPont FILMTEC™ FORTILIFE™ CR100 elements were in operation with the effluent of the industrial wastewater treatment plant run by Clariant. The effluent was pre-treated by ring filter and ultrafiltration and during part of the trial also by granulated activated carbon. The reverse osmosis (RO) installation contained two independent lines. Each line consists of two stages with 4-inch commercial spiral wound elements installed in pressure vessels (three elements/pressure vessel). The first stage has two parallel pressure vessels. The concentrate of these two parallel line are combined and enter to the second stage with one pressure vessel. Therefore, each line contains 6 elements in first stage and 3 elements in second stage.

Treatment line with UF200 and RO1 (red line) incorporates the heterogeneous catalyst (MOL®LIK) in the respective feed tanks upstream of the ultrafiltration and reverse osmosis modules (Figure 6.2). Treatment line with UF201 and RO2 (blue line) serves as reference line. The RO unit is equipped with online flowmeters, pressure controllers, dP indicators and conductivity meters.

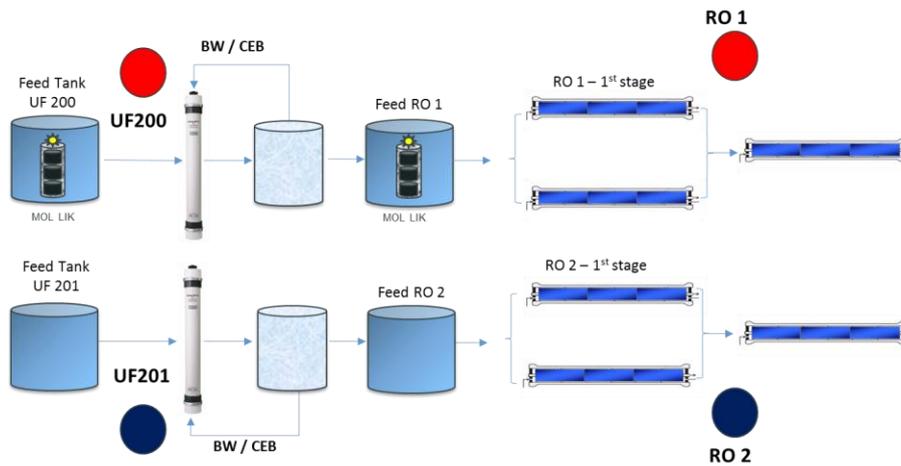


Figure 6.2 Scheme of the UF-RO pilot plant with two RO lines

The operation of the single unit processes started at different dates and lasted 16 months (see installation of RO in Tarragona in Figure 6.3). To be remarked that RO system was started in 5th December 2017. Since 6th March 2019, all unit processes were operated during working days only (Monday to Friday) to assure good supervision of the systems. The common end of the piloting was 12th April 2019. However, it is important to remark that the study undertaken within this project did not intend a comparison with the conventional product, nor demonstrate the sustainable operation over time in this challenging and variable environment with industrial wastewater feed.



Figure 6.3 Picture of the RO system used during the study



6.2.1 Strengths and weaknesses of fouling resistant reverse osmosis elements

The technology is among market leaders and especially interesting for applications with high fouling potential looking for an improvement of the cost-efficiency and robustness of the process (see SWOT analysis Table 2.2).

The features and benefits of the latest, third-generation, fouling-resistant RO membrane (see history of development in Figure 6.4) are provided in Table 6.1.

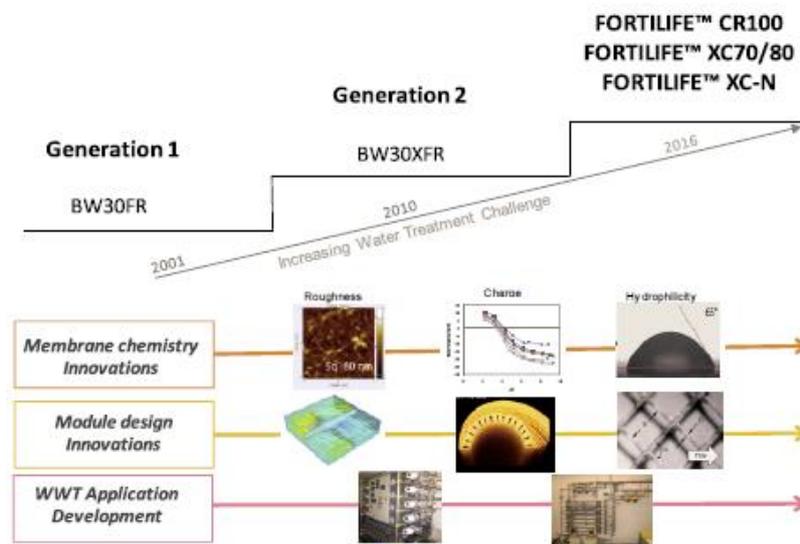


Figure 6.4 Generations of fouling resistant RO technology

Table 6.1 Features and benefits of the fouling-resistant RO membrane

FEATURES	BENEFITS
Low differential pressure	Up to 50 percent less cleanings because of fouling improved hydraulic balance for more fouling resistance and lower energy
Spiral-wound, reverse osmosis module	
Organic fouling-resistant membrane chemistry	Reduced rate of normalized flux loss in challenging waters
Durable membrane across a wide pH range	Highly cleanable element with long lifetime
High membrane water permeability	Up to 10 percent lower energy operation
Competitive salt rejection	High permeate quality to facilitate blending with higher TDS waters for reuse



H2020-IND-CE, SPIRE-01-2016

A third-generation RO membrane can help provide an exceedingly low pressure drop module design for an 8-inch, 400-square-foot module compared to the previous generation of RO membranes, shown in Figure 6.5 and 6.6. It is equipped with an organic fouling-resistant membrane chemistry that reduces blocking by organic biofilm attachment and natural organic matter. Furthermore, after installation, the product reduces cleaning frequency of an RO system, which, in turn, helps improve uptime and reduce maintenance costs.

As shown in Table 6.2, the fouling resistant element FILMTEC™ FORTILIFE™ CR100 has several strengths, combining the design of the elements, reduced pressure loss between feed and concentrate that directly leads to a reduced energy consumption. The specific element design also leads to lower growth rate of fouling and consequently reduce the frequency cleaning needs.

Table 6.2 Overview of SWOT analysis of fouling resistant RO elements

INTERNAL	<ul style="list-style-type: none"> • Reduced number of cleanings required • Lower feed-concentrate pressure drop • Less fouling rate • Higher energy efficiency 	<ul style="list-style-type: none"> • Reduces fouling rates but does not prevent any kind of fouling and scaling, (which means that cleaning is reduced but not eliminated)
	<div style="display: flex; justify-content: space-around; font-size: 2em; font-weight: bold;"> S W </div> <hr style="border: 1px solid black;"/> <div style="display: flex; justify-content: space-around; font-size: 2em; font-weight: bold;"> O T </div>	
EXTERNAL	<ul style="list-style-type: none"> • Among the market leaders for resistance against fouling • Application range for streams with high fouling potential, especially water reuse 	<ul style="list-style-type: none"> • Uncertainty on how customer will accept the new product • Competitors by new developments



6.2.2 Key performance indicators of fouling resistance reverse osmosis elements

For demonstration and monitoring of the performance of the technology, three main key performance indicators can be considered. These parameters are shown in Table 6.2. These three parameters are significantly lower with the fouling resistant element in comparison to conventional reverse osmosis elements, but also depend on the feed water characteristics (see examples in Figure 6.5 and 6.6).

Table 6.2 Overview of key performance indicators for demonstrating and monitoring the performance of the fouling resistant reverse osmosis element

KPI description	Unit	KPI
Cleaning frequency	Cleanings per year	Lower than conventional products
Pressure drop feed-concentrate	Bar	Lower than conventional products
Energy consumption	kWh/m ³ permeate	Lower than conventional products

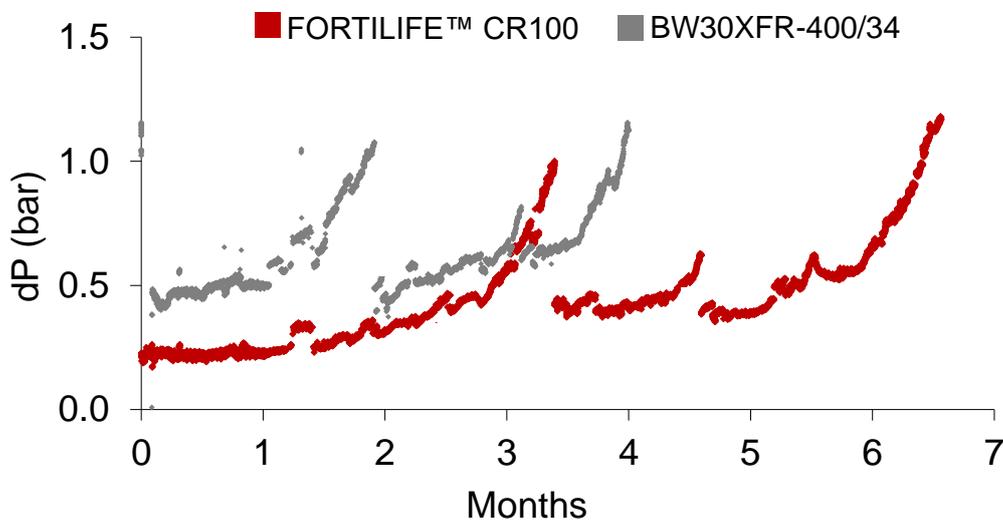


Figure 6.5 Example 1 – Waste Water Case Study - Pressure drop (dp) progress over time on FILMTEC™ BW30XFR and FILMTEC™ FORTILIFE™ CR100 showing lower cleaning needs¹

¹ Vila et al. (2018) How to Reduce the Cleaning Frequency in Water Reuse: Case Studies

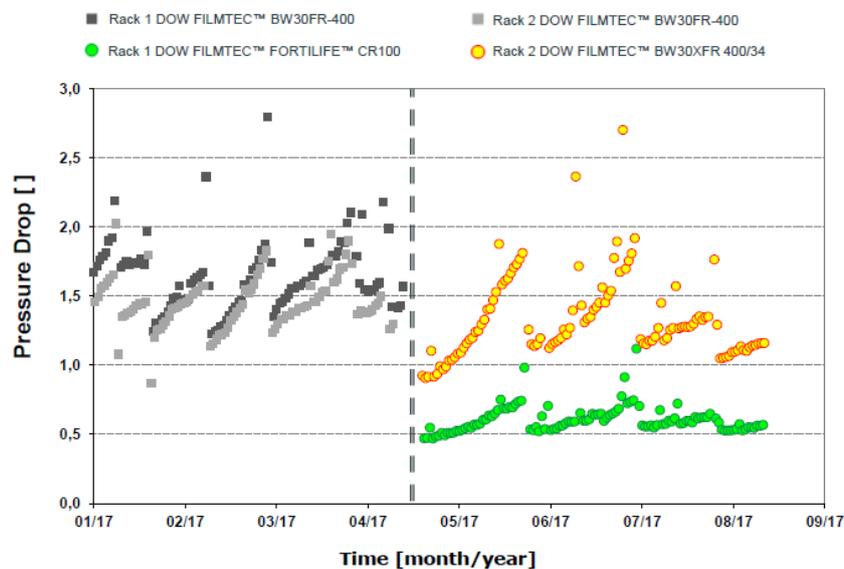


Figure 6.6 Example 2 – River Water Case Study - Pressure drop (dp) progress over time on FILMTEC™ BW30XFR and FILMTEC™ FORTILIFE™ CR100 showing lower energy consumption²

6.3 Market perspective

6.3.1 Market overview - Opportunities and threats of fouling resistant reverse osmosis elements

Reverse osmosis (RO) treatment technology has become the gold standard for industrial water users. It provides a low-cost way to produce high-purity water, which is used in boilers, cooling towers and industrial reactions. Years ago, water demand was typically met by treating groundwater or surface waters with low contamination and total dissolved solids (TDS). In light of declining supply, industrial users are increasingly using local wastewater sources and recycling their own process wastewater for operational reuse. These waters contain higher contamination and TDS levels, which can cause sub-optimal RO system operations. RO membranes designed for treating challenging water are necessary for industrial plants to remain efficient and cost-effective.³

Years of research by Filmtec, studying the fouling nature of reverse osmosis element, have provided generational advances in fouling resistant products. Each advance has combined optimized fouling resistant membrane chemistry and module designs. This has translated into

² Vila et al. (2018) How to Reduce the Cleaning Frequency in Water Reuse: Case Studies

³ Arrowood et al. (2017) <https://www.watertechnology.com/fouling-resistant-ro-membranes/>



the development of FILMTEC™ BW30XFR-400/34 RO elements in 2010, and FILMTEC™ FORTILIFE™ CR100 RO elements in 2016.

The FILMTEC™ BW30XFR-400/34 element are ideal for reverse osmosis plant managers and operators dealing with challenging waters and wastewaters and seeking consistent high performance, long element life, increased productivity and higher water quality coupled with outstanding fouling resistance.⁴

The FILMTEC™ FORTILIFE™ CR100 element is one of the industry's most advanced fouling resistant element, specially designed to provide relief from biological fouling. The element's ultralow differential pressure provides improved hydraulic balance (more even distribution of flux across all the elements in the system) in a biological fouling environment such as wastewater treatment. This product also boasts a reliable and durable membrane chemistry, providing organic fouling resistance, cleanability, low energy operation, and excellent solute rejection.⁵

6.3.2 Economical evaluation

Comparing the performance of FILMTEC™ BW30XFR-400/34 elements to FILMTEC™ FORTILIFE™ CR100 RO elements show that the use of FILMTEC™ FORTILIFE™ CR100 leads, on the basis of the operating conditions described in the case studies above (wastewater and river water treatment), to:

- a reduction in cleaning frequency due to biofouling of 40% per year
- an increase of 66% of operating time due to lower frequency needs, and increment of the system recovery
- energy savings due to the 53% lower pressure drop achieved
- all the above can eventually lead to longer life-time of the membranes due to lower chemical exposure

NOTE: The results included in this summary may vary based on different operating conditions

6.4 Roadmap

6.4.1 Current technology readiness level

Years of research studying the fouling nature of reverse osmosis elements has provided generational advances in fouling resistant products, as commented before.

⁴ DOW (2015) FILMTEC™ BW30XFR-400/34i Element, Product data sheet, Form No. 609-02170, Rev. 2

⁵ DOW (2016) FILMTEC™ FORTILIFE™ CR100, Product data sheet, Form No. 609-50243 Rev 2



6.4.2 Barriers and challenges for implementation

Organic fouling and biofouling in reverse osmosis (RO) water treatment occurs when the feed channel in the RO module is blocked partially or fully by bacteria-produced biofilm. This can cause the pressure drop (dP) across the RO module to increase, leading to hydraulic imbalance and possibly damage the module. Moreover, biofilms can affect membrane transport properties and create a drop in trans-membrane pressure (TMP) which lowers the flux. Each of these effects increases the energy of operation but also leads to frequent cleanings (CIP) to regain membrane performance. In total, biofouling affects energy consumption, membrane lifetime, water productivity and cost of water produced.⁶

6.4.3 Actions for implementation

- New product development (process optimization)
- Piloting with prototypes
- Piloting with commercial products to generate case studies prior to the product launch
- Business plan ready for product launch
- Congresses participation to accelerate the product launch and promotion
- Customer testimonials, case studies and reference list update to show and convince the market of the benefits of the new product

6.5 Conclusion

Challenging feed water conditions and severe fouling environments may drive the reverse osmosis systems to long down-time operations, and increase the total cost of produced water. FILMTEC™ FORTILIFE™ family products may help the systems to (in comparison with conventional FILMTEC™ BW30XFR-400/34 product):

- Reduce the cleaning frequency due to biofouling of 40% per year
- Increase of 66% of operating time due to lower frequency needs, and increment of the system recovery
- Energy savings due to the 53% lower pressure drop achieved
- Longer life-time of the membranes due to lower chemical exposure

⁶ Matina et al. (2011) Biofouling in reverse osmosis membranes for seawater desalination

Table 6.4 Business model CANVAS – Fouling resistant reverse osmosis elements

<p>Key partners</p> <ul style="list-style-type: none"> OEMs Water and wastewater treatment plant operators <p>Motivations: efficient production of high quality product water with high process stability</p>	<p>Key activities</p> <ul style="list-style-type: none"> Understanding industrial market needs (survey, Voice of Customer (VOC) etc.) Develop a product that meets customer expectations and needs (fouling-resistant properties) 	<p>Value proposition</p> <ul style="list-style-type: none"> Providing membranes that are able to provide sufficient product water quality in a reliable way Fouling resistant membranes High process efficiency Low energy consumption Low cleaning requirements High membrane life time 	<p>Customer relationship</p> <ul style="list-style-type: none"> Reliable provider of high quality membranes Technical service and support to ensure high performance of membrane elements 	<p>Customer segments</p> <ul style="list-style-type: none"> Municipal wastewater treatment Industrial wastewater treatment Municipal water use as drinking water Seawater desalination Water reuse Wastewater treatment Industrial water treatment, e.g. food and beverage Oil and gas industry
<p>Key resources</p> <ul style="list-style-type: none"> Know how membrane and water High quality and reliable manufacturing R&D for new generation products 		<p>Channels</p> <ul style="list-style-type: none"> OEM's loyalty Conferences and seminars Distributors Direct contact with end-users (plants) 		
<p>Cost structure</p> <ul style="list-style-type: none"> Raw materials Production costs (energy cost...) 			<p>Revenue Streams</p> <ul style="list-style-type: none"> Performance, reliability and technical service 	

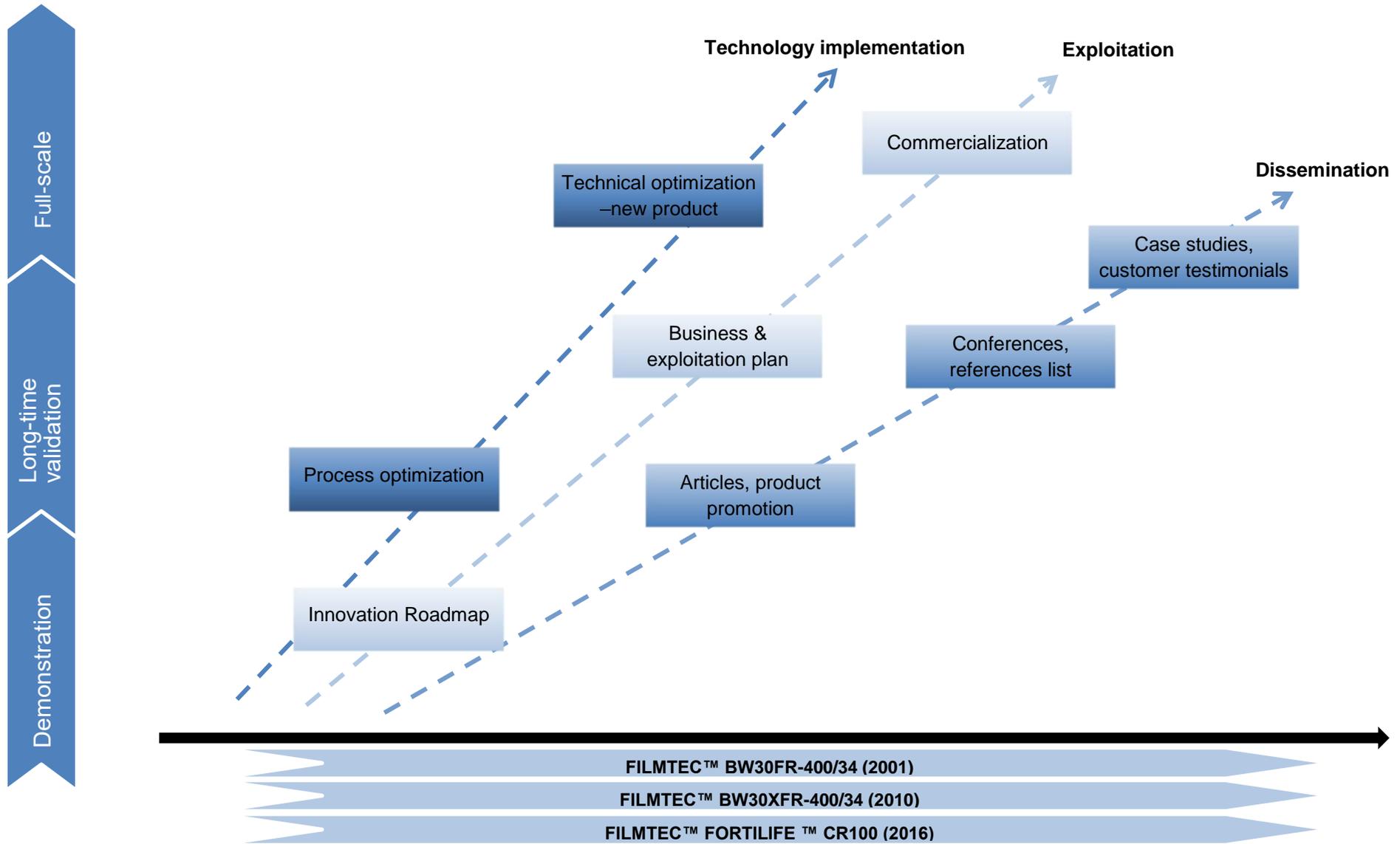


Figure 6.7 Innovation Roadmap of fouling resistant RO membranes