Sustainable industrial wastewater reuse using ceramic nanofiltration: results from two pilot projects in the oil and gas and the ceramics industries

Matan Beery, Christian Pflieger and Marcus Weyd

ABSTRACT

The federal research project, PAkmem, deals with the recycling of industrial wastewater. The aim of the project is to develop and pilot an innovative integrative process for produced water treatment in the oil and gas industry utilizing flotation and ceramic micro-nano-membrane filtrations (MF-NF membranes) as well as the wastewater treatment of the ceramic industry with ceramic NF-membranes and electrodialysis (ED). The process utilized should remove fine particles, organic matter and divalent ions in order to make the water dischargeable or reusable (direct disposal or reuse as process water in the ceramic industry and the enhanced oil recovery reinjection in the oil and gas industry in which the water is conditioned in order to increase the oil production yield). Three pilot plants were designed and built according to strict safety standards and were operated on industrial manufacturing sites in Germany in 2019. Two innovative optical fine particle measuring techniques (inline and online) have been specially adapted for the project and integrated into the pilot plants. The results show promising technical potential for the use of ceramic membranes in the above-mentioned applications.

Key words | ceramic membranes, flotation, nanofiltration, produced water

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HIGHLIGHTS

- Produced water treatment using ceramic membranes shows promising results in removing both oils and solids.
- Economics of flotation in combination with ceramic membrane filtration prove more advantageous than external disposal.
- MF, ED and RO remove COD, suspended solids and conductivity from industrial ceramic wastewaters.

INTRODUCTION

With the UN's sustainable development goals in mind, increasing water availability worldwide, combined with the search for constant industrial improvement, increasing efficiency and conserving resources, are all on the top of the agenda of the global water industry. Industrial wastewater especially poses major challenges for the relevant stakeholder groups. Due to their great complexity and diversity, the treatment and/or recycling of such waters is often associated with high costs, high energy consumptions and non-trivial safety issues. One of the major obstacles are organic and inorganic contaminants of loads and kinds that are rarely seen in municipal or agricultural waters. Industries with a particularly high water consumption, such as the oil

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and gas industry, are more of a subject to scrutiny than others.

The federal grant-financed R&D project PAkmem was launched in late 2016 with the goal of developing and piloting multi-stage separation processes for the treatment of industrial wastewaters with the goal of recycle or reuse in mind. The processes are based around ceramic nanofiltration with state-of-the-art membranes developed and produced in Germany. Additional pre-treatment by means of induced gas flotation and ceramic microfiltration, as well as post-treatment by means of reverse osmosis or electrodialysis, were tested in conjunction with novel optical measurement methods for water quality (Figure 1). A total of nine project partners comprising representatives of different stakeholders in the value chain, including research institutes, small and large companies, have joined the national consortium. The project was financed by the German Ministry of Education and Research (BMBF) as part of the WavE-initiative and concluded in March 2020.

Produced water in the oil and gas industry

When oil is extracted from the ground, water and gases are also brought to the surface in addition to the hydrocarbons. The so-called water cut of this three-phase mixture can be as high as 95% by weight. This necessitates the further processing of the extracted oil using different fluid separation technologies. After the oil and gas have been separated, the remaining water that is usually reinjected into the reservoir in several spots around the production well maintains the pressure underground and continues to push out more hydrocarbons to the surface, in a process known as water flooding. The injected water often still has considerable amounts of hydrocarbons and dissolved salts. Fine suspended solids are typically also present, which entails a risk of the fine formation pores becoming blocked. The remaining hydrocarbons inside the produced water also reduce the overall oil yield as some of the product (oil) is simply being put back in the ground. Since the produced water does not belong to the natural water cycle reservoirs (the earth layers are typically much deeper than the ones where ground water is found) there are often no or very lax water quality values for reinjection into the reservoir water. Nonetheless, when the reinjection is done for disposal purposes, the regulation becomes stricter and less favorable for the producers and requires more advanced treatment techniques (Fink 2012). In the old onshore oil fields of Germany, the typical treatment chain of the produced water–hydrocarbon mixture will only include three phase gravity separators followed by large settling tanks with sufficient residence time before most of the water is re-injected into the ground. However, due to old infrastructure and pipelines, as well as varying oil-water composition, the actual water quality at the reinjection spot could be of poor quality, containing too many hydrocarbons or fine solids. A better solution, such as one employing ceramic membranes (Ebrahimi *et al.* 2008, 2010; Pedenaud *et al.* 2011) for this wastewater reuse scheme is therefore needed to increase productivity and reduce the water footprint.

In the context of enhanced oil recovery (EOR), partial demineralization of the reinjection water by means of nanofiltration for divalent ion removal was also set out to be investigated in the project. Oil molecules bind to rock particles in the deposit by bridging divalent ions. Removing at least some of these ions (especially Mg^{2+} and Ca^{2+}) by means of nanofiltration can lead to relaxation of the

Figure 1 | The integrated process steps being inspected in the PAkmem project (*sources*: Fraunhofer IKTS, akvola, LUM, SOPAT).

ion-bridges, expanding the electrical double layer and enabling the bridges to be replaced by non-bridging monovalent ions, which in turn can mobilize more oil molecules and increase the overall production yield (Robbana *et al.* 2012; Mainwaring 2015). Additionally, the use of nanofiltration for produced water treatment can avoid blocking of the porous oil formation upon injection and potentially inhibit corrosion and biofouling on the internal wall of the injection piping (Gamal Khedr 2015). These additional benefits in yield and productivity make the process at hand economically interesting for the oil industry and create an additional incentive to the environmental one.

Ceramics industry

Similar separation challenges come about in the treatment of wastewater from the ceramics industry. Ceramic raw materials such as Al_2O_3, SiO_2 or TiO_2, as well as the organic ingredients used in the processing of the ceramic bodies and fluorescent organics used for quality assurance, are typically the main components in these wastewaters.

Classical wastewater treatment processes for the removal of fine inorganic suspended solids such as ceramic particles, glazes and various salts by physical-chemical processes (flocculation/precipitation, sedimentation, flotation, dewatering in filter press) usually require the addition of conditioning chemicals to ensure adequate separation of the inorganic (fine) fraction. Besides the additional components, all the substances contained in the wastewater are mixed unselectively with the minerals in the mass produced. The recycling of such materials has so far not seen general acceptance in the industry. Furthermore, the processes used to date do not usually provide adequate water quality that can be reused in some way in the plant (e.g. process or washing waters). In particular, the remaining COD and salt amounts are most problematic. This conventional cleaning must be upgraded through some demineralization technique if the water cycle is to be closed or wastewaters reused. The investigation of polymeric nanofiltration membranes for this purpose is documented in the literature (Moliner Salvador *et al.* 2012). Besides the use of ceramic nanofiltration for the upgrade of such wastewater treatment processes, it was also decided to evaluate the use of electrodialysis with bipolar membranes in the process as introduced by Fraunhofer IKTS in Dresden (Friedrich 2015). The bipolar membranes used consist of cation and anion exchange membranes and a catalytic intermediate layer to accelerate water dissociation in between. By applying an electric field, chloride and sulfate ions migrate through the anion exchange membrane into the anolyte stream, are retained on the cation-selective side of the bipolar membrane and form hydrochloric and sulphuric acid together with the protons produced in the bipolar membrane. In the same way, the anion-selective side of the bipolar membrane produces a sodium hydroxide solution and a desalinated diluate is formed in the inlet chamber.

METHODS

Oil and gas industry

The treatment of the produced water from the E&P site Barnstorf in Lower Saxony (Figure 2) by means of flotation and microfiltration followed by nanofiltration was first

Figure 2 | Main treatment facility of the oil field Barnstorf in Lower saxony, Germany (*source*: Google, Wintershall Dea).

Table 1 | Typical water composition of the produced water on the feed to the pilot

Sampling location	**Reinjection water Barnstorf**	**Unit**
Date	05.11.2018	
Density at 20 °C	1.1213	g/cm^3
Max temperature	35	°C
pH	6.1	
Calculated total hardness	2,451	°dH
Iron	78	mg/L
Manganese	5.6	mg/L
Aluminium	<1	mg/L
Calcium	13,500	mg/L
Strontium	943	mg/L
Barium	91	mg/L
Magnesium	2,160	mg/L
Sodium	49,800	mg/L
Potassium	337	mg/L
Lithium	5	mg/L
Ammonium	113	mg/L
Zinc	<0.5	mg/L
Lead	<0.5	mg/L
Chloride	107,500	mg/L
Bromide	732	mg/L
Iodide	13.2	mg/L
Nitrate	1.8	mg/L
Bicarbonate	146	mg/L
Borate	166	mg/L
Phosphate	<2	mg/L

tested on a laboratory scale. The flotation-microfiltration process, akvoFloat®, can remove suspended solids and hydrocarbons from the produced water and is described in greater detail elsewhere (Ludwig *et al.* 2015). The treated produced water came from the primary separation treatment in-site following a three-phase-separator and sedimentation tanks to remove most of the gases, solids, and crude oil.

Based on water analysis tests, the expected feed water quality and composition is shown in Tables 1 and 2. The produced water is highly saline, having especially high mass concentrations of iron, calcium, magnesium, sodium and chloride. As seen in Table 2, an oil content of approximately 95 mg/L and a solids content of approximately 73 mg/L (mostly sand particles) were to be expected in the raw wastewater.

The concentration of particles and organic load, especially hydrocarbons, was to be reduced in the project using induced gas flotation and ceramic microfiltration. The generated permeate should have a maximum oil content of 25 mg/L according to DIN ISO 9377-2 (H53 method) and a particle size proportion of less than 1% for particles larger than 3 μm (according to the internal specifications of Wintershall DEA for reinjection). An analysis of the organic load based on HS gas chromatography has shown it to be composed of 13% BTX, 0.23% PAH and 86.75% other hydrocarbons.

The samples collected at the beginning of the project were put to a lab flotation-filtration test using ceramic microfiltration membranes. The oil content could be consistently reduced by more than 95% but the solids contents were reduced by only 25%, the reason being that the produced water, when coming into contact with oxygen, immediately became turbid orange, indicating iron oxidation and precipitation. This phenomenon was well documented by the oil field services team on-site, deeming it critical for the actual treatment in the pilot plant (and later in the commercial application) to be oxygen-free for more than just the common explosive safety reasons.

The samples were then filtered by tubular ceramic nanofiltration membranes (Figure 3) having a molecular weight cutoff (MWCO) of 200 Da. These multicoated membranes are based on previous developments (Puhlfürß *et al.* 2000; Voigt *et al.* 2013; Pflieger *et al.* 2018) and have shown the

Table 2 | Expected feed water quality in terms of oil and solids content

			Particle size (μm)					
	Oil content (mg/L)	**Total solids (mg/L)**	**2**	**5**	**15**	**25**	**50**	**100**
Mean	94.4	72.6	957.436	627.958	44.607	8.101	430	40
Standard deviation	40.8	149.1	53.520	66.499	32.133	7.467	313	28

Figure 3 | Ceramic tubular NF membranes used in the pilots (*source*: IKTS, Rauschert).

removal of up to 90% of divalent ions from saline solutions in lab tests.

According to the promising laboratory results, a pilot project was carried out at Wintershall DEA in Barnstorf (Bockstedt) over a period of 23 weeks. The pilot plant was built according to strict industrial safety regulations and was operated in an oxygen-free environment with the flotation operated with nitrogen in a stainless steel sealed tank with a positive over-pressure including an off-gas activated carbon treatment and all equipment being inertized before coming into contact with the liquid medium. The produced water was taken via a bypass from the reinjection line and was fed into the pilot plant. The cleaned water (permeate) was returned to the reinjection line with the possibility of bypassing the NF while sludge and retentate were collected and disposed of in separate IBCs (see the pilot plant's process flowchart including the corresponding volumetric flow rate ranges and the detailed process and instrumentation diagram in Figure 4).

The raw wastewater (produced water post primary treatment) had a pre-pressure of approximately 11–12 bar. Both for safety and operational reasons, the pressure was reduced to 1 bar using the pressure reducer PC-10/V-107. In the event of a technical malfunction of the pressure reducer, a safety shut-off valve was installed to go off above 4.5 bar. The downstream pneumatic valve V-102 was used to regulate the level of the tank T-1 of the flotation-MF system, but also to shut the feed line in case the maximum level in the tank was reached (measured by the level sensor LS 01). The chemical tanks T-12 and T-13 with the dosing pumps P-12 and P-13 made it possible to add pre-treatment chemicals such as acids, coagulants and emulsion breakers into the process, if necessary. A static mixer was installed for better mixing of these chemicals. The first treatment step using flotation occurred in the T-1 tank using Micro-Gas, an induced gas fine bubbler generation device. Nitrogen was used as the flotating gas to avoid the previously mentioned problem of Fe^{2+} ions oxidizing to Fe^{3+} ions resulting in the precipitation of iron (III) hydroxide complexes. In the next treatment step, the wastewater was filtered under negative pressure through the submerged flat sheet ceramic microfiltration membranes (SiC, 0.1 μm) that were integrated into the flotation tank, T-1. For this purpose, the water was sucked through the membranes by the pump P-21 and pumped through the permeate line, feeding the backwash tank, T-21, and the NF feed tank, B-51. The pump P-22, in addition to the pumps P-23 and P-24, were periodically used to backwash the MF membranes with or without chemicals stored in T-23 and T-24. As previously mentioned, a bypass was placed in the permeate line for direct integration into the reinjection line. An on-line particle characterization was carried out both before and after the NF system. The NF was composed of three stainless steel pressure vessels holding the membrane modules with a surface area of 1.2 m^2 each. Pump P-01 was the main feed pump building-up the pressure (up to 20 bar) with P-02 serving as a circulation pump assuring the most efficient crossflow regime in every module. The permeate produced by the NF was fed into the tank B-52 and could then be pumped through the pump P-04 into the reinjection line or used with the pump P-03 to backwash the nanofiltration membranes. The sludge and drainage (emptying) of the flotation-MF system, as well as the retentate of the nanofiltration, were collected as waste in the two IBCs T-25 and T-26 and transported to a nearby disposal facility. A comprehensive HAZOP analysis was executed before starting up the pilot plant.

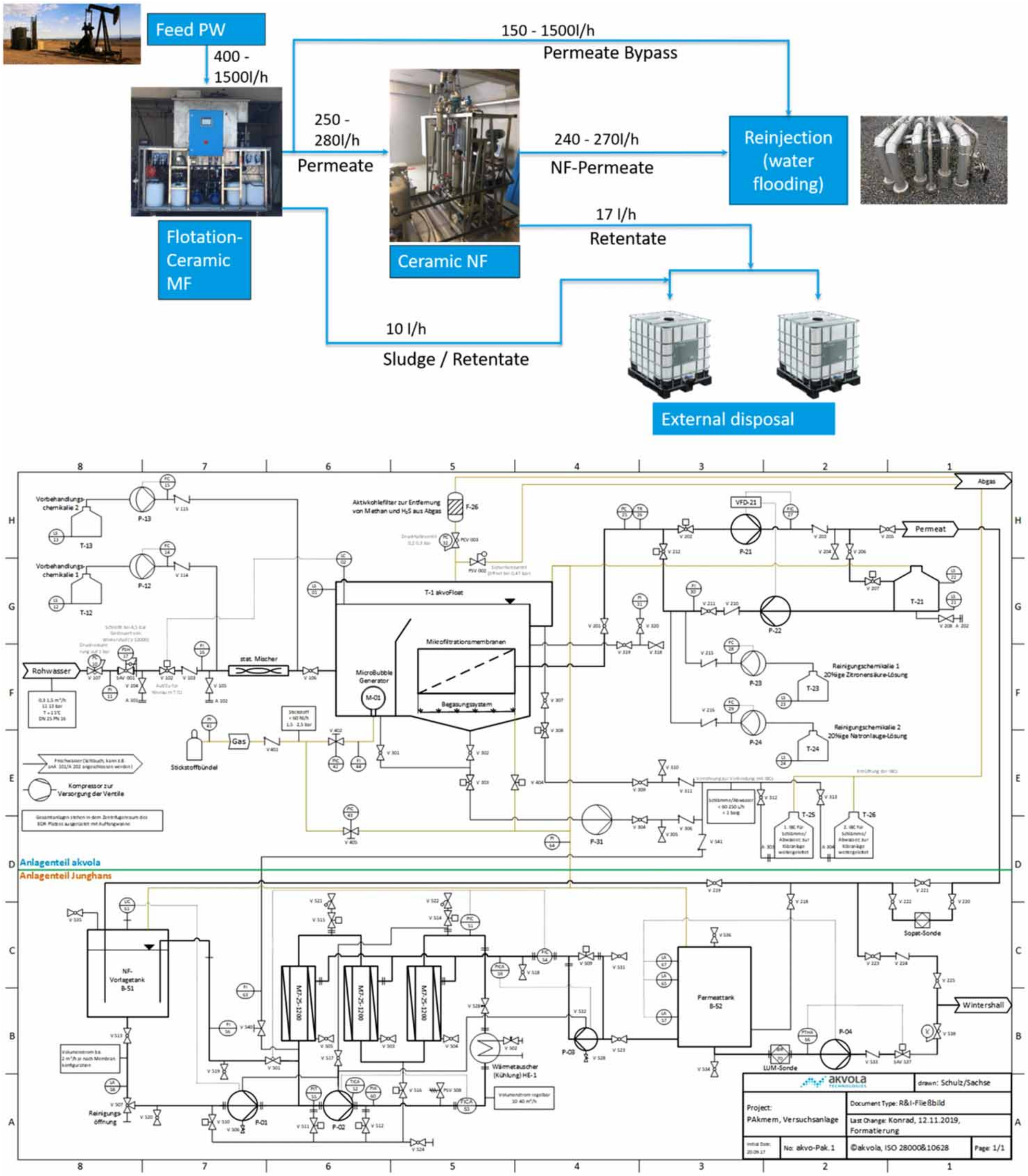

Figure 4 | Flowchart (top) and P&ID (bottom) of the produced water pilot plant (*source*: akvola, Junghans).

Ceramics industry

The central concern of the industrial partner in the ceramic industry is the treatment of industrial water to replace the use of municipal drinking water in production. Solids must be completely removed (also important for the electro-membrane process technology) and the conductivity of the water thus obtained should be in the order of 250 μS/cm. Higher and lower conductivities are unfavorable as they will inevitably lead to changes in the design and operation parameters of currently installed unit operations in the process line.

Preliminary tests on single-channel tubular geometries and selected 19-channel tubular geometries were promising, but the target value for conductivity is challengingly low. At Fraunhofer IKTS, a plant was set up for the on-site tests. Figure 5 shows the pilot plant consisting of: (1) ceramic MF/UF/NF pressurized tubular membrane system (partially automated, automatic filling device), (2) backwash system (operating mode: pulse or volume method), (3) industrial cooler (approximately. 5 KW), (4) buffer tank (180 L) and (5) RO system (brackish water spiral wound module).

For the first on-site tests, 19-channel tube geometries with the lowest cut-off, i.e. the smallest pore size and highest possible selectivity, were used. These initially did not yield a sufficient reduction in conductivity. Permeates produced in this way were therefore further processed by a brackish water reverse osmosis system (BWRO). The final waters produced in this way had, however, too low conductivities. In the pilot phase, process water was therefore produced for preparation tests by blending NF and RO permeates.

The on-site tests were also used to produce test water for use with electrodialysis (ED). The permeate water of the NF stage has already been successfully treated with ED in advance. For the extended test series on ED, two new ED stacks, each with 10 membrane pairs and with a membrane area of approximately 2 m^2 each, were designed for the pilot tests during the period under investigation (Figure 6).

RESULTS AND DISCUSSION

Oil and gas industry

The akvoFloat® flotation-MF system was initially operated without the following NF and the permeate produced was pumped directly into the reinjection system. This gave the opportunity to test the plant at higher fluxes and to evaluate the operating behavior. Extensive jar tests on-site as well as during the first live pilot runs with coagulants and emulsions breakers did not show any improvements and so the further use of pre-treatment chemicals was deemed as unnecessary for the rest of the pilot. In total, after the start-up phase the pilot plant was running for a period of 13 weeks while constantly going through operational optimization to maintain stable operation at the highest possible fluxes. During that time, the feed water quality of the produced water changed frequently in composition. Figure 7 shows the visual

Figure 5 | NF pilot plant used in the ceramics industry.

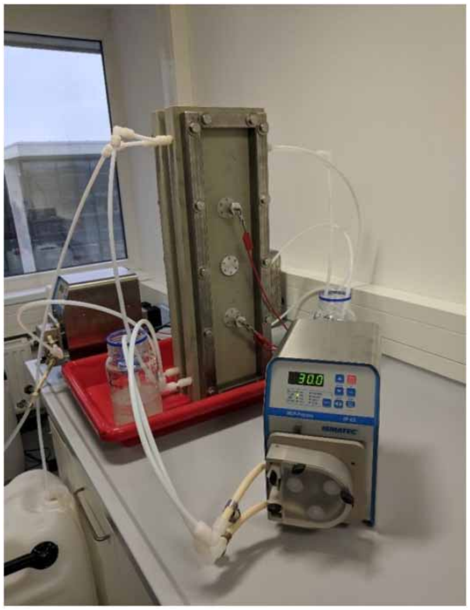

Figure 6 | The ED system used for treatment of the membrane permeates out of the pilot in the ceramic industry (*source*: Friedrich, IKTS).

Figure 7 | Samples taken from the feed to the plant (left bottles, produced water after primary treatment) and post flotation-MF (right bottles, always clear water). The two images were taken on two consecutive days.

difference in feed water quality on two consecutive days in September 2019 and the constantly high quality permeate produced by the flotation-MF system despite the changing feed water quality. Figure 8 shows a typical development of the trans-membrane pressure-loss (TMP, bar) over time (hours) with two constant filtration fluxes used at 125 and 66 L/m^2/h ('lmh'). The peaks in the diagram depict backwash events with the red lines showing chemically enhanced backwashes (CEB) or cleaning in place (CIP) events performed in the 42-hour period. A recovery of 98% could be reached. The crossflow nanofiltration system finally reached stable operation at constant pressures of 20 bar with hourly backwashes (Figure 9).

Samples of the feed and the permeate were occasionally taken for lab analysis. The hydrocarbons and suspended solids were efficiently reduced by the integrated process to below detection levels utilized by the analytical methods [DIN EN ISO 9377-2 : 2001-07] and [DIN EN 872 : 2005-04] accordingly. The divalent ions detected by atomic emission spectroscopy and ion-exchange chromatography could only be reduced to some extent with the best lab removals listed in Table 3. On most occasions, however, only lower or even zero ion removal by the NF membrane was detected, suggesting membrane defects.

Cost analysis

Based on the data collected during the pilot phase, a business case calculation was performed for a site in which the current reinjection system is not able to treat the full capacity of the produced water being generated and about two-thirds of the water is currently being externally disposed of at 80 €/m^3. The over-capacity should be treated by a new integrated flotation-ceramic membrane filtration plant (MF + NF/RO) with a plant capacity of 46,667 m^3/year, operating on 50 weeks/year and 7 days/week resulting in 133.33 m^3/d or (at 24 h/d) a nominal capacity of 5.56 m^3/h. For the sake of simplification, a new plant capacity of 6 m^3/h was considered. Assuming the membrane concentrate would still have to be externally disposed of (at 600 €/m^3) and the permeate could be indirectly discharged into the local sewage system (at 3 €/m^3), the overall yearly OPEX savings of €568 k could pay back the estimated CAPEX investment of €1.2M in just over two years (see Table 4 for details). It is worth pointing out that such a business case is only economically attractive if the alternatives are too expensive (as in the case of external disposal) or if the environmental regulator forces such a complex treatment scheme.

Ceramics industry

On average, approximately 14% conductivity reduction could be achieved for the ceramic NF stage (specific permeate flow in the range of 8 L/(m^2 h bar)). Further

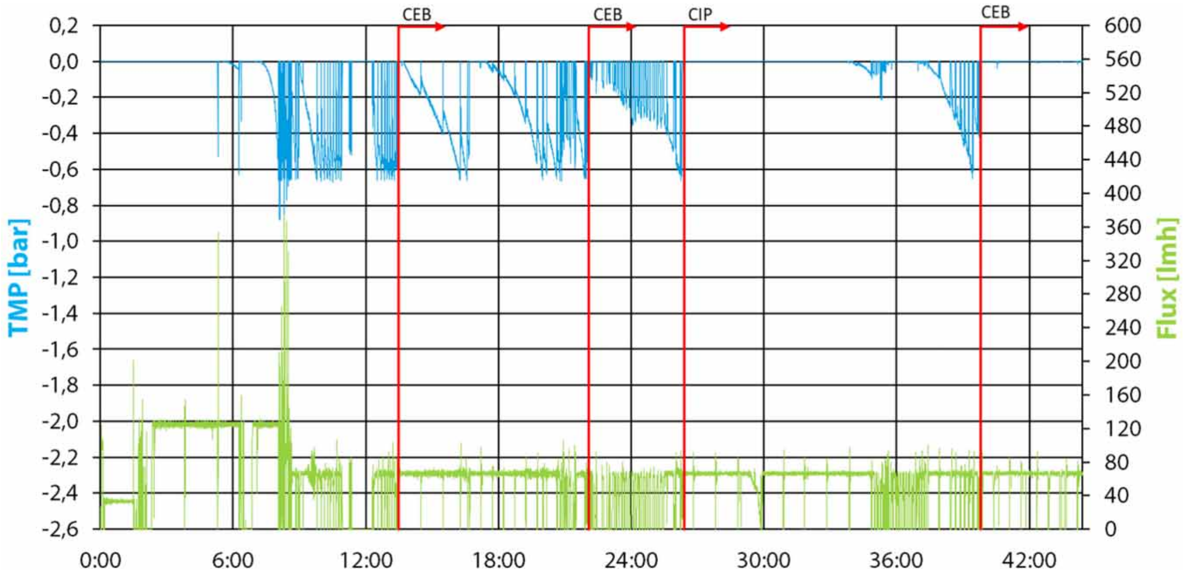

Figure 8 | TMP-flux diagram for the microfiltration system. Please refer to the online version of this paper to see this figure in color: http://dx.doi.org/10.2166/wrd.2020.029.

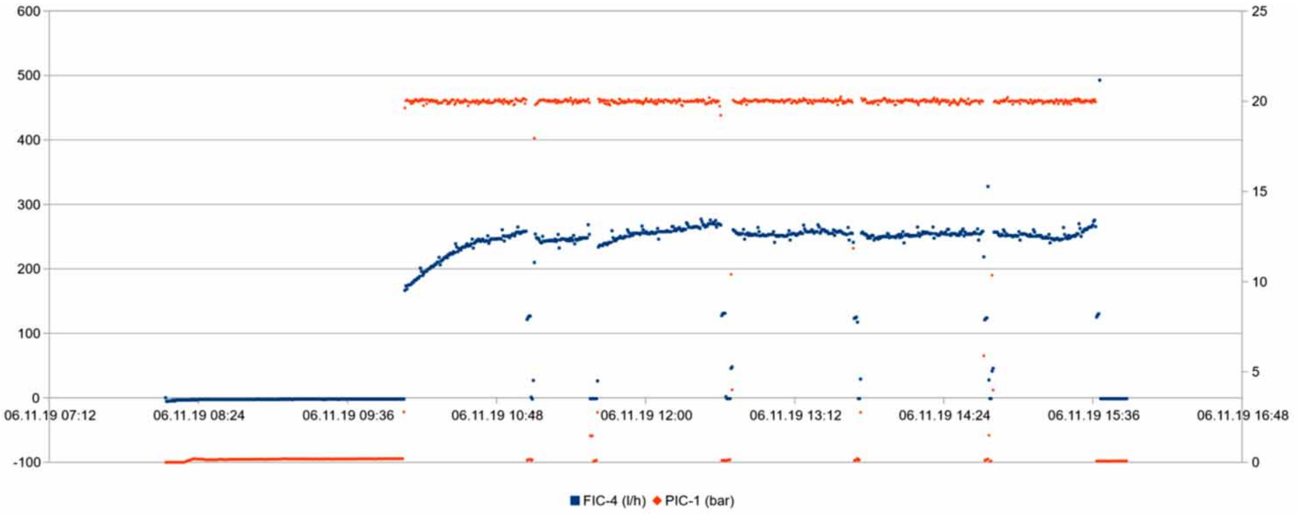

Figure 9 | Flow (L/h, left axis) – Pressure (bar, right axis) diagram for the nanofiltration system.

Table 3 | Outtake of water analysis results from the produced water membrane filtration system

Parameter	Value in feed (mg/L)	Value in permeate (mg/L)	Removal efficiency
Hydrocarbons (C10-C40)	<30	<0.1	>99%
Total solids	>20	n.d.	>99%
Ca^{2+}	13,700	11,700	14.6%
Mg^{2+}	2,290	1,340	42.5%
SO_4^{2-}	117	35	70.1%

processing by means of RO showed retention in the range of 98% (related to the initial conductivity of the NF permeate). By mixing permeates, the industrial partner, Duravit, was provided with treated wastewater with a conductivity of 243 μS/cm that could be re-used in the industrial processes. As filtration tests were carried out, a new batch of prototype membranes was manufactured and tested at Duravit. In comparison, the new batch delivered higher specific permeate flows in the range of

Table 4 | Business case calculation for a new flotation-ceramic membrane filtration (CMF) plant as an alternative to external disposal of produced water

Current situation

External disposal

	Description	**m^3/y**	**€/m^3**	**€/y**
1	Produced water total	70,000	0€	0€
2	Produced water being externally disposed	46,667	80€	3,733,333€
	Tot (€/y)			3,733,333€
OPEX				
General				
	Produced water to be treated	46,667	m^3/y	
	Recovery	90,0%		
Flotation + CMF plant				
		kWh/m^3	€/m^3	€/y
1	Energy + compressed air (@0.2€/kWh)	5.0	1.0€	46,667€
		m^3/y	€/m^3	€/y
2	Cleaning	46,667	0.3€	14,000€
3	Chemicals	46,667	3.0€	140,000€
4	Concentrate disposal	4,667	600€	2,800,000€
5	Maintenance			
6	Indirect discharge fees	42,000	3.0€	126,000€
		h/week	€/h	€/y
7	Labor	40	20€	38,400€
Tot. OPEX Flotation + CMF (€/y)				3,165,067€
Total costs calculation				
Running costs				
	Description	Current situation: external disposal		New situation: flotation+ CMF
1	External disposal	3,733,333 €		2,800,000 €
2	Operation (energy, cleaning, labor, etc.)			365,067 €
	OPEX (€/y)	3,733,333 €		3,165,067 €
Investment costs				
				New situation: flotation+ CMF
1	Flotation + CMF plant			1,200,000€
	CAPEX (€)			1,200,000€
Summary				
OPEX savings (€/y)				568,267€
Payback time (y): CAPEX/(OPEX savings)				2.1

12 L/(m^2 h bar) and achieved a conductivity removal of 10%. The permeates obtained in this way were also made available to Duravit for testing as batch water. As far as the COD removal goes, the only way to achieve stable operation and high removal efficiency was by combining the NF filtration with MF pre-treatment. The

results showed a reduction from COD levels of 950 to <50 mg/L. Both conductivity and COD removal are depicted in Figure 10.

During on-site tests, various NF and MF permeates were collected for use in the testing of the of ED. Around 800 L of the filtrate produced in this way (0.85 mS/cm) was treated by electrodialysis by IKTS and treated waters with three different conductivities (0.32, 0.38 and 0.42 mS/cm) were produced (see Figure 11). Duravit was supplied with 50 L of each of these for preparation trials. For the extended series of tests in the field of electro-membrane process engineering, no further optimization was necessary of the ED module. However, the membrane area was doubled to 10 membrane pairs and the throughput increased as a result.

CONCLUSIONS

The pilot tests showed that an integrated membrane system containing ceramic nanofiltration membranes could

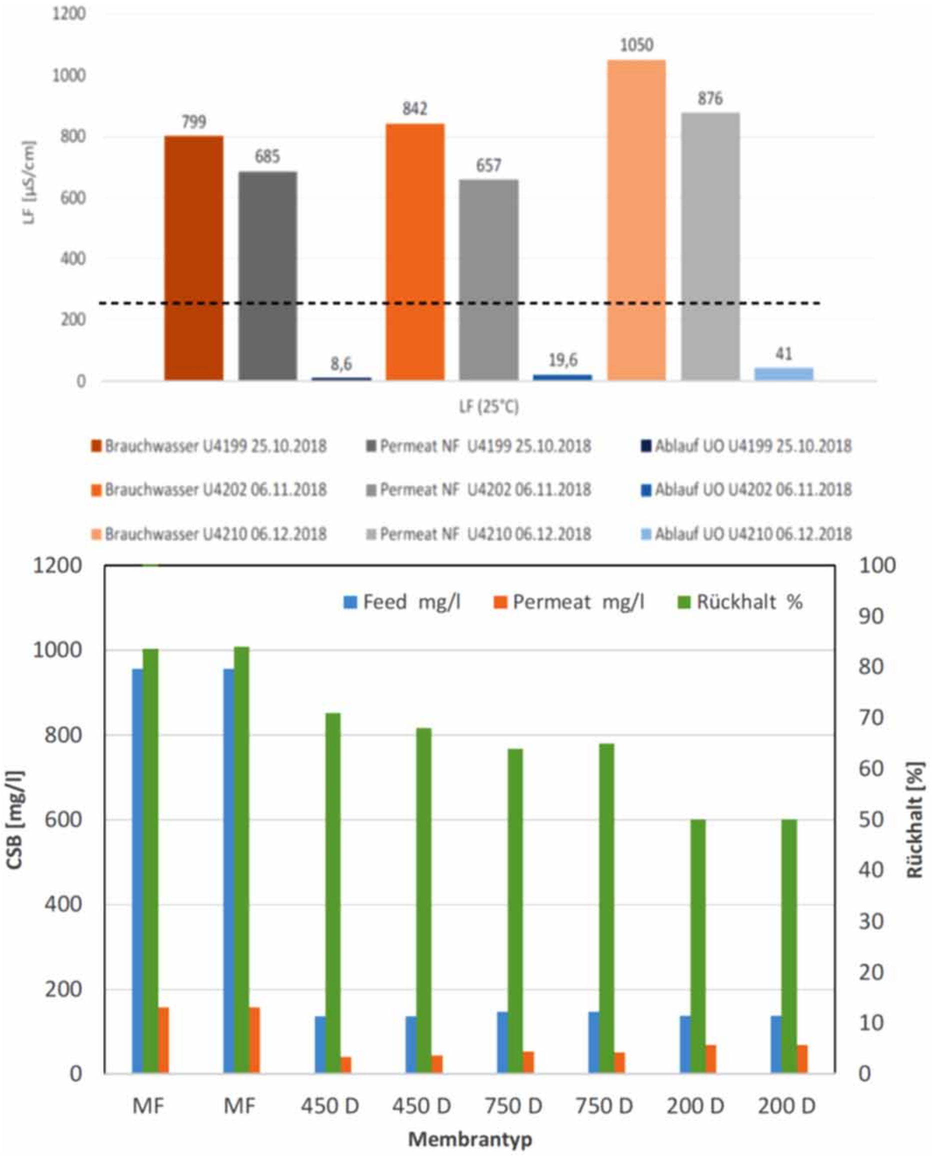

Figure 10 | Conductivity removal using NF + RO (top) and COD removal ('CSB'£') using MF + NF (bottom).

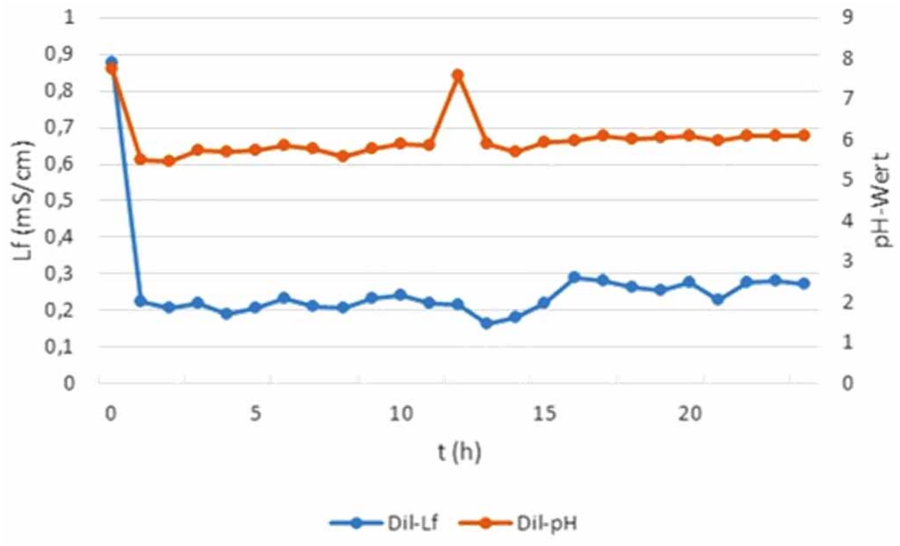

Figure 11 | Treatment of NF filtrate by ED: Reduction of conductivity (blue) and pH (orange).

effectively treat industrial process waters to such an extent that:

1. Oil, particles, and divalent ions (to some extent) could be removed in continuous operation with real on-shore produced waters in an oxygen-free environment using flotation and MF as pre-treatment.
2. COD, suspended particles and conductivity could be removed from industrial ceramic wastewaters using MF as pre-treatment and ED or RO as post-treatment.

As a result, the water quality was such that industrial wastewater reuse schemes could be technically feasible on a large scale. Their economic merits can, however, only be established if the local drinking water and/or the effluent discharge are heavily restricted and costly. One should also add the caveat that the capacity of the membranes to sustain long stable operations with manageable fouling and efficient CIP cleanings while the operators successfully manage the handling of the waste streams (membrane concentrates) is recommended to be proven in a longer term (12 months) test run.

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DATA AVAILABILITY STATEMENT

All relevant data are included in the paper or its Supplementary Information.

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