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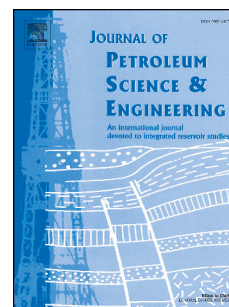
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Cleaning of ceramic membranes for produced water filtration

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Abstract

The application of ceramic microfiltration membranes to the tertiary treatment of produced water from an Arabian Gulf oilfield has been studied using a dedicated pilot plant. Studies were based on a previously published protocol in which the retentate stream was recycled so as to successively increase the feed concentration throughout the experimental run. Chemical cleaning in place (CIP) was applied between each run and the flux and permeability recovery recorded for various cleaning protocols studied, the CIP being based on the combination of caustic soda (NaOH) and citric acid. Surface analysis of the membrane, and specifically its hydrophilicity, was also conducted.

Results indicated the main influencing factor on permeability recovery from the CIP to be the employment of backflushing during the CIP itself. A final flux of $700 \text{ L m}^{-2} \text{ h}^{-1}$ was sustained through the application of 6 wt% NaOH with 6 wt% citric acid combined with backflushing at approximately twice the rate of the filtration cycle flux. A consideration of the impact of this flux value on the viability of two commercially-available ceramic membrane technologies indicated the footprint incurred to be slightly lower than that of the upstream induced gas flotation technology and corroborated a previously published estimate. The flux was sustained despite surface analysis indicating a loss of the innate hydrophilicity of the ceramic membrane.

Keywords *Produced water; ceramic membranes; chemical cleaning; footprint; flux.*

1 Introduction

Produced water (PW) generated from oil exploration requires rigorous removal of suspended matter (free oil and particulate solids) as tertiary treatment (downstream of hydrocyclone and gas flotation) if it is to be desalinated for reuse (Alzahrani et al, 2014) or re-injected into low-permeability reservoirs (Judd et al, 2014; Xu et al, 2016). The application of membrane technology for this duty has been recently reviewed (Munirasu, 2016; Dickhout et al, 2017), and the option of ceramic membrane filtration widely explored (Ebrahimi et al, 2010; Guirgis et al, 2015; Weschenfelder, 2015, 2016).

The viability of membrane processes generally is largely dependent on sustaining a high membrane flux to minimise the process footprint, a particularly important attribute on offshore oil platforms where available space is at a premium. The use of silicon carbide (SiC) ceramic membranes for this duty has been demonstrated to provide a reliably high treated water quality (6.3 - 7.6 mg/L oil and grease (O&G), 4 - 8 NTU turbidity) for microfiltration (MF, pore size $2 \mu\text{m}$) and ultrafiltration (UF, pore size $0.04 \mu\text{m}$) membranes based on recent pilot-scale studies (Zsirai et al, 2016) using real PW from an offshore oil platform. The larger-pore MF membrane was shown to provide a significantly higher flux than the UF membrane, but was also subject to greater flux (and permeability) decline. Moreover, it was noted that there was a marked deterioration in both permeate water quality and permeability with successive experimental runs on the chemically-cleaned membrane. The work

emphasised the need to improve the efficacy of the chemical clean-in-place (CIP) applied between runs to recover both the permeability and selectivity of the membrane.

The current paper reports a series of trials conducted to identify the optimal protocol for applying a CIP so as to sustain a viably high permeate flux. The impact on the technology footprint was then determined, using a previously published protocol (Judd et al, 2014), based on the specifications of two commercially-available candidate ceramic membrane filtration systems for tertiary PW treatment.

2 Materials and methods

The pilot plant operation methodology has been described previously (Zsirai et al, 2016). The pilot plant (Fig. 1) and membrane elements were supplied by Liqtech International (Ballerup, Denmark), the membranes having been pre-conditioned through their used for 15-17 trials in a previous campaign prior to the commencement of the current study. The plant comprised two streams fitted with tubular membrane modules operated in crossflow mode. The plant, allowed operation at a fixed conversion with automated physical cleaning provided by backflushing and manually-applied chemical cleaning in place (CIP). Experimental runs were conducted with recirculation of the retentate (the concentrate stream from the membrane), intended to increase the feedwater suspended oil and solids concentration over the course of the run and so increase the challenge to the membrane. All runs were operated at a transmembrane pressure of 0.55-0.6 bar with backflushing for 5 s every 600 s at a back-pressure of 3 bar and a filtration cycle conversion of 20%, these conditions having been identified as being optimal in previous studies (Zsirai et al, 2016). The selection of the cleaning reagents was similarly based on this study.

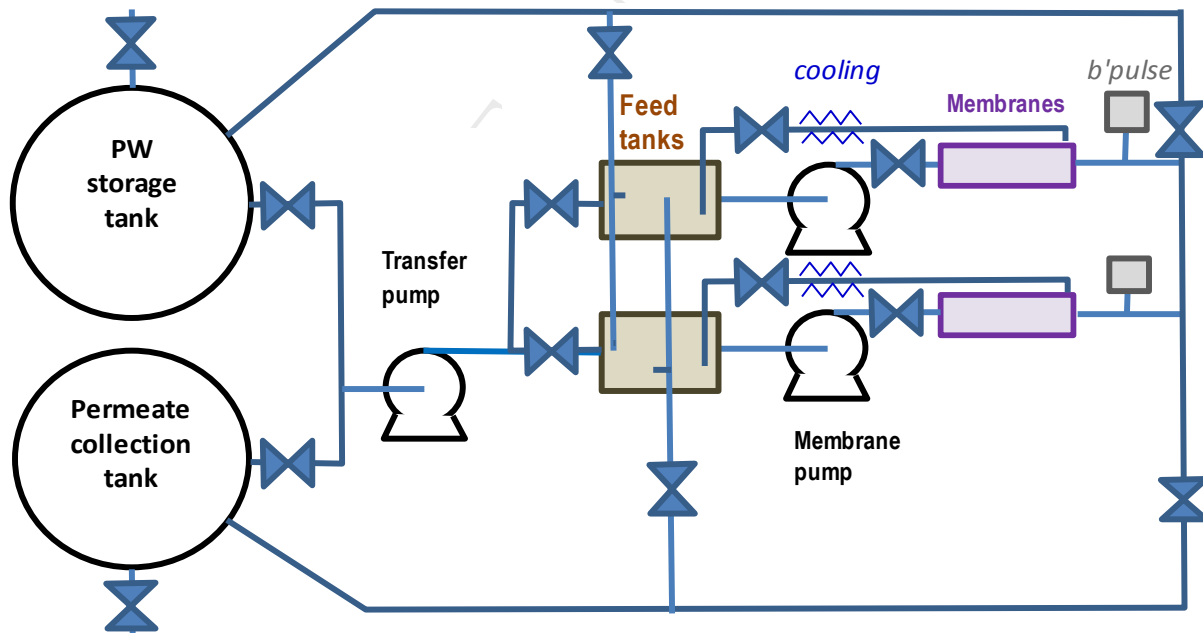


Figure 1: Pilot plant

Experiments were conducted on single MF and UF SiC membrane elements (25 mm diameter, 305 mm long, 37 channels of 3 mm square and with a total membrane area of 0.09 m²) which were chemically cleaned between runs. The MF and UF membranes were rated as 2 µm and 0.04 µm pore size respectively. The elements were challenged with PW shipped in 5-tonne batches from an oil platform operating in the Arabian Gulf, two different batches being employed throughout the trials with the PW nitrogen-blanketed to suppress oxidation.

The PW was sampled downstream of the induced gas flotation (IGF) step of the PW treatment train of the oil platform, the train being based on a classical two-stage hydrocyclone-IGF process. The work was limited to eight runs conducted on the two preconditioned membrane elements.

Experiments were conducted on 200 L feed volumes of PW with retentate recycling throughout which served to agitate the feed by jet mixing during the experimental run. Further jet mixing of the storage tanks was conducted prior to dispensing of the 200L volumes to ensure an even dispersion of the oil and solids in the stock solution. Chemical clean in place (CIP) studies were then carried out between each filtration run according to a multiple-stage protocol (Table 1), applying supplementary backflushing during the CIP for the final three runs (c – e). The backflushing was designed to help ensure that any dislodged solids were not immediately re-deposited onto the membrane surface by the flow of cleaning reagent. Reagents employed comprised an organic acid (citric acid), supplemented with sulphuric acid to lower the pH to 2, and caustic soda at pH 13 (Table 1). Whilst both base-acid and acid-base sequential cleaning were initially studied, no apparent consistent change was noted between the two sequences. The feedwater quality and CIP protocols employed for the trials conducted based on two different batches of PW, denoted by trials 1-3 (Batch 1) and a-e (Batch 2), are summarised in Table 2, the increase feed concentration arising from the recirculation of the retentate.

Table 1: Cleaning-in-place procedure

Reagent, ~50L aliquots	Recirculation time and temperature
1. Mains water	30 min at ~25°C
2. 6% NaOH	30 min at pH 13
3. Mains water	30 min at ~25°C
4. 6% citric acid	30 min at pH 2
5. Mains water	30 min at ~25°C

Table 2: Feed water quality and CIP conditions

Run	Feed O&G		Feed turbidity		Cleaning reagents		Cleaning sequence	BF
	Start	End	Start	End	NaOH	Citric acid		
1-3	24-25	32-84	21-34	39-74	2%	2%	B-A	N
a	52	148	123	202	2%	6%	A-B	N
b	40	76	99	158	6%	2%	A-B	N
c	47	52	72	461	2%	2%	A-B	Y
d	50	95	408	955	2%	6%	A-B	Y
e	42	93	663	998	6%	2%	A-B	Y

Runs 1-3 refer to Batch 1, Runs a-e refer to Batch 2; B-A base followed by acid; A-B acid followed by base; BF backflush during CIP.

Surface analysis of the virgin and used membrane material was by contact angle measurement, conducted according to standard methods (APHA, 2001), to assess the surface hydrophobicity. SEM-EDAX measurements were also made to assess the extent of formation of calcium, barium and iron alkaline scales. The autopsies were intended to provide an indication of the degree of permanent fouling from oil and inorganic reagents. Analysis for turbidity and oil and grease (O&G) was according to standard methods (SMWW 2130 B and 5520 C).

3 Results

The flux and permeability profiles followed the same trends as in previous studies (Zsirai et al, 2016), with the flux decline over the filtration cycle and recovery during the backflush

cycle both decreasing with time (Fig. 2). As outlined in previous work, the end permeability refers to the permeability resulting from the same hydraulic load and, if based on the same PW batch, roughly the same pollutant load between tests. The start and end permeabilities between tests (respectively the green and red bars in Figure 3) are therefore directly comparable.

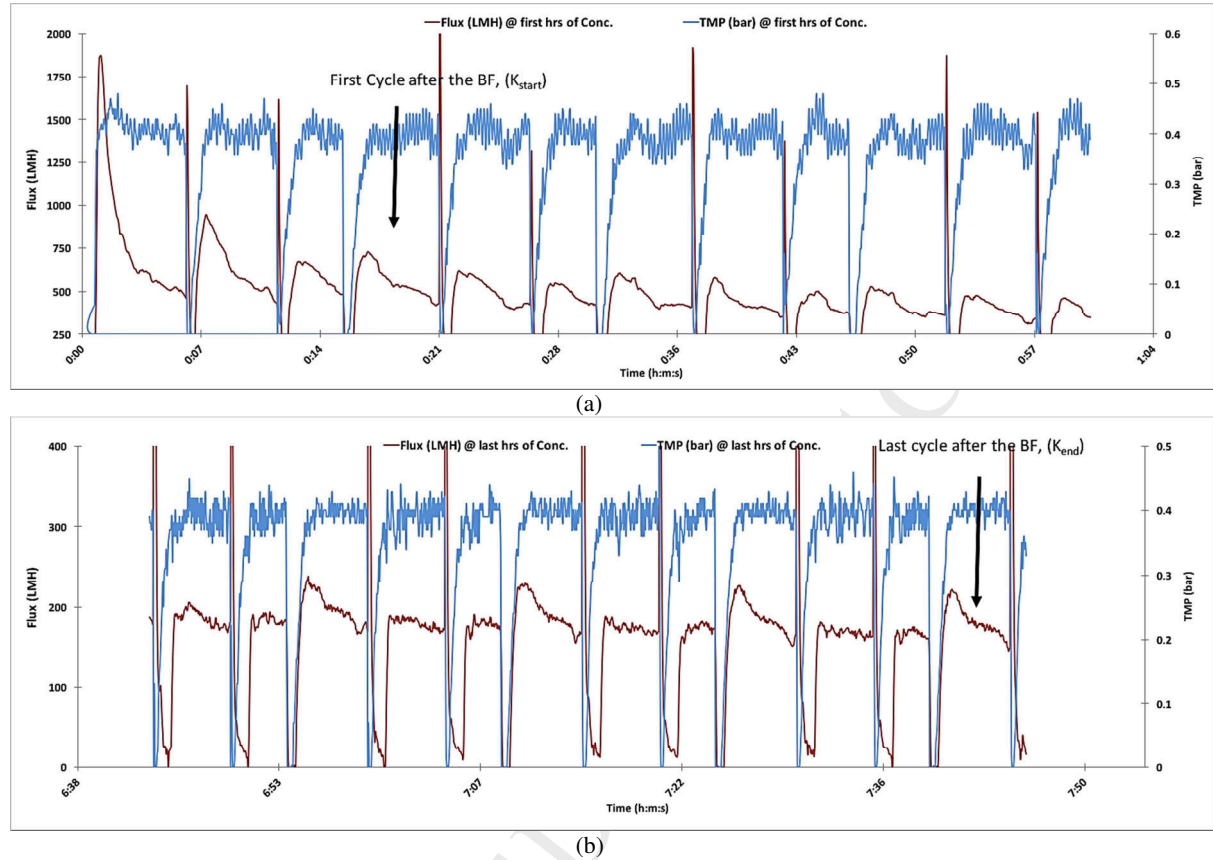


Figure 2: Typical filtration transient: flux and pressure transient from the first (a) and the last hours (b) of the concentration test.

Results indicate a significant difference in fouling propensity between the two batches. There are a number of possible explanations for this, including an approximate doubling of the O&G content and more than order of magnitude increase in turbidity (Table 2). It is also possible that Batch 2 had a higher concentration of organic matter of higher fouling propensity. This would include production chemicals based on flocculent polymers, which are known to foul polymeric membranes though are less onerous to ceramic materials. However, the very significantly higher colloidal content, manifested as the turbidity, is the most likely cause of the decreased end permeability from Run 3 to Run a for the MF membrane (Fig. 3b). This is corroborated by the far less significant corresponding decrease for the UF membrane (Fig. 3a), which would be expected to be more resistant to colloidal fouling.

Notwithstanding differences in fouling propensity of the two batches the greatest impact on sustaining permeability is from the application of backflushing during the CIP (Runs c-e) for both the UF and the MF membranes, although for the final UF run the CIP failed to recover the permeability (Fig. 3a). In the case of the MF a final permeability of between 1190 to 1280 LMH/bar was sustained for the three runs (Fig. 3b), with the CIP recovering the permeability to 1480-1630 LMH/bar for the start of the following run. This compares with final permeabilities of 210-310 LMH/bar for Runs a and b, prior to the introduction of backflushing during the CIP. In the case of the UF membranes the corresponding final

permeability values were 367-384 LMH/bar for the backflushed CIP tests compared with 202-232 LMH/bar prior to the introduction backflushing during the CIP. The increased efficacy of the backflushed CIP was thus almost four times greater for the MF membrane compared to the UF.

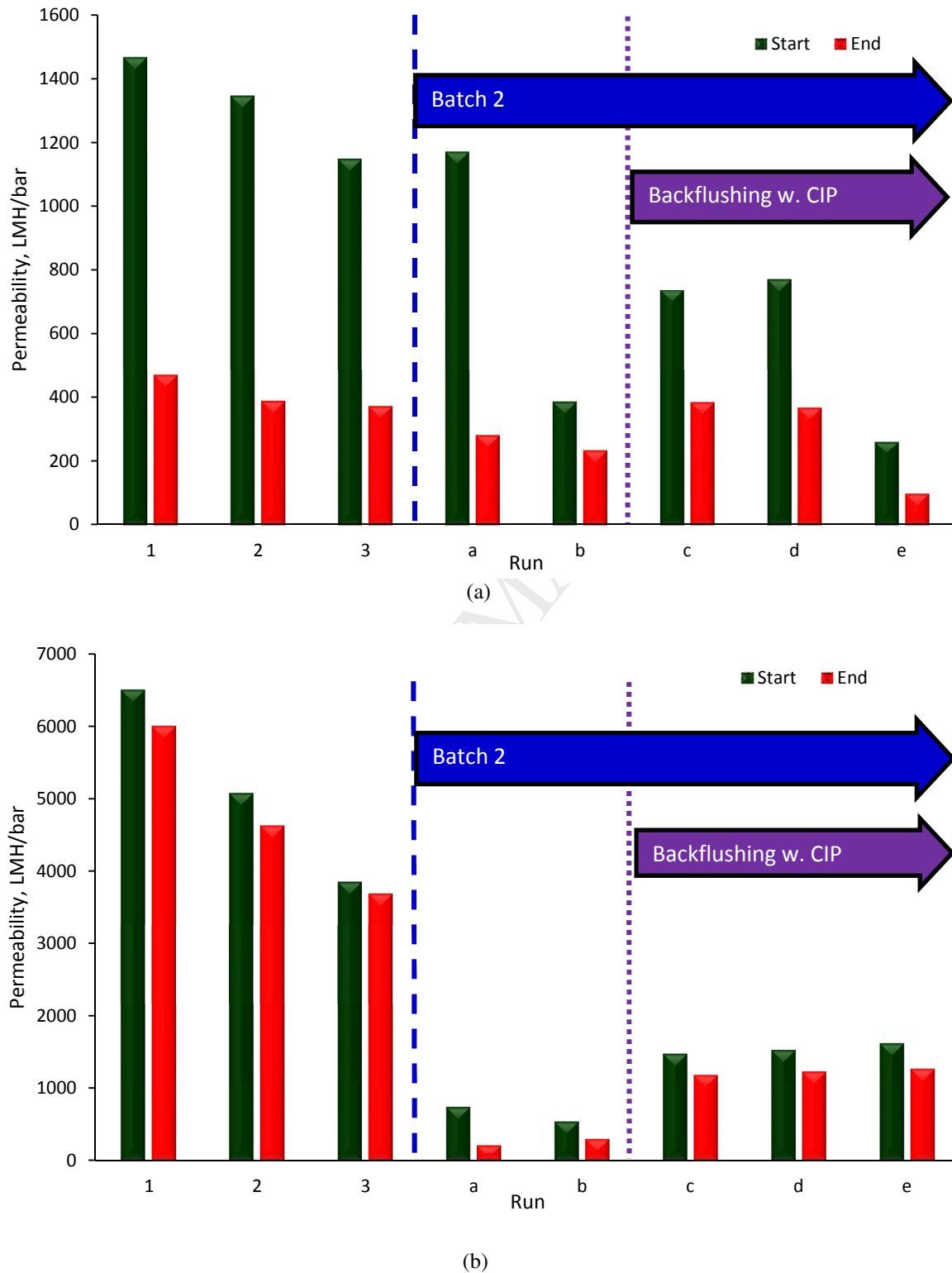


Figure 3: Permeability trend with successive experimental runs (a) UF, and (b) MF

A mean treated water quality of $<7.1 \pm 1.6$ mg/L O&G (oil and grease) in for the UF membrane and 8.5 ± 1.6 mg/L for the MF was maintained throughout. This arose despite the increase in feed water O&G concentration and the impact of recycling of the retentate, and

was in keeping with the outcomes of the previous study employing the same protocol (Zsirai et al, 2016). The use of the backflush during the CIP also appeared to stabilise the permeate turbidity to <25 NTU, corresponding to >98% removal which is assumed to take place at the membrane surface. The rejection provided by the MF membrane following the backflushed CIP was slightly higher than that of the UF for both turbidity and O&G, which is counter-intuitive given the significantly higher selectivity expected for the UF membrane given the rated pore size.

The membrane autopsy indicated only negligible quantities of inorganic scalants and metal hydroxides (calcium and iron) on the membrane surface for both the MF and UF membranes, as might be expected given the strength and rigour of the acid cleaning sequence of the CIP. This was despite the PW being heavily supersaturated with hardness (between 9 and 15 g L⁻¹ as CaCO₃). However, the surface was evidently affected by contact with the oil, with the contact angle measurements ($\theta = 106\text{-}116^\circ$) indicating significant hydrophobicity for the used membranes, compared with complete hydrophilicity ($\theta = 0^\circ$) for the virgin membrane material. It can therefore be surmised that the decline in permeability is primarily attributable to the oil content of the PW, rather than the inorganic scales. Since the membranes had already been used for a number of tests prior to the current study (Section 2) it is likely that they in a hydrophobic state throughout the campaign.

Examination of the filtration transients for Runs c-d, where backflushing during the CIP was applied, indicated a negligible permeability decline between backflush events for the final 5-7 backflushes of each run. A final flux of around 700 LMH was correspondingly sustained for the MF membrane, notwithstanding the loss of membrane surface hydrophilicity.

4 Discussion

There have a number of studies of the application of ceramic membrane technology to the treatment of real petroleum industry wastewaters reported since 2010 (Table 3). Few of these have been conducted at pilot scale, challenged with real oilfield produced water (OFPW), and/or operated for a significant period of time. An early site-based trial (Lee et al, 2005), apparently employing a spiral-wound hydrophilic polymeric membrane, achieved a flux of 10-20 LMH and maximum permeabilities of 6-8 LMH/bar after an extended operating period of 650 hours. Since then somewhat higher fluxes and permeabilities from similar site based trials using ceramic membranes have been reported, ranging from 60 (Pedenaud et al, 2011), to more than 1000 LMH/bar (Prado-Rubio et al, 2011) depending on the feedwater quality and the state of the membrane (Table 3).

However, it is evident that the sustainable flux attainable is greatly dependent on the CIP efficacy. Permeability recoveries varying by factors of 2-3 have been reported from pilot plant studies based on real PW (Prado-Rubio et al, 2011), with significant permeability reduction ensuing from insufficiently cleaned membranes. A conclusion common to all studies based on real petroleum effluents is the low membrane permeability due to the gradual build-up of hydrophobic matter on or within the membrane material. Enhanced permeability recovery has been demonstrated both by adjustment of the physical cleaning (i.e. backflushing) conditions during the filtration cycle (Silalahi and Leiknes, 2011) and by sequential base-acid cleaning (Weschenfelder et al, 2015b). The current study suggests that further improvement in permeability recovery can be attained through backflushing during the CIP, yielding a final sustainable flux of around 700 LMH and a corresponding permeability of ~1200 LMH/bar – somewhat higher than previously reported values (Table 3).

Table 3: Studies of MF/UF membrane filtration of PW and other petroleum effluents

Oil (TSS) concn mg/L, water source	Scale	Material	Pore size μm	Init flux, LMH	Fin flux, LMH	TMP, bar	Fin perm (range), LMH/bar	time, h	T, °C	Reference
200-1000, tank dewatering effl	b(m)	AlO	0.2	128	28	1	28	2	60	Ebrahimi et al, 2010
200-1000, tank dewatering effl	b(m)	TiO	0.05	80	4	1	<5	2.5	60	Ebrahimi et al, 2010
200-1000, tank dewatering effl	b(m)			120	30	1	120-30	2	60	Ebrahimi et al, 2010
OFPW	p	TiO, SiC	0.01-0.1	200	-	0.5-3.5 0.25-1.5	60 135	120	-	Pedenaud et al, 2011
221-722 (7-17), OFPW	p	SiC	0.04-0.1	-	135-590	0.35-0.95	450-1020	26	-	Prado-Rubio et al, 2012
20 (2.9), OFPW						0.6	520			
3-25 (24-74), OFPW	p	SiC	0.1-0.5	25-120	50	0.3-1.5	150	12-14	45	CoMeTas, 2011
52-458, gas field PW	p	Al-Zr	0.05	-	170-255	-	190-240	600	25	Subramani et al, 2011
-, SAGD effl	p	AlO	0.05	200	-	1.52	132	-	45	Guirgis et al, 2015
~250, refinery effl.	b	ZrO	0.1	1000	290	1.5	193	-	45	Weschenfelder et al, 2015
9-43, PW	p	AlO	0.2	-	295-312	2.5	118-125	-	35-60	Reyhania & Meighani, 2015

KEY LMH litres per m² per hr; SAGD Steam-assisted gravity drainage; OFPW oilfield produced water; TMP transmembrane pressure; TSS total suspended solids.
b bench; b(m) bench, based on membrane module; p pilot
AlO aluminium oxide; SiC silicon carbide; TiO titanium oxide; ZrO zirconium oxide

There are three aspects to the technical viability of the process for the duty envisaged:

- Attainment of required water quality,
- Limitation to a maximum footprint, and
- Impact on operation of other PW process technologies.

Determination of the footprint demands certain assumptions regarding process technology design/configuration and the flow and loads on the oil platform respectively.

4.1 Water quality

It is evident from all measured water quality data for the membranes tested that a high treated water quality is sustained regardless of the membrane material or pore size, the residual oil assumed to be in the dissolved form. Permeate quality values reported from other studies suggest a total suspended solids (TSS) concentration below 5 mg/L, unless the membrane has been made irreversibly hydrophobic (Prado Rubia et al, 2011) causing breakthrough of free oil. The permeate water is otherwise potentially appropriate for reinjection into the reservoir or overboard discharge. The retentate stream would be expected to have a similar composition to that of the return stream of the upstream IGF, and could thus be managed in a similar manner.

4.2 Footprint

The footprint of classical and tertiary PW treatment processes has been determined with reference to flow normalised against the area (F_A) and volume (F_V) occupied. F_A thus takes units of m/h: m³/h per square metre of floor area in m². F_V correspondingly has units of h⁻¹, it being the flow in m³/h per m³ volume occupied. A ceramic membrane skid based on vertically-aligned 1 m modules has been determined to have an area and volume footprint of ~7 m/h and ~2.6 h⁻¹ respectively based on literature information (Judd et al, 2014), consistent with a recently-published study (Weschenfelder et al, 2016) in which a value of 6.5 m/h was determined for F_A . This value is comparable with the nutshell filter (NSF) alternative tertiary technology in terms of floor area but more compact with respect to volume.

To provide a more accurate estimation of the footprint demands reference to existing commercial ceramic membrane technologies, of which there are two which have been implemented for PW filtration: the Liqtech International *M99* skid and the Veolia Water Technologies *ROSS*TM system based on the company's *Ceramem* membranes. The Liqtech technology comprises 99 x 250 mm diameter 0.328 m² membrane area modules, compared with the Veolia technology which has 52 x 142 mm dia. 10.5 m² modules. These data coupled with the skid dimensions for the respective technologies (Table 4) allow the packing density, the membrane surface area per unit skid footprint or volume, to be determined. As can be seen, the two technologies are similar in specification.

Table 4: Technical specification of two commercial ceramic membrane filtration technologies

Technology	Skid dimensions, m				Module		Skid	Packing density*	
	L	H	W	A, m ²	No	A _m , m ²		m ² /m ²	m ² /m ³
<i>Veolia</i>	18.3	2.7	1.8	33.5	52	10.7	556	17	6.1
<i>Liqtech</i>	1.2	4.5	1.5	1.80	99	0.328	32.5	18	4.0

A: skid floor area; packing density: membrane surface area per unit skid footprint or volume

A_m: membrane surface area. *With reference to skid.

Determination of the F_A and F_V values for the two technologies (Table 5) demands assumptions regarding both the conversion of feedwater into permeate (taking account of the use of permeate for backflushing) and the redundancy arising from the requirement for taking a stream off line for periodic chemical cleaning. Redundancy can be assumed to be

manifested as three skids each providing 50% of the flow capacity (i.e. duty-duty-standby), adding 50% to the footprint. Based on backflushing for 1% of the time at 150% of the operating flux, the adjusted conversion is calculated as being 92.5% assuming a conversion of 96% for the forward filtration cycle. The resultant net F_A and F_V values are calculated as being 7.2-7.8 m/h and 1.7-2.6 h⁻¹ respectively. These figures roughly corroborate the values of 7.0 m/h and 2.6 h⁻¹ for F_A and F_V originally estimated in the review article by Judd et al (2014).

Table 5: Footprint determination, based on two commercial ceramic membrane filtration technologies

Techno -logy	Uncorrected			Corrected for redundancy		<i>Backflushing data</i>				Corrected for backflushing	
	Flux m/h	F_A m/h	F_V h ⁻¹	F_A m/h	F_V h	<i>Freq</i> h/d	<i>Flow</i> m ³ /h	<i>Vol</i> m ³	<i>Conv- ersion*</i>	F_A m/h	F_V h ⁻¹
<i>Veolia</i>	0.7	11.6	4.2	7.8	2.8	0.24	584	140	92.5%	7.2	2.6
<i>Liqtech</i>	0.7	12.6	2.8	8.4	1.9	0.24	34.1	8.2	92.5%	7.8	1.7

Redundancy: three skids each providing 50% of the flow capacity (i.e. duty-duty-standby)

Ancillary equipment excluded: assumed to be comparable for each technology

*Assumes filtration cycle conversion of 96%, based on the experimental data for a conversion of 20% for the 300 mm element, feedwater pre-concentrated by a factor of ~2.5, and two stages of 850 mm-long full-scale modules.

4.3 Impact

It would be expected that the treated water could either be discharged overboard, provided the regulated discharge limits are based solely on suspended matter and/or total oil (of 15 mg/L or more in the case of the latter), or used for reinjection (PWRI). Whilst the high permeate water quality generated is generally considered to be appropriate to PWRI, the compatibility is dependent on the precise permeability and other characteristics of the reservoir. These include the formation water chemistry, and specifically the likelihood of the precipitation of supersaturated scalants.

In addition to the treated water, a further stream (the retentate) is created which comprises 4.5% of the feed flow and >90% of the solids and organic load. This would need to be managed in the same way as the concentrate streams from the upstream secondary treatment processes (the HC and IGF steps). The impact of the additional concentrate stream will therefore be dependent on the capacity of the management system, but the additional hydraulic load from the membrane return stream would be unlikely to add more than 10% to the hydraulic load. This is based on the assumption that the HC and IGF concentrate streams comprise around 5% of the feed flow. Further analysis of the impact of the backflush stream demands flows and loads information of the PW as managed on an individual platform. A further stream which requires management is the wastewater from the chemical clean, which makes up no more than 0.5% of the feed flow.

Finally, the rate of chemical consumption is determined both by the total membrane area and the cleaning frequency (assumed to be daily). Chemical storage requirements depend upon shipping costs and any logistical constraints, but given that the reagents would be stored as concentrates the storage volume and associated footprint would be relatively low.

5 Conclusions

The operation of a pilot plant fitted with silicon carbide (SiC) microfiltration (MF) ceramic membranes and fed with real produced water (PW) derived from an Arabian Gulf oilfield has indicated that a flux of 700 LMH with a corresponding permeability of 1400 LMH/bar can be

sustained over a number of filtration cycles. This flux is attained through the application of backflushing during the clean in place (CIP) between filtration cycles, and appears to be sustained despite the tendency of the ceramic membrane to become hydrophobic as the number of filtration cycles (and so exposure to the PW free oil) increases.

A consideration of two commercially-available SiC-based technologies, each fitted with vertical membrane modules, indicates that a flow per unit membrane skid area footprint of 12-14 m/h (or 2.8-4.2 h⁻¹ per unit volume occupancy) can be attained at the ~700 LMH flux measured for the MF membrane. These values decrease to 7.2-7.8 m/h and 1.7-2.6 h⁻¹ respectively if 50% redundancy is assumed and filtration cycle conversion and backflushing are taken into consideration which reduce the overall conversion to 92.5%. On this basis the footprint incurred by the ceramic membrane technology is comparable to values previously estimated, though this excludes ancillary equipment such as the valve and pump skids, the control panel and the cleaning in place holding tanks and related equipment. Whilst the footprint and process efficacy, with respect to water purification capability, suggest the process to be viable for offshore applications, benchmarking against the alternative media-based tertiary filtration process (the nutshell filter) is needed to assess the process viability on-shore. Moreover, benchmarking should necessarily take account of the economic implications of technology selection (i.e. a technoeconomic analysis). Planned future work in this area will encompass a sensitivity analysis of individual operation determinants related to the cleaning cycles of both technologies so as to assess the relative impact of enhanced cleaning and flux (or filtration velocity) on overall cost with reference to reagent use and residuals generation and management.

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Highlights

Pilot-scale testing of SiC ceramic membranes challenged with real produced water
Two membrane pore sizes tested and various chemical cleaning protocols assessed
Outcomes used to determine area and volumetric footprint of full-scale installation
Backflushing during chemical cleaning found to be critical in recovering permeability
Calculated footprint values for optimised cleaning comparable to published estimates

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Cleaning of ceramic membranes for produced water filtration

Zsirai, T.

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