

TREATMENT OF PRODUCED WATER USING SILICON CARBIDE MEMBRANE FILTERS

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ABSTRACT

Large quantities of effluent water are produced from oil and gas wells during exploration and production stages. This produced water contains significant amounts of undesired components and is typically disposed of in abandoned oil wells or in salt water disposal wells, or is re-injected into new and/or operating wells. Due to growing environmental concerns and water scarcity, more attention is given to reusing this water. However, scale forming ions, bacteria and residual oil must be removed prior to re-injecting the produced water into the wells. Otherwise, oil output is reduced due to plugging of the channels in the deposit. Current methods to remove these contaminants often use large amounts of chemicals and settling tanks to condition or clean the water. These methods are often expensive and have large footprints. Silicon carbide (SiC) membrane filters have shown good results in filtering produced water. They have a small footprint and effectively remove bacteria, suspended solids and residual oil in simple steps. SiC filters have shown high flux compared to polymeric and other ceramic filters. They exhibit good abrasion and corrosion resistance, are easily cleaned with any chemical and do not exhibit severe fouling tendencies. In this article, field trials of SiC filters in a produced water treatment plant are discussed. Results show that SiC filters can effectively remove suspended solids, bacteria and dispersed oil in a much simpler circuit than conventional treatment systems.

INTRODUCTION

Oil and gas deposits contain large amounts of water in the formation rock. This water gushes out along with the oil and gas during production and is called produced water or formation water. Today, nearly 115 billion barrels per year (bbl/y) of water are produced worldwide as a by-product of oil and gas¹. In average, for every barrel of oil, three barrels of water are produced from oil wells. As the well ages, this ratio dramatically increases, sometimes rising as high as 50 barrels of water per barrel of oil produced¹. During fracking operation, nearly 3-5 million barrels of water are injected into the well to cause fracturing in the formation rock to capture the oil and gas from the deposit. Between 20% and 60% of this amount of injected water comes out during the initial stages of production as produced water (often called flowback water). As the oil well ages, several enhanced oil recovery (EOR) techniques are used to maintain the reservoir pressure. One of the most common techniques practiced for enhanced oil recovery is water flooding.

Treatment and disposal of the produced water is gaining increasing importance due to environmental concerns, scarcity of fresh water and lack of disposal wells in certain areas. More and more produced water is reused in the formation to enhance oil recovery. There is also a growing interest and increasing usage of recycled produced and flowback water from fracking operations. As the industry grows, more and more fresh water is tapped from nearby surface

water and ground water sources. This competes directly with domestic and agricultural usage of water. Lately due to drought and overuse, ground water levels are dropping in arid areas such as California and Texas. Water recycling is therefore becoming ever more important to the sustainability of our water resources. The quality of the recycled water significantly affects the long-term recovery of oil. Scale forming ions, bacteria, solid particles, etc. can plug the pores in the formation rock, blocking oil recovery. Thus, treatment of the produced water to remove detrimental components is very important.

Types of Contaminants in Produced Water

The properties of produced water vary from site to site depending on the geology of the formation rock, life of the well and the extraction process used. Understanding the composition of produced water is important to design suitable treatment processes and disposal methods. In this section, we will discuss the constituents that affect direct reuse of produced water in the well. Contaminant removal to meet regulatory restrictions is beyond the scope of this article.

The major components which can affect the recovery of oil from an injection well are: residual total oil and grease (TOG), suspended solids, high salt concentration, scaling ions, and bacteria. The effect of these contaminants is described below.

Oil and Grease

Residual oil and grease from primary separation in produced water is a major concern for both onshore and offshore operations. The oil and grease can cause fouling of the downstream treatment equipment, and can reduce future oil output by creating emulsions in the deposit. Oil must therefore be removed from the water prior to disposal or reuse of the wastewater. Oil can be present in the following forms in produced water: (1) Free oil, (2) Dispersed oil, (3) Dissolved oil and (4) Emulsified oil. **Free oil** is constituted of oil droplets which have a diameter greater than 30 μm . Typically, given adequate settling time, these oil droplets rise to the surface of the settling tank and are skimmed off. **Dispersed oil** refers to smaller oil droplets in the water which are stable due to surface charges and range from sub-micron to hundreds of microns in size. They are formed from both aliphatic and aromatic hydrocarbons. Dispersed oil is often difficult to remove by conventional techniques, and is better removed by new technologies such as dissolved air flotation (DAF) or membrane filtration. **Dissolved oil** in produced water includes low molecular weight organic compounds such as organic acids, polycyclic aromatic hydrocarbons, phenols and volatile hydrocarbons. BTEX (benzene, toluene, ethylbenzene, and xylenes) are of particular concern due to their risk of contamination of soil and ground water and their harmful effect to the central nervous system. Dissolved oil is almost impossible to remove by physical separation techniques. **Emulsified oil** is often present in produced water, and is generated during prior treatment processes where agitation is involved. Emulsified oils are stable due to the presence of surface active agents in the oil-water interface, and are difficult to remove by mechanical means alone.

Suspended Solids

Produced water contains very fine particulates originating from the geological formation and the well operation, and the total suspended solids (TSS) can vary from a few ppm to several hundred ppm. The suspended solids contain precipitated salts from dissolved minerals, sand, clay, fine proppants, eroded minerals, etc. These can re-precipitate in the deposit and reduce oil and gas flow. They adversely affect the oil-water separation and sometimes produce oil-water sludge which is difficult to clean. Very often, the suspended solids contain toxic metals. Metals

contained in the produced water can also react with chemicals added to the initial water, e.g. fine particles of iron can interfere with the cross-linked polymers used in frack water. Iron is also the key contributor for corrosion. Reaction between iron and hydrogen sulfide generates iron sulfide which causes corrosion. Iron sulfide also forms scale similar to the scale forming ions mentioned below. Iron bacteria and sulfate reducing bacteria in the presence of iron can also generate iron sulfides. For these reasons, suspended solids need to be removed prior to reinjection or reusing the water. Today, flocculation and agglomeration, followed by settling, are the main methods used to remove TSS from the produced water. These are often slow and inefficient methods. Ceramic membranes have a good potential to remove TSS from water.

Scale Forming Ions

Bi-valent cations Ca^{2+} , Mg^{2+} and Ba^{2+} are the main scale forming ions in produced water. These cations enter the water when salts in the formation are dissolved. In older wells where the water has more time to react, the produced water dissolves more salts and in turn carries high amounts of scale forming ions. Unless controlled, they easily form scales by re-precipitating at lower temperature and pressure. They can clog flow lines or create oily sludge which is difficult to remove. They can also create hard-to-break emulsions in the deposit resulting in reduced recovery. In general, scale forming ions are either precipitated or controlled by adding softening agents. Barium, although found in smaller quantities, is the most damaging to pipelines in the oil wells. To-date, limited chemical treatment procedures exist to clear cement-like barium plugs, and the oil and gas industry spends millions repairing damaged wells from barium sulfate formations in their systems.

Micro-organisms

Bacteria, virus and other microorganisms are often present in produced water. Unless removed they can easily scale the oil wells and cause corrosion by forming iron sulfide. The key microbes present in produced and flowback water are: iron bacteria, sulfate reducing bacteria (SRB), acid producing bacteria (APB) and archaea. Today, the majority of microorganisms are removed by adding biocides (oxidizing and non-oxidizing) to the produced water. UV treatment and high pH conditions are also used to destroy bacteria. The treatment of bacteria using ultraviolet rays is considered as a green alternative but its efficiency is limited due to the turbidity of water.

CONVENTIONAL WATER TREATMENT SYSTEMS

Today, produced water is managed in three different ways: (1) by disposing of the water in injection wells, (2) by reusing the water after mixing with fresh water, (3) by recycling the water for reuse. State regulations, logistics, financial implications and availability of fresh/river water dictate the method of management for a particular oil field. Although today the majority of water is either disposed of or re-injected in injection wells or reused by blending, increasing attention is given to recycling treatments.

Today conventional water treatment plants utilize oil-water separation, coagulation and flocculation methods in the upstream of the process, and a combination of settling tanks, flotation, filtration and microbial disinfectants at the downstream to separate oil, remove iron, suspended solids and microbes, and reduce hardness. The process employs biocides in combination with other chemicals and technologies such as UV treatment to remove bacteria and other organisms. A schematic of a generic water treatment plant is given in Figure 1. Today water treatment plants employ conventional processing equipment from the mining, municipal

and other industries. Although these technologies work in many places and applications, they encounter several shortcomings for the oil and gas industry. These processes tend to be heavy in footprint, use large amounts of chemicals and biocides, require intensive labor, and can be high in capital cost. Removal of fine particles in conventional settling tanks takes a long time, which significantly reduces throughput. Traditional bag filters remove particles larger than 2-5 μm , but do not capture submicron particles. Efficiency of dispersed oil removal is often complex, expensive and inadequate. The use of large amounts of biocides to remove bacteria can be expensive and harmful to the environment. As a result of these shortcomings, a lot of novel processes have been introduced in the last few years including electro-coagulation, new-and-improved evaporation techniques, ozone treatment and H_2O_2 oxidation, etc. One of the promising treatment technologies currently being evaluated is micro- and ultrafiltration, particularly with ceramic membrane filters made from silicon carbide (SiC). These filters can remove suspended solids, bacteria and dispersed oil in a single step. Details of this method are described in the following sections.

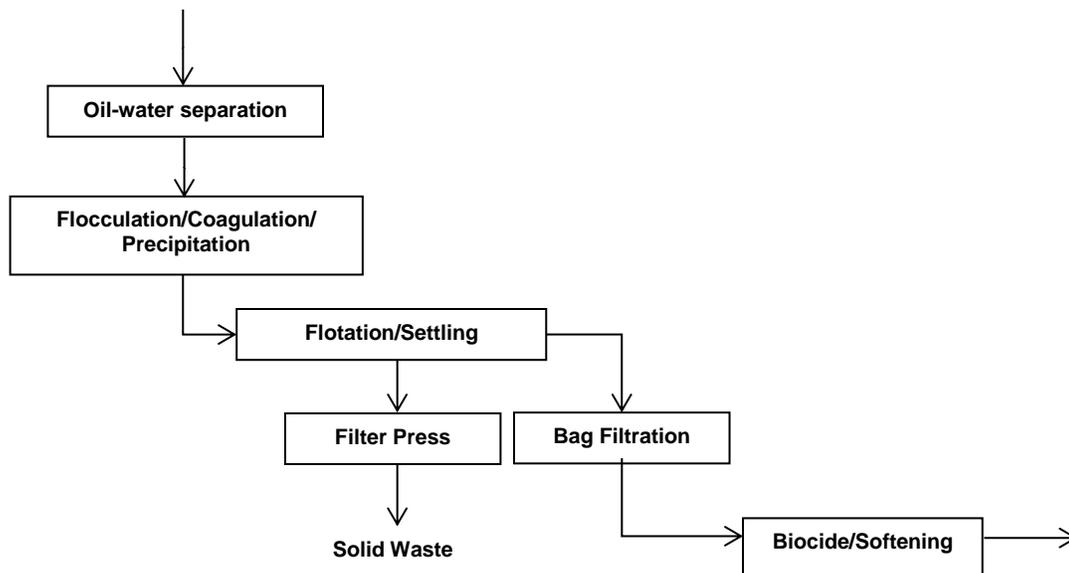


Figure 1. Schematic of a typical produced water treatment plant.

CERAMIC MEMBRANE FILTERS

Ceramic filters can be used in a variety of applications ranging from industrial wastewater treatment, to treatment of effluent waters in the petrochemical and oil & gas industry, to cleaning swimming pools, and to filtration of food, beverage and pharmaceutical products²⁻⁶. Commonly, these applications operate in the microfiltration range, given by the pore size of the ceramic membrane. A key advantage of ceramic membranes in comparison to polymeric membranes is their resistance to heat and chemicals, making them ideally suited for treatment of hot, corrosive wastewaters^{2,4,7,8}. Produced water temperatures can rise up to 180°F (82°C), and unlike many polymer filters, ceramic filters can easily withstand these temperatures. The chemical stability also allows for chemical cleaning without affecting the chemistry and stability of the filter material. In addition, a narrow, controlled pore size distribution enables efficient removal of particulates at high flux rates⁷.

Ceramic filters can be made from a variety of ceramic materials such as alumina, zirconia, titania and silicon carbide^{3,4,6,8}. Depending on the application and the system design, they can be shaped into plates, discs, tubes and cartridges. Tubular, cross-flow filters are a typical design for filtration of liquids with high solid loadings. The feed water enters the filter on one end and passes through channels inside the filter tube. The channels are coated with porous membrane material of a certain pore size through which the water is filtered. The clean permeate exits the filter through those porous walls while the retentate exits the filter at the opposite end of the tube. The process is illustrated in Figure 2. The performance and durability of the filters is directly related to the microstructure and material of the membrane. Uniform and well-connected pores are needed for efficient filtration at desired flow rates without excessive fouling or clogging.

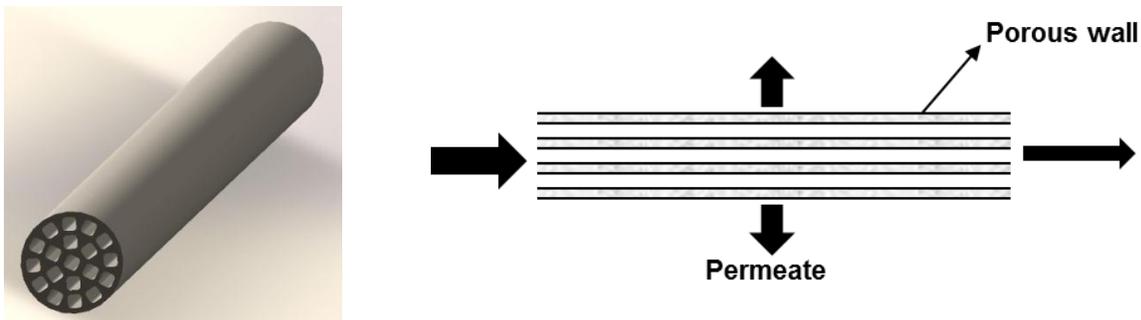


Figure 2. Schematics of cross-flow filtration.

Uniform membrane microstructures can be achieved by carefully controlling the manufacturing process parameters such as grain size distribution, purity of the raw materials and firing cycle. Typically, ceramic filters are manufactured via a multi-step process involving subsequent sintering cycles at different temperatures. First, a ceramic tube with wide channels is manufactured and sintered to a certain, coarse-grain microstructure. Layers of ceramic material yielding much smaller pore sizes are then applied onto the surface of the channels of the carrier tube and form the actual filtration membrane. The finer grain size of the membrane layer is tailored through suitable slurry formulation and sintering profile. Figure 3 shows a cross-sectional view of a Saint-Gobain cross-flow ceramic filter with a SiC carrier layer (coarse grain) and a SiC membrane layer (fine grain). Silicon carbide filters are very suitable for harsh environments, as they have good wear and corrosion resistance over traditional ceramics.

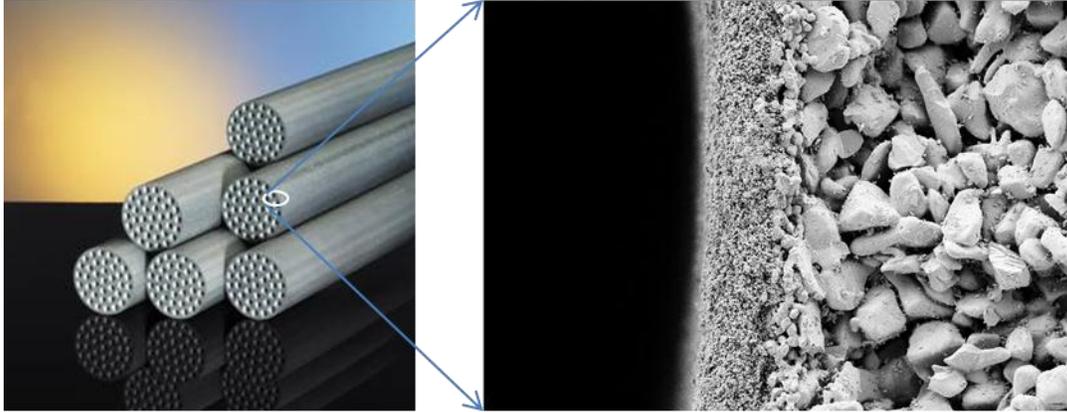


Figure 3. Cross-sectional view of a Saint-Gobain SiC cross-flow filter. The membrane layer on the inside of the channels consists of a fine-grain ceramic microstructure while the carrier material, which makes up the actual filter tube, has a much coarser microstructure.

FIELD TRIALS

CASE STUDY I – Filtration of produced water from the Uintah Basin, UT

In the first case study, SiC membrane filters were tested in a water treatment plant in the Uintah Basin, Utah. The plant is owned and operated by Environmental Clean Systems, LLC (ECS), a water management company based in Phoenix, AZ. The filters were manufactured by Saint-Gobain Ceramic Materials. The plant commonly treats produced water from conventional oil wells to remove oil, bacteria, suspended solids, sulfur, while also reducing the hardness, before the water is re-injected into the oil wells. Typical characteristics of the incoming water to the plant are given in Table I.

Table I: Typical characteristics of incoming produced water

Constituent	Incoming water
Total suspended solids	50-350 ppm
Total dissolved solids	~15,000 ppm
Oil	Up to 150 ppm
Bacteria	60,000 CFU/ml
Hardness	200-20,000 + ppm
Iron	1-150 + ppm
Dissolved gases	varies

The particle size distribution of the suspended solids in the incoming produced water is shown in Figure 4.

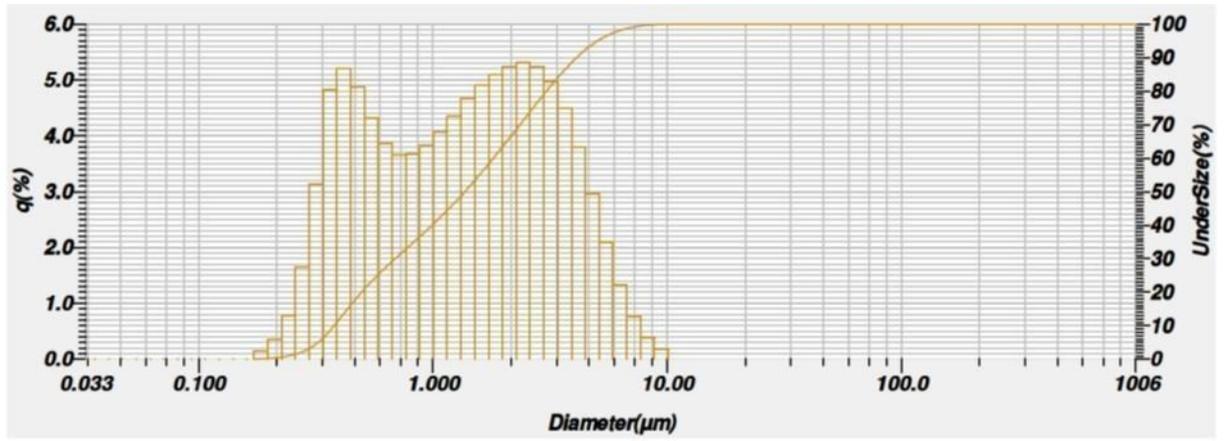


Figure 4: Particle size distribution of suspended solids in produced water.

A simple schematic of the process flow for our filtration tests using SiC filters is shown in Figure 5. Water was first pre-treated to simultaneously agglomerate very fine particles and precipitate sulfur from H_2S . Coagulated particles enhance flux through the filter by removing particles with sizes close to the membrane pore size in the filter.

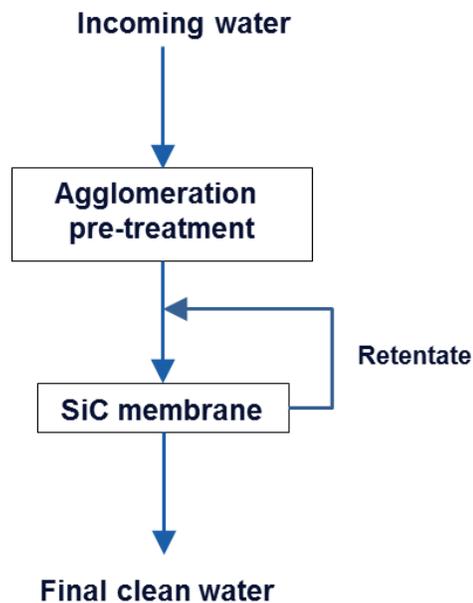


Figure 5. Process flow chart for filtration trials.

Saint-Gobain SiC cross-flow filters with 250 and 1000 nm membranes were used for filtration tests. Table II summarizes the main characteristics of the filters. The filters were contained inside a PVC housing, and were run for several hours with and without backwash at different

trans-membrane pressure (TMP). The permeate flow rate and the water quality of the permeate were closely monitored.

Table II: Characteristics of Saint-Gobain SiC filters with 250 and 1000 nm membranes used for the filtration tests.

Design ref.	Membrane pore size [nm]	Length [mm]	Outer diameter [mm]	Number of channels	Filtration area (nominal) [m ² /m]
25-16-5.0	250 and 1000	1178	25	16	0.254
25-19-3.4	250	1178	25	19	0.231

CASE STUDY II – Filtration of produced water from the Bakken formation, North Dakota

Our second case study was conducted at the ECS produced water disposal facility in North Dakota. High salinity water from a North Dakota oil field was tested to determine flux changes when using SiC membrane filters for treatment of water at high salt content. Saint-Gobain SiC filters with 250 nm membranes were used as shown in Table II. The incoming water had a salinity of ~234,000 ppm (Total Dissolved Solids (TDS)). In order to vary the salinity, the water was diluted with tap water to ~137,000 ppm. Since the H₂S content was low, the water was run through the filter as received. Results from these tests are discussed in the following section.

RESULTS AND DISCUSSION

Effect of Filtration Membrane Pore Size and Backwash on Flux

Figure 6 shows the permeate flux as a function of time for the 250 and 1000 nm membranes (Design ref. 25-16-5.0) at two different TMPs, 0.4 and 1 bar. For both membranes and TMP conditions, the high initial flux quickly dropped, followed by a more gradual, steady decrease. In the “steady-state regime”, the permeate flux was highest (>200 LMH) at 1 bar TMP for both membrane sizes, in contrast to the test condition at 0.4 bar TMP (~130 LMH). At constant TMP, permeate flux was similar for both the 250 and 1000 nm membranes, which indicates the built-up of a dynamic membrane or filtration cake. This implies that solid particles and oil droplets present in the feed water are pushed to the wall of the membrane by differential TMP and create a transient cake (compact layer) on the membrane surface. This transient cake is seen to drive the overall permeability, as similar flux results are obtained for both the 250 and 1000 nm membranes. It is worth noting that the formation and structure of this transient cake, and hence its permeability, strongly depend on the interactions with the SiC membrane surface. Consequently, different results should be expected for other membrane materials, especially oxide ceramics.

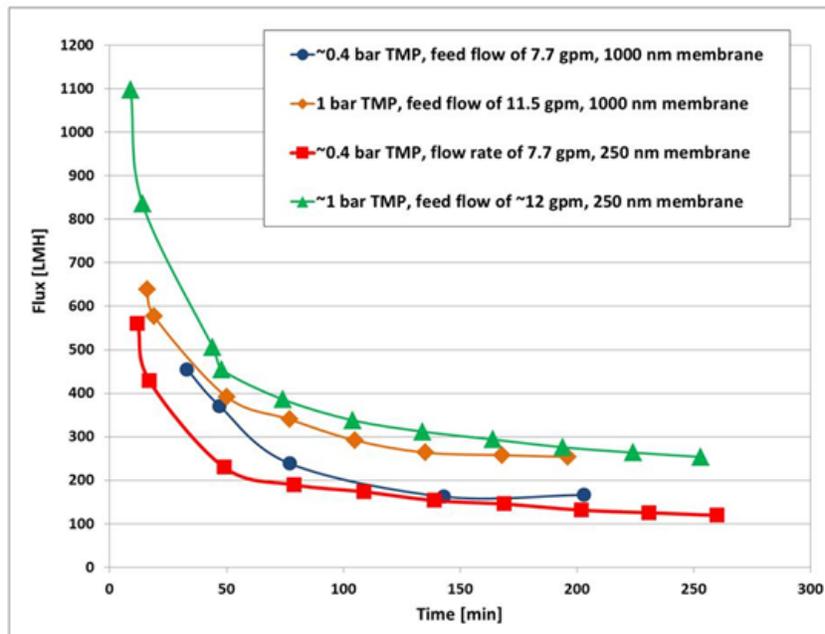


Figure 6. Permeate flux for 250 and 1000 nm SiC membranes (Design ref. 25-16-5.0) at 0.4 and 1 bar TMP. Note: in order to achieve the higher TMP, the feed flow rate was increased from ~7.7 to ~12 gpm.

Figures 7 and 8 show the direct comparison of measured permeate flux for filters with 250 and 1000 nm membranes at the two TMP conditions and for multiple backwash cycles. The flux decreased at a similar rate for both 250 and 1000 nm membranes. The decrease and then stabilization of the permeate flux again indicates that a dynamic membrane is building up on the inside of the cross-flow filter channels. The purpose of these tests was to observe whether flux rates could be restored with simple backwash. As seen from Figures 7 and 8, after each backwash, the initial flux increased substantially. This demonstrates that the sediment at the membrane surface can be easily removed by backwash. From these tests, it is also concluded that the filters in this case do not need to be backwashed very frequently or that backwash conditions can be optimized (low duration, low pressure) to increase permeate throughput and save energy.

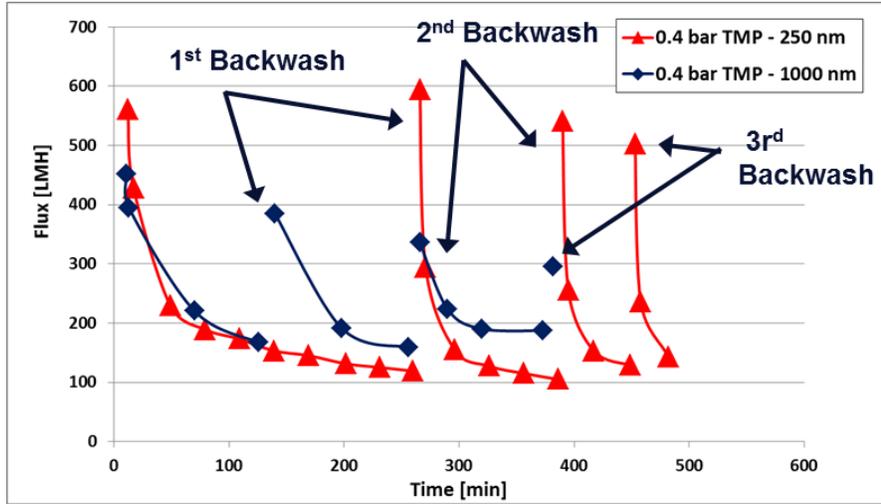


Figure 7. Permeate flux for 250 and 1000 nm SiC membranes (Design ref. 25-16-5.0) at 0.4 bar TMP. The filters are backwashed after different time intervals.

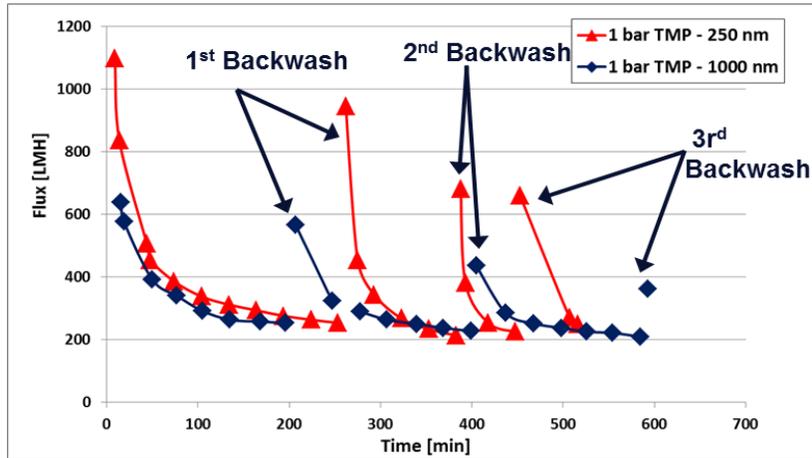


Figure 8. Permeate flux for 250 and 1000 nm SiC membranes (Design ref. 25-16-5.0) at 1 bar TMP. The filters are backwashed after different time intervals.

Effect of pH on Flux

NaOH was added to the incoming water to reduce its hardness. The produced water contains Ca and Mg in the form of dissolved chlorides: Ca^{2+} , Mg^{2+} , Cl^- . Increasing the pH through the addition of NaOH (or $\text{Ca}(\text{OH})_2$) causes Ca and Mg to precipitate as CaCO_3 and MgCO_3 . These carbonates are solid and not soluble in water. By adding polymeric flocculants, the fine precipitates of carbonates can be flocculated for enhanced removal through the SiC filter.

As pH was increased from 8.4 to 12.2, hardness of the permeate water decreased from 325 ppm to 35 ppm. Figure 9 shows that permeate flux increased substantially at higher pH when hardness decreased through this pre-treatment. This could be a result of the lower hardness that reduced the electro-viscous friction of water passing through the membrane. Ca and Mg ion concentrations also decreased substantially as the membrane removed the corresponding precipitated salts (Table III).

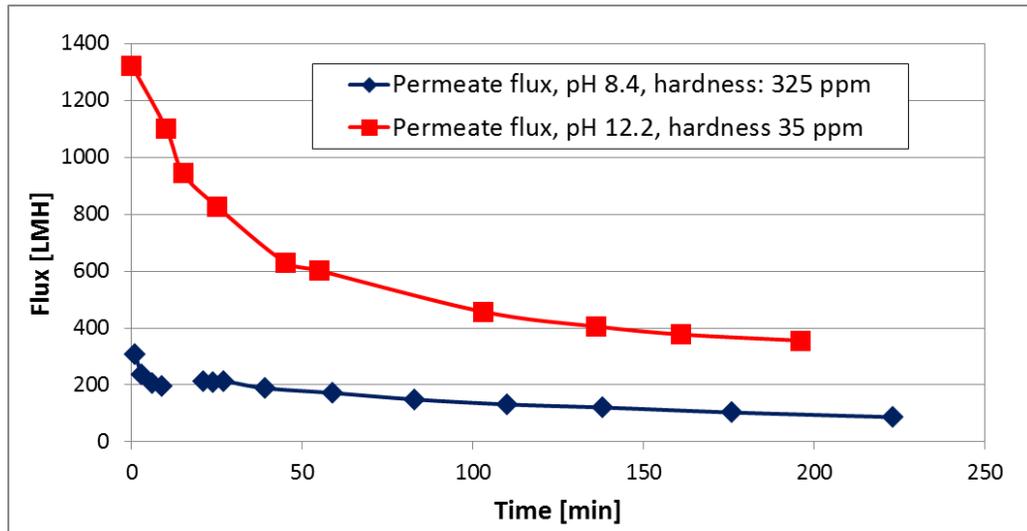


Figure 9. Permeate flux at varying pH. Flux increased as hardness of the produced water decreased. A SiC filter with 250 nm membrane (25-19-3.4) was used for this test.

Table III: Water analysis for pre-treated produced water filtered through SiC membranes with and without additional pH treatment.

Water sample	TDS, ppm	pH	Temp °F	Alkalinity, ppm	Ca, ppm	Mg, ppm	Iron, ppm	Hardness, ppm	TOG, ppm
Without pH treatment									
Feed water	8738	8.4	95	1125	116	14	35	350	86
Permeate	9189	8.4	95	1200	112	11	7.1	325	12
With pH treatment									
Feed water	8733	12.2	114	1125	160	18	35	475	92
Permeate	9201	12.2	114	4003	1	8	0.6	35	12

Removal of Suspended Solids and Impurities

Figure 10 compares the permeate water with the feed water. The permeate shows good clarity with very low turbidity; turbidity could not be measured with the turbidity meter due to the low value ($\ll 1$). This indicates that the SiC filter removed TSS very effectively.

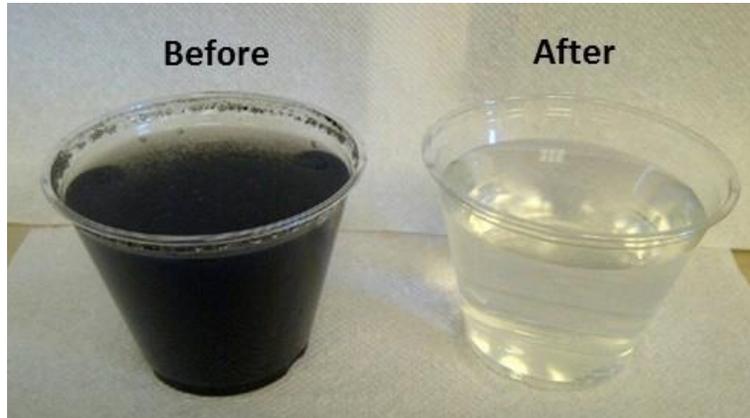


Figure 10. Comparison of turbidity before and after filtration. Permeate water from the filter shows very good clarity.

Comparison of the chemical analysis for the produced feed water and the permeate water confirms good TSS removal by the SiC membrane, as indicated by a significant decrease in Fe level after filtration (Table III). The amount of TOG dropped to ~ 12 ppm, while the typical standard water treatment process reduces TOG to ~ 11 ppm.

Bacteria removal

Bacteria content was determined by using serial dilution vials (which contain a proprietary reacting culture) for both sulfate reducing bacteria (SRB) and acid producing bacteria (APB). The sample water (1 ml) was injected with a syringe in a first vial and mixed well. After mixing, 1 ml of the sample from that vial was injected into a second vial and so on. The vials were stored in an oven maintaining a specific temperature; the duration of storage was 7-30 days. If the sample water contains bacteria, the clear SRB vials will turn black and the red APB vials will turn yellow. If the sample water has no bacteria, no changes in the color will be observed. Figure 11 illustrates the process.

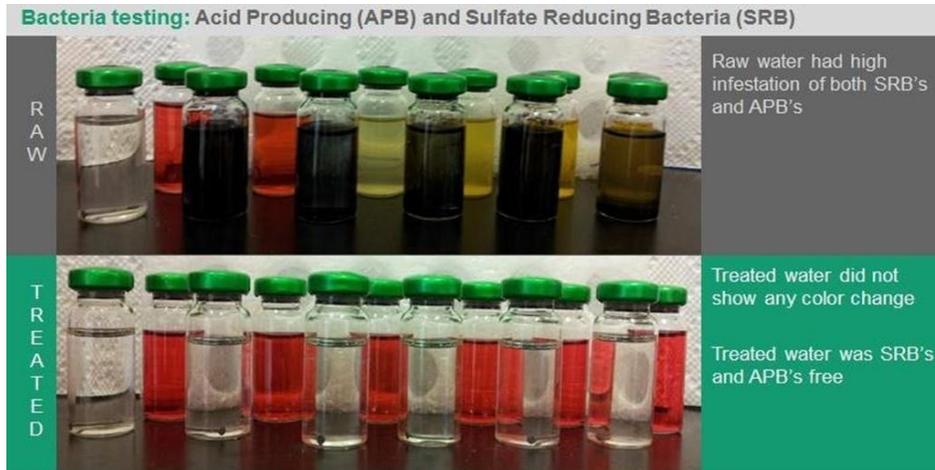


Figure 11. Test method for bacteria.

Table IV summarizes the results for the detected levels of bacteria in the feed water and the permeate water. “0” means that no bacteria are present, “1” means that vial no. 1 has bacteria, “2” means that vials no. 1 and 2 have bacteria, “3” means that vials no. 1, 2 and 3 have bacteria, and so on. The target is to get “0” in all vials. This characterization method for bacteria levels corresponds to a logarithmic decrease as the vials are sequentially diluted. Thus a “0” in all four vials will indicate a 4-log removal of bacteria. Our results show that the filters enable 1- to 4-log removal of APB and 2- to 4-log removal of SRB. Some bacteria were still present after the test. However, the use of SiC filtration membranes will require much less biocide than conventional processes to reach a 4-log removal. No biocide treatment was required when the pH of the incoming water was increased to the highly caustic range. SRB were completely eliminated through the pre-treatment while the remaining APB were fully removed by the SiC membrane.

Table IV: Analysis of bacteria in feed water (inlet) and permeate (outlet).

Sample Collection Date	Filter pore size, nm	INLET		OUTLET	
		APB	SRB	APB	SRB
7/16/2014	1000 nm	4+	4+	2	1
7/17/2014	250 nm	4+	4+	0	0
8/6/2014	1000 nm	4+	4+	3	1
8/7/2014	1000 nm	4+	4+	2	1
8/8/2014	1000 nm	4+	4+	2	1
8/11/2014	1000 nm	4+	4+	0	0
8/12/2014	1000 nm	4+	4+	3	2
8/13/2014	250 nm	4+	4+	2	1
8/14/2014	250 nm	4+	4+	2	1
pH pre-treatment (pH 12.2):					
10/29/2014	250 nm	2	0	0	0

Effect of Salinity on Flux

The effect of salinity on flux was studied at the Bakken oil field in North Dakota during our second case study. The incoming water had a salinity level of ~234,000 ppm (TDS level). In order to vary the salinity, the water was diluted with tap water to ~137,000 ppm. Results of permeate flux at different salinity are shown in Figure 12. A reduction in TDS by a factor of ~2 led to an increase in flux by a similar factor. Lower salt content reduces friction of the water and enhances flux.

As shown in Figure 13 and Table V, the SiC membrane reduced the amount of TSS; the permeate water was very clear.

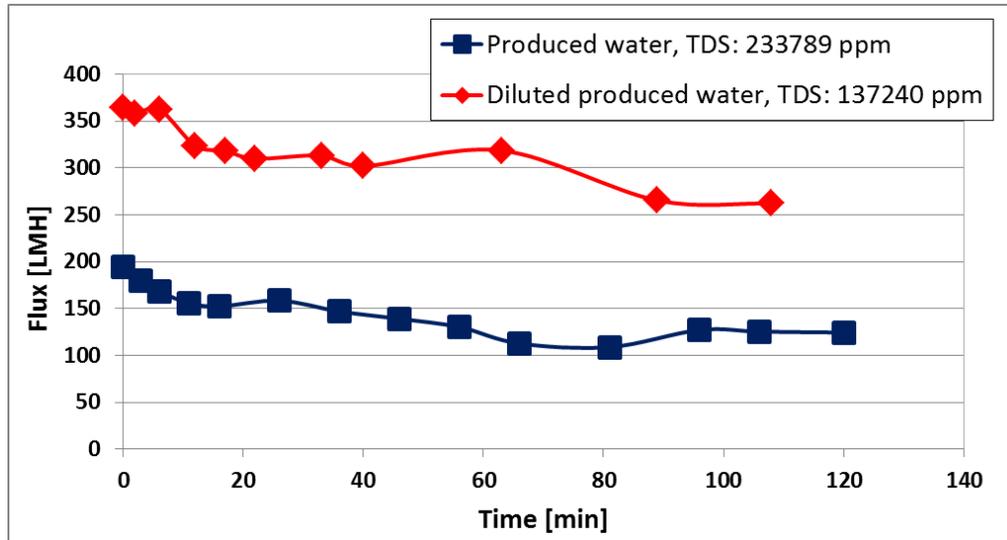


Figure 12. Effect of salinity on permeate flux. SiC filter used: 250 nm membrane, design ref. 25-19-3.4

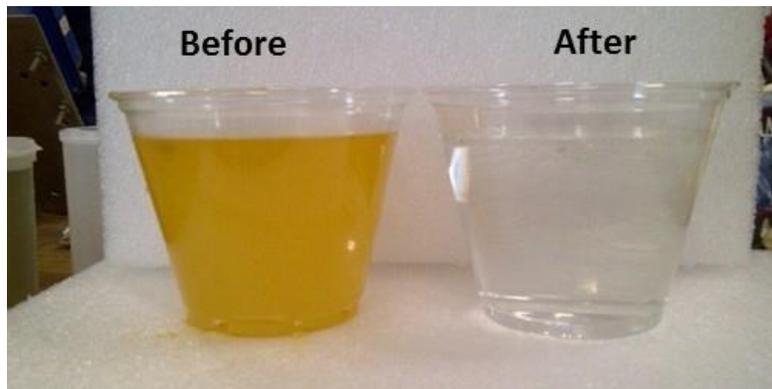


Figure 13. Produced water from Bakken, ND, before and after filtration through SiC membrane.

Table V: Water analysis for filtration samples from tests at the Bakken oil field in North Dakota.

Date	Water	pH	Specific gravity @ 77 F [g/cc]	TSS [mg/L]	TDS (calculated) [ppm]
11/4/2014	Produced water as-received	6.05	1.18	865	233789
11/4/2014	Permeate	6.1	1.18	116	233950
11/4/2014	Produced water diluted ~50%	6.33	1.105	455	137240
11/4/2014	Permeate	6.43	1.1	NA	138915

CONCLUSIONS

The tested Saint-Gobain silicon carbide filters provide a simple and efficient alternative to conventional treatment methods of produced water from oil and gas fields. In comparison to the standard water treatment processes that involve several process steps at a large footprint, similar water quality can be achieved by a single-step filtration process using SiC membranes in combination with a precipitation and agglomeration step. The initial testing of the SiC membrane showed that the membrane has a potential of being not only a purification technology but also a separation and microbial removal technology in the produced water management process. Depending upon the quality of the water needed, the membrane itself is capable of removing hydrocarbons, iron, TSS and bacteria without any necessary pretreatment. SiC membrane filtration technology can be a more environment-friendly and less costly alternative to the current produced water management process.

PUBLICATION

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