Fouling Control for Polymeric Membrane Processes

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Abstract

Issues with fouling and membrane degradation are the most important factors influencing the success of polymeric membrane produced water treatment installations. Controlling fouling is critical and requires a solid understanding of produced water and membrane chemistry.

Polymeric membrane materials are susceptible to many forms of fouling and oxidation. Recent academic and industrial research has focused on improving membrane materials to better handle trouble constituents. Even with significant improvements in membrane material pretreatment and effective membrane cleaning is required to manage fouling. Selecting an appropriate pretreatment strategy is critical to the success of the project.

This paper will focus primarily on the chemical composition and properties of commonly available polymeric membranes, produced water chemistry as it applies to membrane treatment and strategies employed to control fouling through pretreatment and cleaning. Due to the limited number of successful produced water membrane treatment projects, the paper will draw on case studies from successful municipal and industrial membrane treatment projects and their relationship to coal bed methane produced water applications.

Introduction

In a 2006 white paper written for the US Department of Energy National Energy Technology Laboratory (NETL) and the State of Wyoming, All Consulting reported that an average of 550 million barrels of produced water was produced in Wyoming annually. A significant portion of this water is relatively high quality CBM produced water with potential for water reuse. Due to slightly high total dissolved solids (TDS) levels and sodium adsorption ratio (SAR), additional treatment is required to fully utilize this resource. Since the paper was written, significant centralized treatment facilities have been implemented with the intent of utilizing produced water as a resource. Because drainages in some coal bed methane basins cross state borders there has been a significant push for zero liquid discharge (ZLD) facilities to ensure that existing water quality is not diminished by coal bed methane development.

In the event that ZLD is required, or beneficial reuse is sought, reverse osmosis (RO) should be considered because RO produces extremely high quality treated water. In such events it is unlikely that RO can be applied as a stand alone treatment process because the permeate produced by RO is acidic requiring post treatment stabilization prior to discharge and RO treatment produces a brine stream that must be treated with an extremely salt tolerant desalination process or properly disposed of. If the treatment process is correctly designed, RO can be simple and cost effective to operate on many different produced water chemistries.

There are two likely scenarios for produced water treatment: centralized facilities and well head systems. Implementation of a centralized treatment facility will simplify treatment system operations and maintenance (O&M) and reduce capital costs through economy of scale. When the coal bed methane wells feeding the treatment system are distributed across large distances, well head systems may greatly reduce the cost of transportation associated with the produced water treatment. In both cases, the use of RO will reduce the volume of brine requiring disposal while producing a useable treated water source. For each system RO pretreatment will dictate the economic feasibility of treatment because inappropriate pretreatment selection will lead to frequent membrane element replacement, servicing and extremely labor intensive operation.

Reverse Osmosis Process Description

During reverse osmosis, water is preferentially transported across a semi permeable membrane creating a highly purified permeate stream and a concentrated reject stream. There are several mechanisms that contribute to water and solute flux through a membrane. These mechanisms have been described by three process models: the solution-diffusion model, the pore flow model and the preferential sorption-capillary flow model.

In the solution diffusion model, the membrane is permeable but nonporous. Water and solutes dissolve into the membrane material, diffuse through the solids membrane material and then liquefy on the permeate side of the membrane. Dissolved ions behave as liquids because of charge gradients in the membrane. Transport occurs as water and solutes travel along the membrane through interstices between polymer molecules. Separation happens when the flux of water is different from the flux of solutes. Flux through the membrane is produced by both solubility and diffusivity. As a result of the model, low solubility produces a low driving force for transport and low diffusivity produces a low diffusion coefficient.

The pore flow model differs by assuming that liquids flow through void spaces in the membrane. Straining is the dominant mechanism for rejection. In the pore flow model, charge repulsion and ion size account for differences in rejection among species with similar molecular weight.

The preferential sorption-capillary flow model assumes that the membranes have pores. Components are preferentially adsorbed to the pore walls and transported through the membranes. Preferential adsorption produces separation. The sorbed layer migrates through the membrane in response to a Gibbs Energy Gradient. In the sorption-capillary flow model, separation is a function of surface chemistry of the membrane and solutes rather than pore dimension.

Many mathematical models have been developed to describe water transport and membrane rejection. These models commonly couple the transport mechanisms to help describe the rejection phenomena observed during testing and operation. Basic rejection models consider electrostatic repulsion at the membrane surface, chemical solubility, diffusivity and straining of solutes. In many rejection models, negatively charged molecules are thought to be well rejected by charge repulsion and positively charged molecules may be rejected to maintain electro neutrality in the system. Polar compounds and molecules with hydrogen bondable functional groups generally pass through the membranes while large molecules are thought to be rejected through straining. Using empirical methods, each major membrane manufacturer has developed projection software to model the rejection of their membrane products.

Today, the majority of reverse osmosis membrane products on the market are thin film composite (TFC) membranes. These materials were originally developed by Francis, Cadotte and Riley of Northstar labs in the late 1970's. As the name implies, TFC membrane uses a membrane skin layer applied on a support layer. The membrane skin is very thin and has a tight pore structure to improve separation and the support layer is thick and loose to provide additional structural support while minimizing hydraulic flow restrictions. The use of two layers allows independent optimization of each layer. For reference, a typical chemical formula for a polyamide skin layer has been included in Figure 1, which is located at the end of this document. Membrane selection is application specific and membrane properties are strongly dependent on proprietary membrane manufacturing processes. The difference in membrane properties can produce sharp differences in rejection of charged ions and low molecular weight polar species which may cause noticeable differences in the quality of treated water produced by the RO system or accelerated membrane fouling.

A majority of RO membranes are supplied in a spiral wound configuration. Figure 2 illustrates a spiral wound membrane module. The use of spiral wound membrane technology improves the overall packing density of the RO membrane material. A benefit of the spiral wound elements is very high packing density or amount of membrane area provided per equipment footprint. Figure 3 depicts spiral wound membrane elements installed in a pressure vessel. The spiral wound elements are loaded in series into pressure vessels. Brine seals on the membrane module ensure that the feed flow enters the membrane through the feed channels in the membrane element. Feed spacers in the membrane element improve hydraulic distribution within the element.

Many different feed spacer thicknesses are available. Thicker spacers allow slightly larger molecules to pass through the feed channel while use of a thinner spacer will allow more membrane per element. Proper selection of feed spacer thickness is important in a produced water application because poor selection of the thickness may affect the fouling rate of the RO process.

Membrane Fouling and Oxidation

Major RO foulant and oxidizers are classified as biofoulant, colloids, metals, oxidizers, scale forming compounds and organic compounds. Exhaustive research has been performed to identify and control the conditions that lead to fouling and scale formation.

Biofouling typically requires transport to and deposition of bacteria on the membrane surface. Following the initial deposition, the bacteria adhere to the surface of the membrane. Bio films form as the cells grow and replicate. The main issue with biogrowth is fouling caused by the extracellular polymeric substance (EPS). Accumulation of EPS increases concentration polarization and provides a nucleation site for inorganic crystallization. Biofouling is usually controlled by a combination of chemical, biological and mechanical pretreatment upstream of the RO process.

Colloids are suspended particles that can be large or small. Typically the size range of 0.5 to 5 microns are difficult to remove upstream of RO. They can harm the membranes through physical damage or fouling. Colloids are capable scouring, sandblasting and scratching the membrane surface. In the presence of oils and organic carbon, colloidal fouling can bond to the membrane and if combined with saturated salts, colloids may seed the scaling process. Colloidal fouling is generally observed on the front end of the train especially when oils are present and typically controlled through chemical treatment and filtration upstream of the RO treatment process.

Metals such as reduced iron and alum will bond to the surface of an RO membrane during the RO process. Depending on valence, the metal may be water soluble or insoluble. Insoluble metals such as ferrous iron will foul the front end of the RO train. Soluble metals will precipitate when equilibrium conditions are disturbed. Conditions capable of precipitating metals occur at the back end of the RO train where the reject stream is most concentrated. This type of fouling is difficult to remove and capable of seeding other inorganic scaling. Metals are generally controlled through chemical oxidation to an insoluble form followed by filtration upstream of the RO treatment process.

TFC membrane is incompatible with strong oxidizers including ozone and halides. The membrane structure is susceptible to attack at both the rings and carboxylic functional groups. Strong oxidizers are capable of cleaving the membrane polymer. This type of damage leads to reduced salt rejection that cannot be restored. Oxidizers can be controlled through chemical neutralization upstream of the RO process.

Inorganic ions and sparingly soluble constituents will form scale in an RO process if the concentration of the salt exceeds the saturation limit for the species. Scale formation typically limits the overall water recovery of the treatment process and can be controlled through upstream antiscalant and acid injection. Some salts are difficult to control with antiscalant compounds. One such salt is calcium phosphate which is present as multiple species over the pH range of 6 to 8. Each species has different crystal structure and solubility product, requiring different antiscalant properties and membrane cleaning strategy to control.

Many organic carbon species, including suspended oils, have charged functional groups that can bond to the membrane surface. Because of the size of these molecules, pretreatment filtration may not completely remove trouble constituents from the feed water source. Organic carbon is also a carbon source for bio growth. Some colloidal matter will bond with organic carbon and the membrane surface creating a foulant that is very challenging to remove.

Commonly Practiced RO Pretreatment

Produced water pretreatment will benefit from commonly used and well understood pretreatment processes including antiscalant addition, chemical oxidation, filtration and oil water separation. The most common form of RO pretreatment is antiscalant addition. Antiscalants control chemical precipitation through three main mechanisms:

- Threshold Effect and Chelation: Antiscalant physically hold the ions in solution. The difference between the threshold effect and chelation is the amount of chemical that must be present. The threshold effect occurs at dose levels less than the stoichiometric amount required for chelation.

- Crystal Distortion: Antiscalant bonds to the face of a crystal and alters the growth rate in the crystal growth plane.

- Dispersion: Antiscalant bonds to precipitated particles retarding crystal growth producing small and unsettlable solids.

Most antiscalant formulations are proprietary chemical blends of polyacrylates, phosphonates, organo phosphorus compounds or dendrimers. Antiscalant selection is extremely water chemistry dependent. Pilot testing is recommended for antiscalant selection because of the cost of antiscalant compounds and the potential cost savings through antiscalant dose optimization. Some antiscalant producers have developed dose projection software that can be used as a starting point for chemical selection and cost development. Antiscalants are commonly used in conjunction with sulfuric acid, which depresses the feed pH and ultimately the concentrate pH in an effort to minimize the concentrate stream scaling potential.

Chemical oxidation is usually practiced when reduced iron is present in the RO feed stream. The use of an oxidative process for iron removal requires additional filtration or sedimentation to recover the insoluble oxidized iron. Five forms of filtration are commonly used as RO pretreatment: automatic backwashing strainers, conventional multimedia filters, bag filtration, cartridge filtration and membrane filtration.

- Automatic backwashing strainers are commonly available from 10 micron to 500 micron nominal filtration rating. These strainers consist of a screen and backwash mechanism. The filters commonly operate in normal filtration mode until a differential pressure switch is activated sending the strainer into a backwash. The design of strainers is robust and the automatic backwashing feature minimizes operator requirements of the system. In a typical RO or MF process, automatic strainers are provided for large particle removal and membrane protection.

- Conventional multimedia filters can be either gravity fed or pressurized. In a gravity fed configuration, filtration is driven by gravity and filtrate is pumped to the next process. In pressurized filtration, filtration occurs inside of a pressure vessel and the feed stream is pressurized ahead of the pressure filter vessel. In both cases, the multimedia bed will be designed based on the particle size distribution of the feed water. Multimedia beds are typically constructed using various grades of sand and anthracite filter media. During multimedia filtration, recovered particles are collected during hydraulic backwashing. The waste stream generated by backwashing must be treated or disposed of.

- Bag filters are also provided for large particle protection. These bags are provided in a number of material types and weaves. Polybags are available in micron ratings from 1 micron to 1500 micron nominal rating. The solids loading rates and cost of bag replacement must be considered when evaluating bag filtration for a new project.

- Cartridge filtration uses wound or blown depth style filter elements. Cartridge filters provide highly efficient filtration and recover a large mass of solids prior to blinding. Cartridge filters are manually replaced when spent and as a result are operator intensive especially when feed SDI is moderate to high. Because of the cost and labor required, cartridge filter replacement costs must be considered during filtration system selection for a new project.

- Membrane filtration (MF) systems use micro or ultrafiltration technology to remove particles. These systems are extremely efficient with absolute filtration ratings as low as 0.01 micron. Operation is completely automated and the MF system automatically backwashes to recover filtered solids. The automation improves the water recovery efficiency of the membrane filtration system and greatly reduces operator attention. Using membrane filtration, effluent oil concentrations of less than 10 ppm have been reported. Membrane filtration is capitally expensive but generally justifiable through extended RO membrane life, reduced RO membrane replacement costs and reduced RO downtime for cleaning.

The use of filtration is expected for CBM produced water. The selection of a filtration process will have significant cost implications to the overall treatment process. Effective oil/water/solids (OWS) separation is required for RO produced water treatment because very low levels of oil (<10 ppm dispersed) can cause fouling problems in the RO system. Since RO requires effective TOC and oil removal, the use of ceramic MF will likely be justifiable for produced water RO facilities.

Emerging RO Pretreatment Methods

Emerging technology may play a key role in RO produced water treatment processes. As pretreatment systems develop, and membrane materials improve, the cost of RO produced water desalination is expected to decrease considerably.

Organo clays have long been known for their oil adsorption properties, however recent development has focused on modified bentonite powders. Currently, Aqua Technologies of Wyoming is marketing a modified bentonite adsorption system for produced water treatment. Implementation of organo clays in combination with GAC at a refinery in Wyoming has successfully produced non-toxic water that meets plant discharge requirements.

Development of new pretreatment systems is expected to improve the costs of OWS while improving oil recovery. For example, the DPS separator by Monarch Environmental features a non-clogging filter designed for OWS. During DPS treatment, cake formation at the filter surface is disrupted by a rotating filter element. Centrifugal forces produced by rotation provide classification of rejected solids and improved oil/solids separation. The system is designed to reliable achieve lower than 10 ppm dispersed oil concentration.

Innovative membrane treatment processes, such as vibratory shear enhanced process (VSEP) developed by New Logic Research may lead to cost effective treatment processes. VSEP systems can currently be outfit with micro, ultra, nano and reverse osmosis membrane elements. The elements can be constructed from many materials including Teflon and PVDF microfiltration membranes and may be useful as pretreatment to an RO process.

The recent development of thin film nanocomposite (TFN) membranes by Dr. Freeman at the University of Texas is expected to reduce produced water RO treatment costs. The TFN technology is still developmental and may be available commercially within the next five years.

Case Study: City of Goodyear Bullard Water Campus

During the summer of 2002, water production in Goodyear, AZ was limited when TCE was detected in two of the City's water wells. The City needed additional water quickly to ensure that water production satisfied public demand. RO was implemented to treat water from a few agricultural drainage wells owned by the City. The well pumps were oil lubricated. Low levels of food grade vegetable oil were fed to the new RO system in combination with silt and other colloidal material.

The facility was designed to produce 1.5 million gallons per day of potable water from groundwater wells with TDS ranging from 1,200 to 1,700 ppm. After 3 years of operation, the membranes had become so fouled that they could not be removed from the RO trains. The feed SDI was very high and the cartridge filters, which had been provided to protect the membranes from particles, required replacement on 12 hour intervals. The process was extremely operator intensive and expensive to operate. An understanding was reached by the City that additional pretreatment was required.

The City implemented an additional automatic backwashing strainer process upstream of the RO and an acid feed system. 15-micron rated strainers were provided. The strainers were able to intercept a significant portion of the solids load before RO treatment and the cartridge filter replacement cycle increased to 1-2 weeks. With the use of the acid system achievable water recovery by the facility increased from 72% to 80%.

The reduced solid load greatly slowed the RO system fouling rate. The presence of oil in the RO system required careful CIP cleaning for flux restoration. Because of issues with oil in the RO process, the City eventually replaced the oil lubricated pumps with new water lubricated pumps.

Case Study: City of Scottsdale Water Campus

The City of Scottsdale Water Campus uses MF and RO to treat tertiary effluent from a wastewater reclamation facility. The plant was originally conceived in the early 1990's and the first phase of construction was completed in 1999. At the time of construction, the use of MF upstream of RO was emerging technology that was relatively expensive to implement. Nonetheless, the tertiary effluent produced by the plant contains 5 to 8 ppm of TOC and 1,000 to 1,200 ppm of TDS, and the decision to implement MF ahead of the RO was made by the City.

The RO system with MF pretreatment also requires antiscalant, RO feed pH depression and cartridge filters. As a testament to the pretreatment system selection, the City still uses membranes loaded during the first phase of construction in 1999 and the City's treatment process allows the RO trains to operate at 85% recovery with minimal CIP cleaning requirements. Because of the achievable membrane life at the facility, which is almost 10 years from original membrane element installation, the plant is considered a benchmark for RO process operation.

During conceptual design of the facility, the City made the decision to spend money to provide good pretreatment for the RO process. This decision has significantly reduced the operational labor requirements of the facility and operational costs associated with membrane replacement.

Case Study: Refinery Application

A refinery in California is using a proprietary RO system to treat an aqueous process stream within the refinery. This stream contains low levels of selenium (0.2 to 1.0 mg/L) which must be removed to meet down stream discharge limits to the local sewer authority. The RO system is a part of the selenium control unit. The selenium is concentrated by the RO process with the clean water (permeate) available for other uses in the refinery. The concentrate stream , which is enriched in selenium as well as other dissolved salts and organic matter is further processed in a brine evaporator to achieve near zero liquid discharge.

The RO operation is challenged by high levels of organic (200 to 400 mg/L as TOC) material which has lead to significant fouling of the RO membranes resulting in frequent chemical cleaning and premature membrane replacement. The organic material has not been well characterized but does contain phenols, diphenols and organic acids. Therefore, the organic material is dissolved and not free oil. Membrane autopsies have shown the foulant to be primarily organic in nature. This application has some characteristics of produced water, specifically the presence of organic membrane foulants. In other respects this application is different from produced water in that the total dissolved solids (salts) concentration is relatively low while the temperature and pH are elevated (150 ^{o}F and pH around 10). Various pretreatment strategies have been investigated to control the organic foulant. These include (1) chemical oxidation (2) specialty adsorbents (3) biological treatment and (4) membrane treatment.

Various oxidants have been applied in bench top experiments including chlorine, peroxide, Fenton's reagent and ozone. None of these oxidants have shown to be particularly successful in reducing the organic contaminant even when applied at high concentration for significant periods (90 minutes) and at the relatively high temperature.

A range of adsorbents have been tried including (1) granular activated carbon, (2) a proprietary organo-clay media, (3)combinations of clay and anthracite and various proprietary media specifically designed for organics removal. The proprietary organo-clay media showed the best performance (up to 90% TOC removal) in bench top experiments. The adsorbing capacity of the media on this particular stream is yet to be verified by pilot testing this will verify the economics of the proposed process.

Similarly a number of ultrafiltration and nanofiltration membranes have been tested to investigate the potential for developing a dual membrane system where the organic contaminant would be removed in a UF or NF system upstream of the RO which would improve the performance of the RO significantly. Candidate membranes have been identified which appear to have the necessary properties of passing the selenium while rejecting the organic contaminants. The approach also needs further bench and pilot testing to confirm long term performance and to demonstrate that the membrane fouling problem is not just shifted from the RO membranes to an upstream membrane pretreatment step.

This project demonstrates the range of solutions potentially available to solve RO membrane performance issues which may arise when applying the technology to new and uncharacterized aqueous streams. As produced water can differ from site to site the success or failure of various pretreatment strategies will depend upon how well the contaminants in the produced water are characterized and understood.

Conclusions

As regulations and the need for reuse of produced water develop, the use of RO may become more common in produced water management strategies because water treated by RO provides flexibility of reuse options with minimal additional treatment. The use of RO as a stand alone produced water treatment process is unlikely since there are practical limitations in water recovery imposed by salt solubility, organic fouling and scaling potential of the produced water. Moreover, concentrate from the RO process requires a desalination process cable of operation in extremely salty conditions and the use of RO will require considerable pretreatment with resulting capital cost consequences.

Effective RO pretreatment greatly improves RO treatment system economics. As illustrated by the case studies, the decision to purchase an effective pretreatment system has paid off through reduced operational requirements and extended membrane life.

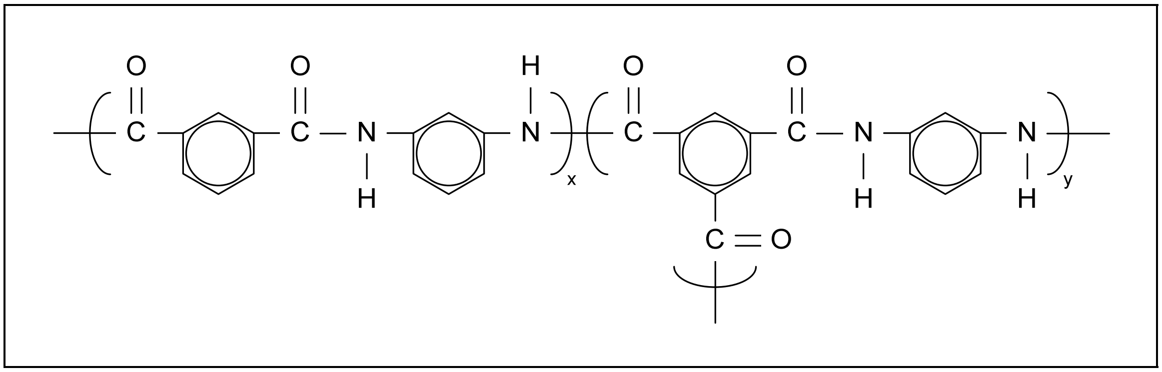

Figure 1 – Typical Thin Film Composite Membrane Polymer Structure

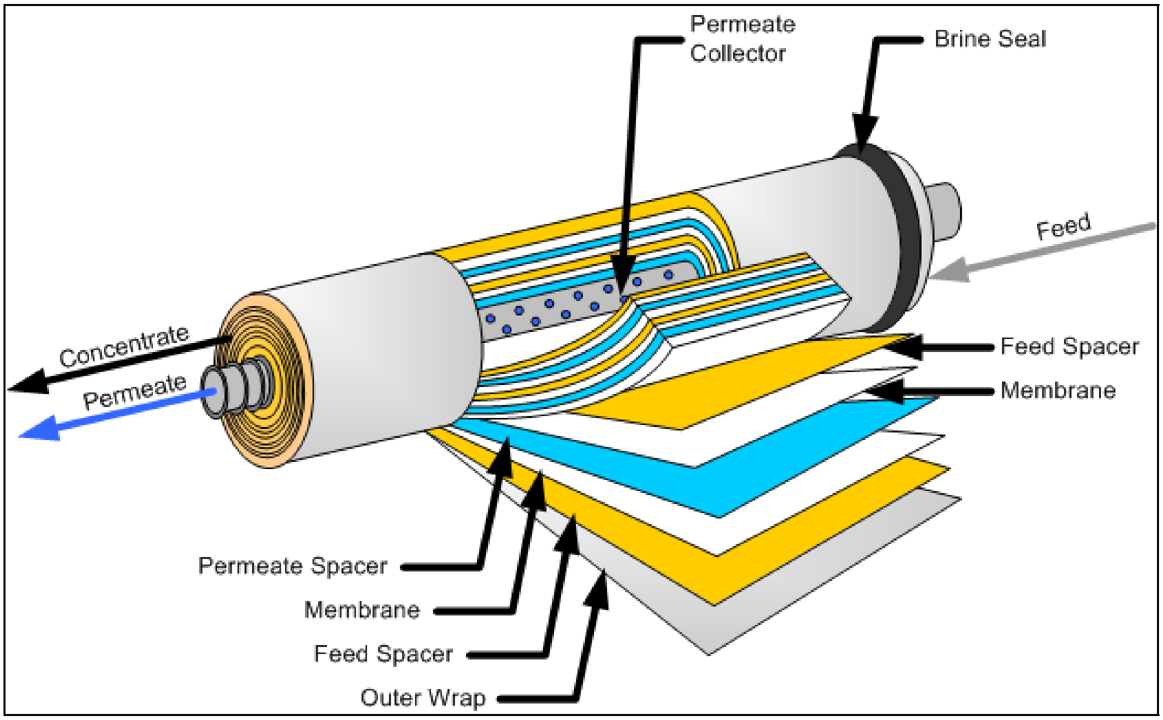

Figure 2 – Typical Spiral Wound Membrane Element

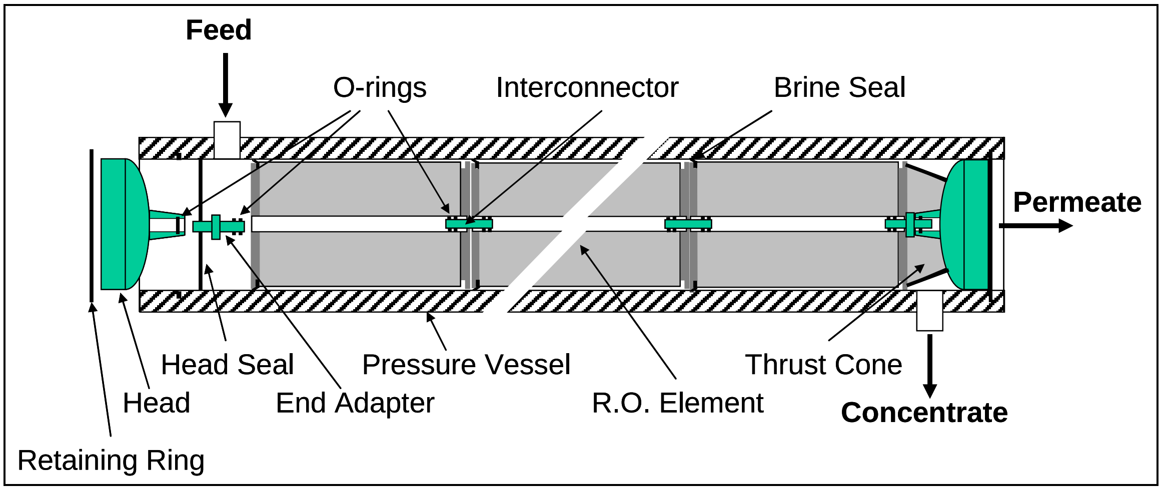

Figure 3 – Typical Spiral Wound RO Pressure Vessel Configuration

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