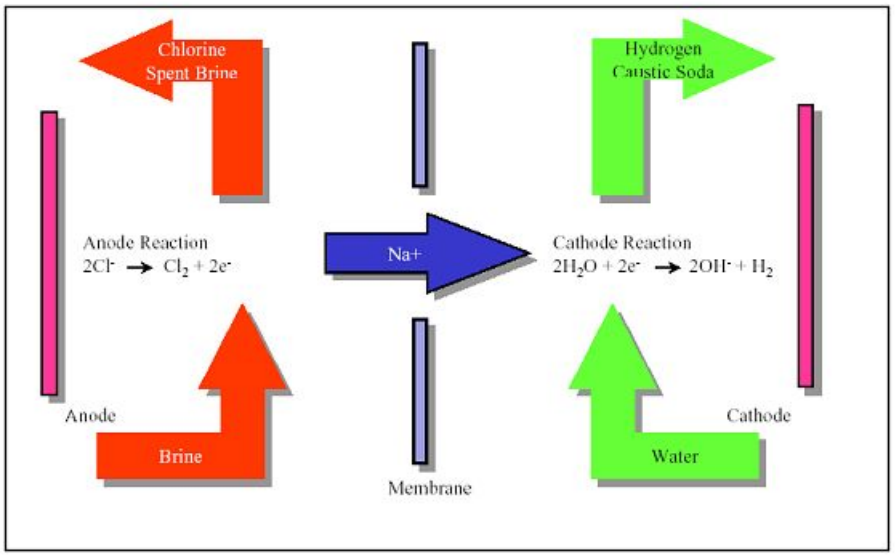
Operation Instruction Guideline for GI-series PFSA Membranes for Chlor-Alkali Processes

Preface:

GI-series PFSA (perfluorosulfonic acid) based ion-exchange membrane is cation exchange membrane and it can be used in NaCl and KCl Electrolysis applications. Cation ions such as Na^+ ion or K^+ ion can be transferred through the membrane under an electric field. PFSA membranes would naturally not allow the transport of Cl^- or OH^- to a certain extend and this will be a function of the electrolysis hardware design and operational parameters.

With GI-series PFSA membrane, 15-32 % NaOH production can be achieved.

A basic schematic of a chlor-alkali process is given in the image below.



Safety related notes:

-Use PPE when handling this membrane.

-Irritation and skin rashes may be observed in the case of handling the membrane with bare hand and not using the proper PPE such as gloves, etc.

-Oral Toxicity Testing Laboratory to mouse, It is Low toxicity. LD > 20000mg/kg.

-The PFSA membranes are air stable and will not ignite with air or low concentrations of oxygen.

-With oxygen-rich environments, necessary precautions need to be taken to prevent the damage to the membrane through other mechanisms such as chemical combustion of H2 gas in the O2 side and creating a fire and other events of this nature.

-At temperatures greater than 100 deg Celsius, PFSA membranes may result in fire under dry conditions or fully hydrated conditions. In such cases, personnel trying to put the fire down should use insulated air respirators and the proper PPE because by-products of the ignition will be composed of F2, HF, and other hazardous compounds such as CO, CO2, and SO3 type gases.

-Ensure the complete physical separation of anode and cathode compartments from each other with the help of a proper gasketing material and hardware design so that H2 and O2 gases do not mix with each other and create flammable or ignitable gas compositions.

Disposal related notes:

-Please follow the local regulations and protocols.

-Thermal decomposition based methods can be used to decompose PFSA based materials as long as the exhaust is connected to a lye absorption bed where the acidic harmful gases are captured and neutralized within the alkaline bed.

Please keep the membrane intact.
Membrane must be installed in strict accordance with instructions and technical procedures according to the supplier's recommendations. Prior to installation of the membrane, please ensure that assembly area does not have any tools or sharp objects that would create pinholes or induce membrane tearing.

Pretreatment of the membrane and specific membrane installation steps may be required according to the instructions given in this manual or according to the end-users own hardware and operational parameters. Information provided in this document is solely for reference and guideline purposes. The manufacturer of the membrane and distributors are not to be held accountable for the damages originating from the improper usage of this product.

Additional Safety Measures
-Minimize the differential pressure across the membrane in order to prevent material's fatigue issues.

-Monitor the concentrations of various gases generated at the anode and cathode compartments with the proper gas sensing devices.

-Prior to idling the operation or shutting down unit, ensure a good nitrogen gas purging to remove the hydrogen, chlorine, and other gases from the respective compartments.

-During purging event, do not intermix the anode and cathode inlet or outlets in order to prevent creating hazardous conditions such as generation of flammable gas mixtures of H_2/O_2 or H_2/Cl_2, etc. Unexpected reactions may occur and this may result in differential pressure increase that would result in membrane rupture or through other mechanisms that would create a safety issue where the membrane is ruptured and anode and cathode chamber products combining with each other and further cascading issues.

-Avoid current reversal or polarity reversal during the operation of the cell.

Membrane handling and installation

During the caustic pretreatment of the membrane, it is important to keep the membrane completely flat and secure between two pieces of restraining components such as plastic mesh and avoid the pinhole, crease, or scratching of the membrane surface. Prior to the treatment, inspect the membrane for any damages , then properly store it until its pretreatment, then cut the membrane with the proper cutting tools or devices to the desired size, and finally install the membrane to the pretreatment bath.

During the transportation of the membrane (whether unused or pretreated), the bending radius needs to be kept at a minimum of 10cm diameter and rolled up around a core with such a large size. If this method is not feasible, then the membrane needs to be transported on a flat surface in the secured form in order to prevent wrinkling, folding, or curling during transportation step. Unnecessary handling will increase the likelihood of physical damage to the membrane.

Shipping Instructions

GI-series membranes are usually shipped in a roll form that is wrapped around around cylindrical core or in the flat sheet forms. Membranes are shipped in the dry format without any liquid in them.

If the external packaging is done with cylindrical PVC pipe, then the core/membrane will be placed inside another wood crate and then shipped.

Storage Instructions

After receiving the package, the core/membrane component needs to be stored in an environment with a temperature temperature is in 20-25 deg Celsius with the storage humidity being kept around 50-60% RH. The membrane can stay wrapped around the core that it came or it needs to be kept flat and secured while it is being stored.

-Caution: DO NOT freeze the membrane or store it under freezing or sub-zero temperatures. Moisture present inside the membrane or absorbed by the membrane during storage will form ice crystals and may potentially result in pin holes or formation of physical damages across the entire membrane roll.

Caution: Keep the membrane away from direct sunlight.

Caution: Do not fold the membrane or force-ably stretch the membrane while handling.

Pre-treatment
GI-series PFSA membranes need to pre-expanded prior to their installations, preferably under conditions similar to the targeted operational conditions in order to prevent expansion or shrinkage inside the hardware.

0.1 wt% alkaline solution or pH 11-12 alkaline solution such as NaOH or KOH can be used for the pre-expnasion of the membrane. Per square meter of film processing, it is estimated that approximately 5 Liters of alkaline electrolyte may be needed assuming that the dept of the pre-treatment bath is around 3-5 cm.

Some cell operating conditions require further expansion of the membrane to prevent membrane further forming wrinkles during operation. In this case, the hot water treatment of the film can be further expanded (water not more than 60 degrees). Deionized water or soft water with less than 5% calcium and magnesium (total concentration) can be used.

Immersion time of 4-12 hours and a temperature range of 18-40 ℃ can be used for this pre-treatment.

The membrane can be hanged or horizontally placed in a flat form to for a complete submersion into the pre-treatment bath or pool.

The membrane must not be folded or creased during pre-treatment step.

If multiple membranes to be stacked up on top of each other during pre-treatment, the physical porous separator needs to be placed in between each one of them in order to ensure sufficient alkaline electrolyte or pre-treatment electrolyte gets in contact both all sides and faces of the membranes.

Installation
Handling the membrane

-During the removal of the membrane from the container or pre-treatment bath, care needs to be given that the membrane does not get folded, creased, and its surface does not get scratched.

-Install the membrane while it is still wet or partially wet. Do not allow the membrane to dry out completely during the assembly or installation stage.

-Depending on the size of the membrane, it may be required to have two or multiple individuals to move/assemble the sheets of the membrane.

Sealing
For best performance, a gasket sealant coating / release agent may be needed for the following reasons:

-To prevent the membrane sticking to the gasket.

-Establish a barrier layer on the surface of the membrane and gasketing and prevent leakage due to irregular surface topology because of the PTFE fabric fibers.

-Provide a lubricated gasket surface to prevent tearing of the membrane during the fastening or clamping stage. PFSA membranes tend to stick to gasketing surfaces and create a situation where the membrane may move during the stack clamping in an uneven way and force the membrane to tear and this can be avoided with the help of a sealant coating or release agent.

-Sealant / release agent is selected according to the type of membrane and its mounting techniques that would be used.

Note: Regardless of what kind of sealant use / release agent, do not apply the sealant or release agent to the surfaces that may allow leaching of the sealant and release agent and get these into the electrochemical device as a contaminant and potentially damage the cell components or negatively affect the electrochemical performance and this needs to be avoided.

Installation of the membrane inside the electrochemical cell

-Before installing the membrane, please check the electrode and remove bumps or burrs or other sharp pointy features from the electrode in order not to physically damage the membrane.

-If there is a differential pressure across the membrane, place the smooth surface towards the high pressure side.

-Carefully place the membrane in the cell while it is still wet. Avoid dragging of the membrane over the edge of the metal electrode to prevent damage to the membrane surface.

-If a folding, wrinkling, or any other phenomenon is observed, pull off the membrane from electrode surface without dragging and retry it.

-If a membrane is to be hoisted with an object like hoisting hook, ensure that the edges are smooth and does not create tearing effect. Avoid pulling off the membrane too tightly. Care needs to be given to the weight and size of the membrane for this approach in order to prevent damage to the membrane from the hoisting hook.

-During the entire installation, the membrane needs to be kept wet or moist. If necessary , the membrane can be sprayed with a solution that is similar to the pre-treatment bath or with an electrolyte that will be used during its operation.

-After installation of the membrane in the cell, add a small amount of anolyte and catholyte solutions to the respective chambers in order to keep the membrane and the cell fully wet or moist to prevent the membrane drying out.

-If a membrane dries out prior to the installation, repeat the wetting process.

-If the membrane dries out inside the electrochemical cell after installation, there will be shrinkage and re-swelling after the wetting, which will adversely affect the lifetime of the membrane.

Electrolyzer operation

The operating procedure for the chlor-alkali cell can vary according to design of the hardware and what the end-user is trying to achieve. The main purpose of this guideline is to provide some suggestions that would improve the performance and membrane lifetime.

New Membrane Activation Protocol

Prior to the assembly of the membrane in the electrochemical cell, please ensure that the following salt solution is prepared and ready to use for anode side: sodium chloride salt solution that is prepared with de-ionized water or distilled water of quality (2 mega-ohms or greater, preferably 16-18 mega-ohms) with a concentration of 210 grams per liter of water or more for the salt and the pH being 2 or somewhere close to this value. For the cathode side, prepare an alkaline solution of NaOH with the concentration being in the range of 15 wt% to 20 wt% and not more than 20 wt%.

-After the assembly of the membrane inside the chlor-alkali cell or electrochemical device, start flowing these anode and cathode solutions at the respective sides. The flow rate of the cathode can be slightly higher than the flow rate of the anode electrolyte during this step if it is needed by the end-user.

-As soon as the anode and cathode electrolytes start to come out of the anode and cathode outlets, start applying a current and make sure the individual cell voltage does not exceed 4 V. While this current is being applied, the temperature of the electrolytes and the cell can be gradually increased to 70 deg Celsius. Do not exceed 85 deg Celsius during the new membrane activation stage.

-Do not wait for the electrolytes and cell to warm in order to bias the cell with the electrical current. In fact, it is essential that the electrical current is applied as soon as the anode and cathode electrolytes are coming out of the outer ports of the respective chambers. Ionically conducting membranes have a back-diffusion or reactant crossover issues when there are two different electrolytes at the opposing sides of the membrane and supplying the cell with some level of electrical current will greatly minimize the crossover based reactant contamination issues.

-During the first 24 hours of the activation protocol, maintain an electrical current density of less then 4 kA/m2. After then, the current can be increased to the desired level as long as the individual cell voltages are less then 4 V per cell.

-For level of impurities, ensure the specifications provided below.

-It is critical to use the essential monitoring, alarms, and adjustments in order to have a good operational lifetime for the membrane and safe operation of the electrochemical cell.

Specifications	Operational conditions	Frequency of monitoring of anode/cathode baths
Electric current	According to the need to control the current density	Constantly
Current density	1.5-6.0 kA/m2	Constantly
Voltage	Less than 4.0V per cell (high voltage means high alkaline electrolyte concentration at cathode, low pH at anode, or some other issue, and these conditions may damage the membrane)	Constantly
Current efficiency	To be decided based on the operaitonal condition or history of the membrane	On a need basis
Anode chamber electrolyet specifications		
Feed concentration	Sodium chloride concentration greater than 210 grams/liter of water	Constantly
Feed brine flow rate	According to the need to control the anolyte concentration	Constantly
Amount of acid addition to maintain the pH	According to the need to control the anolyte concentration	Constantly
Anolyte concentration	200 ± 30 grams of sodium chloride salt per liter of water used to make the anolyte solution	Constantly
Anolyte pH	2 or greater (pH > 2)	Constantly
Sodium chloride content for anolyte	Not to deviate from the optimal setpoint by <20 g/L (The optimal set point is 200-210 grams per liter of water, hence the minimum value is 180-190 grams of salt per liter of water and the maximum value is 220-230 grams per liter of water)	Once a day
Anode electrolyte temperature	80-90 deg Celsius	Constantly

Anode Feed Impurities		
Calcium/Magnesium (Total)	<30 ppb for less than 4 kA/m2 or <20 ppb for 4-6 kA/m2	Once every 8 hours
Strontium	<500 ppb for less than 4 kA/m2 or <400 ppb for 4-6 kA/m2	Once a week
Sodium sulfate	<10 grams/Liter for less than 4 kA/m2 or <8 grams/Liter for 4-6 kA/m2	Once a day
Iodine	<1 ppm for less than 4 kA/m2 or <200 ppb for 4-6 kA/m2	Once a week
Barium	<1 ppm for less than 4 kA/m2 or <500 ppb for 4-6 kA/m2	Once a week
Aluminum silicates	<100 ppb for aluminum silicates or <10ppm for SiO2 for current density less than 4 kA/cm2 or lower than these ranges for current density 4-6 kA/m2	Once a week
Issues with Impurities		
Calcium/Magnesium (Total)	If the concentration of impurities are out the provided ranges, there will be deposit formation on the membrane surface.	
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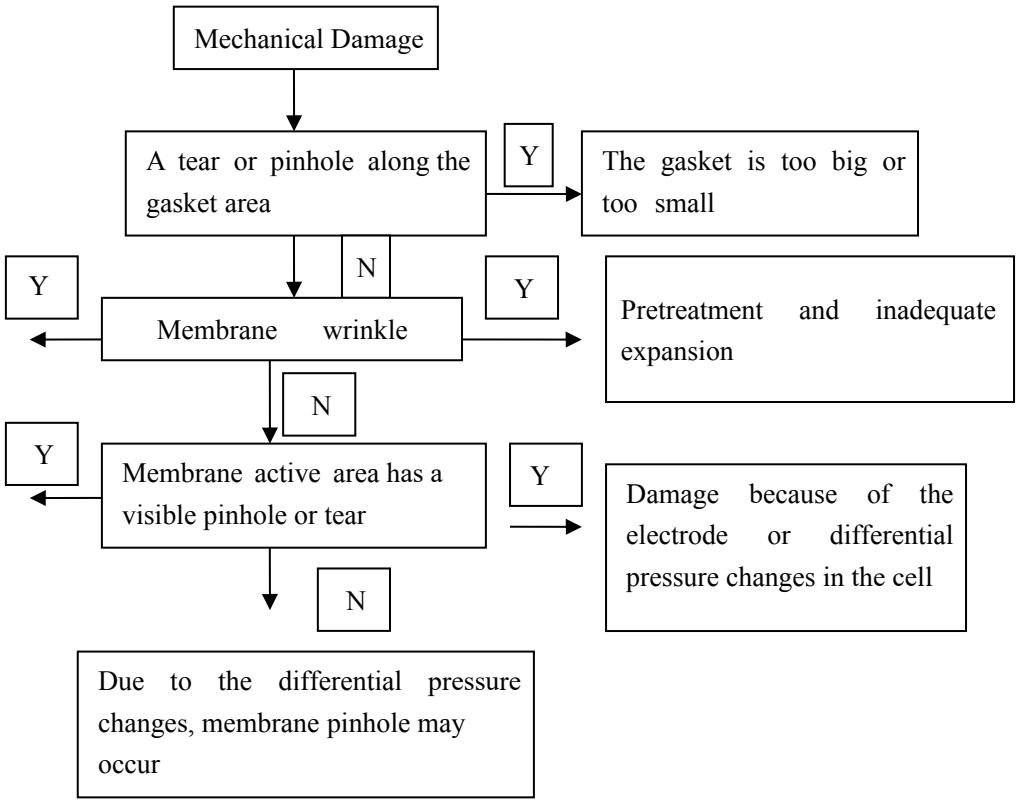
Sodium sulfate	If the concentration of impurities are out the provided ranges, there will be deposit formation on the membrane surface.	
Iodine	Periodate formation at the anode side	
Barium	If the concentration of impurities are out the provided ranges, there will be deposit formation on the membrane surface.	
Aluminum silicates	If the concentration of impurities are out the provided ranges, there will be deposit formation on the membrane surface.	

Troubleshooting the mechanical damage on the membrane component



Operating conditions
-Different operating conditions will naturally have different output for the by-products and how this is going to impact the membrane lifetime. have what effect on the membrane.

Electrolyzer conditions
-Current density: Recommended current density range 1.5-6.0kA/m2 during the regular operation of the electrochemical cell.

-If the current density exceeds 6 kA/m2, depending on operating conditions and other factors, this may exceed the maximum capacity of the membrane. At this point film may be subject to physical damage, the current efficiency decreases. High current densities can result in many different outcomes including micro-hole formation.

-If the current density is less than 1.5 kA/m2, the film will not be damaged.

-Proper adjustments in the anode and cathode electrolyte concentrations and use of the membrane in the range of 1.5 kA/m2 to 6 kA/m2 range will extend the membrane lifetime.

Cell temperature
-The recommended temperature range is 80-95 ℃.

-If the cell is cooler or cold, such as less than 80 deg Celsius, the ionic conductivity of the membrane will be low and this will effect the current efficiency or membrane lifetime. Forcing the electrochemical cell at higher current density while it is cold can potentially damage the membrane.

-If the cell temperature exceeds 95 deg Celsius, the membrane will swell extensively and also by-product gases partial pressures will increase and may generate a unbalanced pressure issue across the membrane. Extensive swelling will increase the likelihood of wrinkle formation. Unbalanced pressure between anode and cathode may increase the diffusion of some of the reactants to the low pressure side or create material fatigue issues.

Electrode spacing
-If the spacing between cathode and membrane is too narrow or short, the fluid transfer will have resistance and this will adversely impact the overall cell potential or cell voltage. In chlor-alkali processes, the most commonly used spacing is 2-3 mm between the membrane surface and anode/cathode electrode surface. Use of proper gasketing and design will ensure that reactants, electrolytes, and by-products would not create any adverse issues for their transfer or removal.

Operational pressure related notes

-A balanced pressure based cell operation is recommend in order to prevent various damages to the membrane and electrochemical cell.

-Design based aspects such as use of long tubes for plumbing or use of narrower or wider tubes for transfer of electrolytes, presence of obstructing design aspects can upset balanced pressure operation by creating differential pressure situations and these needs to be avoided.

-It is always recommend to monitor the pressure values both at anode and cathode for liquid and gases at multiple points and maintain the minimal pressure drop from inlet to outlet ports of the same side and also minimize the differetial pressure from anode to cathode or from cathode to anode (meaning across the membrane).

In the case of unbalanced pressure between anode/cathode compartments or large pressure drop from the inlet to the outlet of the same side cases:

-Membrane can be pressed towards the lower pressure side via balloning effect and results in physical deformation or experience fatqigue issues.It is pressed on the opposite side's electrode too strongly, there may be pinhole or tear formation.

-A reduction in the 2-3 mm spacing between electrode and memrbane surface may restrict the electrolyte flow and block the removal of the by-product.

-Vapor lock formation

Disclaimer

The membrane manufacturer and distributor of this product cannot be held liable for the damages that may occur because of the improper usage of this product. The provided instructions in this document is solely for reference purposes. Different electrochemical devices have different design, operational, and other aspects and the end-user needs to follow the best engineering practices and get themselves familiarize with the technology and most commonly used operational parameters within the industry by reviewing the published research articles, and other documentation that is available in the public domain or through the various publisher outlets. The membrane manufacturer and distributors of this product are not obligated to share such documentation.