IONOMR INNOVATIONS

# PEMION® APPLICATION NOTE:

## Handling, Dispersion, and Ink Formulation

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# PROTON EXCHANGE MEMBRANES: HANDLING, STORAGE AND PRE-TREATMENT INSTRUCTIONS

## GENERAL

Ionomr Pemion® membranes are the only hydrocarbon-based cation exchange membranes with the required chemical stability for operation in fuel cells. They have low ionic resistance, ultra-low hydrogen crossover, high electrical resistance, and offer excellent chemical and mechanical durability.

This document serves as a general guide for the handling, use, and storage of Pemion® membranes.

## BEFORE YOU START

- Refer to the Pemion® Safety Data Sheet (SDS), and follow appropriate safety practices. Although Pemion® membranes are supplied in a dry state, the packaged product may contain residual water or trace solvents.
- Store, handle and process the membrane in a clean, dust-free, and well-ventilated environment, preferably in a relative humidity (RH) and temperature-controlled environment between 35-50% RH and 20-25 °C.
- All surfaces that come in contact with the membrane during handling, inspection, treatment, storage, and installation should be smooth and clean.
- Pemion® membranes are sensitive to moisture content, and may expand or contract in response to changes in relative humidity. Prior to handling of Pemion® membranes, Ionomr recommends opening the packaging and allowing the membranes to equilibrate to the ambient conditions of the environment for 24 hours.
- In addition, Ionomr recommends equilibration of test samples (once cut from the membrane) for another 24 hours prior to their use.

## HANDLING

Gloves should be worn at all times when handling Pemion® membranes.

- The membrane should be handled with care: do not puncture, crease, abrase, or tear the membrane. Any punctures, creases, lacerations, or abrasions of any kind may reduce membrane performance, or increase the likelihood of leaks (e.g., gas crossover).
- When cutting membranes to desired dimensions, best results are achieved by cutting membranes dry (prior to wetting/soaking, if applicable) using a new, sharp blade, or high-quality sharpened die. Care should be taken when cutting membranes and related membrane electrode assembly components via laser, because incorrect laser settings or operation may result in burred edges & negatively impact materials integrity.
- Pemion® membranes are incompatible with soaking in polar solvents such as alcohols for extended durations; Pemion® is readily soluble in polar solvents (see List of Suitable Solvents for Pemion®), but is insoluble in water.
- While on the clear backing sheet, Pemion® membranes are not compatible with temperatures exceeding 80 °C or coating with solvents containing > 50% w/w water.
- Free-standing Pemion® membranes (i.e., once the backer has been removed) are not compatible with temperatures exceeding 160 °C.
- Pemion® membranes are best used as-received. No pre-treatment is necessary, and soaking in aqueous solutions is not recommended. Pemion® will readily ion-exchange with aqueous electrolytes (cations) in solutions containing them.
- For applications requiring radical scavengers or antioxidants, it is recommended that their integration is achieved through incorporation into cell electrodes during cell assembly, rather than by soaking/exchange of the membrane.

## STORAGE

- Long-term storage of Pemion® membranes is best done in the native, dry form, in original packaging with minimum exposure to heat, light, and changes to relative humidity. Storage in original packaging or humidity barrier bags between 50-60% RH is recommended.
- Wet storage is not recommended due to swelling of the material in aqueous environments.

# REMOVAL OF PEMION® MEMBRANES FROM THE BACKING LAYER:

## Primary method

1. With clean gloved hands, hold the membrane on with its backing layer facing down/away.

2. Using a thumb or a finger, rub against the corner edge of the membrane to produce separation from the backing layer.

3. Once corner separation from the backing layer is achieved, carefully and gently, begin to pull the membrane from the backing layer whilst holding the backer down on a clean dry surface.

4. As the backing layer is released, support the membrane as it is removed from the backing layer, until all of the membrane has been removed.

## Secondary method

1. With clean gloves, wet a portion of the membrane/backing layer edge with de-ionized water; this should aid in separation of the membrane from the backing layer. Repeat the steps 3 to 4 in the primary method

2. If the membrane does not separate upon initial wetting, apply a small amount of water across an area close to the edge of the membrane and repeat the primary method. Do not subsequently use the wetted portion of the membrane in the device active area.

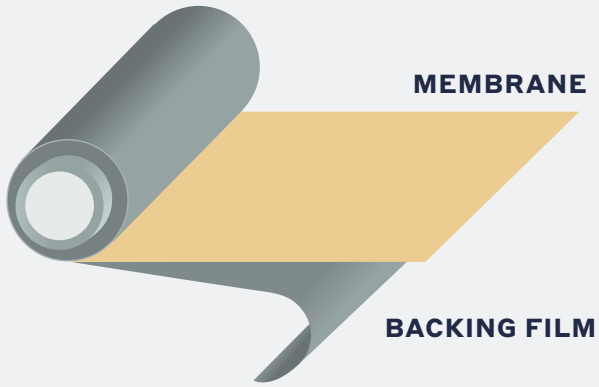
### FOR COATING PEMION® MEMBRANE AFTER REMOVING FROM THE BACKING LAYER

Ensure that the membrane remains flat.

A powder coating masking tape (e.g., McMaster-Carr: https://www.mcmaster.com/7630A24/), or generally low bonding strength tape is recommended to overlap the edges of the membrane prior to coating procedures, to stabilize the membrane in place. This will help maintain the membrane positioning, and eliminate stress lines that may develop.

Use caution when removing tape from the membrane after the coating process, as because tape adhesives may strongly bind to Pemion®.

Roll Unwind Orientation
(Base Film Facing Out)



# PROTON EXCHANGE IONOMERS: DISPERSION AND INK FORMULATION

## DISPERSION AND INK FORMULATION

**The following is a starting point for preparing catalyst inks based on an ink formulation designed for fuel cell catalyst layers.** Please note that different ionomer content may be required depending on the application and operational conditions required. Similarly, depending on the application and performance requirements, further optimization may be necessary, such as solvent composition and weight percent of solids in solution.

1. Calculate the mass of polymer, catalyst powder, and solvents required for electrode composition. This is heavily application-dependent.
The recommended solvent ratio is 1:1 alcohol to water (e.g., 1:1 IPA/water). The primary alcohol can be adjusted after the polymer is dissolved in step 2. The volume of solvents required should be dictated by a final weight percent of total solids (catalyst powder + ionomer), with 1-2 wt% suggested for electrode application by spray-coating, or higher as necessary for direct coating catalyst layer deposition methods. One method of controlling drying characteristics (e.g., membrane swelling causing wrinkled catalyst layers, catalyst layer porosity, etc.) is to alter the alcohol ratio; it is strongly advisable not to increase this ratio above 3:1 (alcohol/water).

**Note: The density of Pemion is 1.2 g/mL**. When adapting existing ink formulations incorporating polymers of other densities, an initial approximation based on polymer density and vol% is suggested. For instance, the density of typical PFSA-based ionomers is approx. 2.0 g/mL. Hence, an ink based on 30 wt% of a ~2.0 g/mL polymer such as a PFSA would be approximately equivalent to 18 wt% Pemion.

Ionomr recommends starting with an ink solids composition of 15 wt% ionomer powder and 85 wt% catalyst powder. For example, if utilizing a Pt/C catalyst powder which contains 45% Pt by weight, this recommended formulation would comprise 15 wt% ionomer, 38 wt% Pt catalyst, and 47 wt% carbon, corresponding to an ionomer-to-platinum ratio of 0.39, and ionomer-to-carbon ratio of 0.32.

### Disclaimer

Ionomr Innovations Inc. is not responsible for any damages or loss of materials when preparing catalyst inks. Always use caution when using or mixing any finely divided metal catalyst particles with flammable solvents as spontaneous combustion may occur. Use proper containment procedures and wear appropriate personal protective equipment at all times.

2. On a magnetic stirring plate, prepare a 3-5 wt% solution of Pemion and suitable solvent or solvent mixture from below (e.g., methanol) to dissolve the polymer. Reserve a small portion (e.g., 5 mL) of the chosen alcohol to capture the concentrated ionomer coating the glassware after dropwise addition (mentioned later).

Ionomr strongly recommends slow addition of its ionomer powder to stirring solvent (e.g., 400-600 rpm), rather than addition of solvent to the ionomer powder, to ensure uniform dispersion and faster dissolution times. Mild heating during dissolution is recommended (e.g., 60 °C). Typically, heating and stirring for 24-48 hours is sufficient when preparing a Pemion ionomer solution. If particles are noted, the ionomer solution can be passed through a glass fiber filter to eliminate any insoluble and contaminants. Di

3. Preferably in a narrow-necked glass bottle and on a magnetic stirring plate, add the catalyst (e.g., Pt/C) powder followed by a stir bar, and all of the calculated water. Stir gently (e.g., 100 RPM) until catalyst powder is fully wetted and dispersed. Increase stir rate until a vortex begins to form but before cavitation (e.g., 400-600 RPM, heavily dependent on ink volume and stir bar size).

4. Into the fully wetted catalyst powder mixture, slowly pour in the calculated alcohol, excluding the 5 mL reserve solvent (from step 2) or the alcohol which is contained in the ionomer solution.

5. Maintain vigorous stirring and add the alcohol/ionomer solution dropwise, visually ensuring surface accumulation of polymer is kept to a minimum. Occasional swishing of the solution may help to integrate catalyst particles that accumulate at the ink-bottle interface. Using the remaining 5 mL reserve of alcohol, rinse the ionomer glassware (i.e., capture the residual polymer so calculated values for ionomer are realized) and dislodge any remaining catalyst powder from the sides of the ink bottle. Stir at moderate rate (e.g., 300 RPM) until use; a minimum of four hours is recommended. Treatment in a low-power sonication bath for 15-30 minutes after an initial 30-60 minutes of stirring may help homogeneity.

# LIST OF SUITABLE SOLVENTS FOR PEMION™ POLYMER PP1-HNN8-00

Dissolution of Pemion® ionomer is typically achieved following stirring and gentle heating (e.g., 250-500 rpm at 60 °C) in a chosen solvent for 24-48 hours. Filtration of the polymer solution following dissolution is recommended. A sample procedure is given below the dissolution table.

| Solvent Type | Comments | Solubility wt% |
|---|---|---|
| Methanol | Low-boiling solvent for spray coating, electrode fabrication, etc. | **1% – 10%** |
| Reagent Alcohol (85% EtOHI/ 5% MeOH/ 5% isopropanol) | Low-boiling solvent for spray coating, electrode fabrication, etc. | **1% - 10%** |
| Ethanol | Low-boiling solvent for spray coating, electrode fabrication, etc. | **1% - 7%** |
| Ethanol/IPA (50:50 by weight) | High boiling point solvents, can cause complications with gas permeability | **1% - 5%** |
| IPA/water (50:50 by volume) | Low-boiling solvent for spray coating, electrode fabrication, etc | **1% – 10%** |
| DMSO | Recommended high boiling point solvent to achieve high viscosity, can cause complications with gas permeability | **1% - 10%** |
| NMP, DMF | High boiling point solvents, can cause complications with gas permeability | **1% - 10%** |

### PREPARING A 5 WT% SOLUTION OF PEMION® IONOMER IN METHANOL:

To a 20 mL glass scintillation vial with a stir bar was added 9.5 g of suitable alcohol or solvent mixture (e.g., methanol). The vial was placed on a magnetic stirring hot plate and stirred at 300 rpm. Pemion® ionomer (0.5 g) was slowly added to the solvent over the course of 10 seconds. The resulting 5 wt% polymer suspension was stirred at 300 rpm with heating at 60 °C for 48 hours, giving a viscous and uniform polymer solution. Slow addition of the ionomer to the solvent helps prevent clumping and improves dissolution characteristics. After cooling to room temperature, the solution was filtered through a 55 mm diameter 11 µm pore size Grade 1 Whatman™ (CAT no. 1001-055) filter paper to remove any insoluble materials before further use.

# DOCUMENT CHANGE HISTORY

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|---|---|---|---|
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| **Revision** | **Prepared By** | **Approved By** | **Effective Date** |
| H | Omid Toussi | Andrew Belletti | Apr. 7, 2022 |

This document is reviewed to ensure its continuing relevance to the systems and process that it describes.

**REVISION HISTORY:**

| Revision | Date | Description of Changes | Approved By |
|---|---|---|---|
| A | Jan. 27, 2020 | Initial Draft | Ben Britton |
| B | Oct. 16, 2020 | Updated backer removal, ink formulation, solvents | Ben Britton |
| C | Nov. 16, 2020 | Additional solvents | Ben Britton |
| D | Feb 26, 2021 | Document design and name updated | Ben Britton |
| E | May 31, 2021 | Pemion-on-backer incompatibilities added; precautionary statement regarding tape added; general revisions | Ben Britton |
| F | Aug. 10, 2021 | Added new sections: before you start, handling & storage | Ben Britton |
| G | Mar. 2, 2022 | General updates | Andrew Belletti |
| H | Apr. 7, 2022 | General updates | Andrew Belletti |